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APPLICATION OF NON-THERMAL TECHNOLOGIES FOR MINIMALLY PROCESSED FRUITS AND VEGETABLES QUALITY INCREASE AND PRODUCT INNOVATION

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Abstract:

A wide variety of minimally processed fruits and vegetables has been developed in order to respond to the increasing consumers' demands for ready-to-eat and ready-to-use fruits and vegetables products. Creating innovative products with high quality and pleasing the consumers palate is always a concern for the food industry. The present thesis mainly discusses the application of non-thermal technologies such as Osmotic Dehydration (OD), High Hydrostatic Pressure (HHP), Pulsed Electric Fields (PEF) and Vacuum impregnation (VI) aimed at product innovation and quality improvement for minimally processed fruits and vegetables. The main results related to the applied non-thermal techniques confirmed their potentiality but also the need to optimize them as a function of raw material characteristics and final product target.

The application of HHP was evaluated as an alternative to heat treatment prior to OD for the production of candied green plums, as it was shown to efficiently accelerate the mass transfer during the OD process, allowing to reduce the dehydration time from more than 5 days to about 2 days. HPP allowed to maintain higher firmness values and a greener colour of the final product, to retain the antioxidant compounds content and the antioxidant activity and to improve the aromatic profile (in terms of total amount and number of volatile components) of the final product.

The pre-treatment with PEF in raw potatoes was proven effective for reducing the final acrylamide content in deep-fat fried potato crisps by favoring the release of acrylamide precursors during the further dipping, with only slight modifications of the final quality of the product.

VI was applied for the aromatic enrichment of potato sticks showing a good potentiality for product innovation. However, based on the characteristics of the raw materials, the process needs to be improved in order to guarantee the quality of the product during storage.

Key words:

Minimally processed fruits and vegetables; Non-thermal technologies; Osmotic Dehydration; High Hydrostatic Pressure; Pulsed Electric Fields; Vacuum Impregnation.

List of publications:

This thesis is based on the following Papers, as attached in "List of papers" section:

I. **Luo, W.**, Tappi, S., Wang, C., Yong, Y., Zhu, S.*, Rocculi, P., Dalla Rosa, M. (2018) Study of the effect of High Hydrostatic Pressure (HHP) on the osmotic dehydration mechanism and kinetics of wumei fruit (Prunus mume). *Food and Bioprocess Technology*, *11*(11), 2044-2054.

II. **Luo, W.**, Tappi, S., Wang, C., Yong, Y., Zhu, S.*, Rocculi, P., Dalla Rosa, M. (2018) Study and optimization of High Hydrostatic Pressure (HHP) to improve mass transfer and quality characteristics of candied green plums (Prunus mume). *Journal of Food Processing and Preservation*, *42*(11), e13769.

III. **Luo, W.**, Tappi, S., Wang, C., Yong, Y., Zhu, S.*, Rocculi, P., Dalla Rosa, M. (2019) Effect of High Hydrostatic Pressure (HHP) on the antioxidant and volatile properties of candied wumei fruit (Prunus mume) during osmotic dehydration. *Food and Bioprocess Technology, 12*(1): 98-109.

IV. Genovese, J.*, Tappi, S., **Luo, W.**, Tylewicz U., Marzocchi, S., Marziali, S., Romani, S., Ragni, L., Rocculi, P. (2018) Important factors to consider for acrylamide mitigation in potato crisps using pulsed electric fields. *Innovative Food Science & Emerging Technologies*. Under review.

V. **Luo, W.**, Tappi., S.*, Patrignani F., Romani S., Lanciotti R., Rocculi, P. (2019) Essential rosemary oil enrichment of minimally processed potatoes by vacuum-impregnation. *International Journal of Food Science and Technology*. Under review.

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1. **Introduction and objectives**

Due to the modern busy lifestyle, as well as the increasing demand for healthy, fresh-like and easy to prepare products, the consumption of minimally processed fruits and vegetables has increased remarkably in recent years (Rico et al., 2007). A wide variety of minimally processed fruits and vegetables has been developed in order to respond to the increasing consumers' demands for ready-to-eat fruits and vegetables products (Allende et al., 2006; Froeder et al., 2007). They have been one of the most successful businesses within the food processing industry (Ayala-Zavala, González-Aguilar, & Del-Toro-Sánchez, 2009).

One important factor to succeed in the food industry, including the minimally processed fruits and vegetables market, is creating innovative products with high quality and pleasing the consumers palate (Kader, 2008). Therefore, products innovation and quality improvement are the one of the main objectives of this research.

Since traditional thermal treatments usually cannot be applied in minimal processing industry, as they will damage the fresh state of the products, many non-thermal technologies have been investigated in recent years to increase the quality and stability of minimally processed fruits and vegetables.

The present thesis mainly discusses the application of non-thermal technologies (Osmotic Dehydration, High Hydrostatic Pressure, Pulsed Electric Fields and Vacuum impregnation) with the aim of improving the quality of minimally processed fruits and vegetables. In particular, the research was focused on the following aspects:

1. Evaluate the effects of High Hydrostatic Pressure (HHP) as a pre-treatment before Osmotic Dehydration (OD), as an alternative to heating, for increasing dehydration rate and quality of green plums (Paper I, II, III);
2. Investigate the potential of Pulsed Electric Fields (PEF) as a pre-treatment for mitigating acrylamide content in fried potato chips (Paper IV);

3. Investigate the potential of Vacuum Impregnation (VI) for the aromatic enrichment of potato sticks in order to obtain an innovative aromatic processed potato product (Paper V).

2. Minimally processed fruits and vegetables

Fruits and vegetables are essential components in the daily human diet (Linus & Alani, 2010), they are rich in vitamins, fibers, minerals and contain high level of antioxidants such as polyphenols, anthocyanins, carotenoids and flavonoids (Hussein, Caleb, & Opara, 2015). These nutritional and health benefits are related to the reduced risk of many diseases, including cardiovascular diseases, heart disease, ageing and some types of cancer (Allende, Tomas-Barberan, & Gil, 2006; Ramos et al., 2013). The consumption of fresh fruits and vegetables is therefore highly recommended in our daily diet by many international organizations, such as World Health Organization (WHO), Food and Agriculture Organization (FAO), European Food Safety Authority (EFSA), United States Department of Agriculture (USDA) and so on (Allende et al., 2006; Ragaert et al., 2004; Su & Arab, 2006).

Minimally processed fruits and vegetables, originated in the early 1980's, are defined as any fresh fruit or vegetable or combination thereof physically altered from its original form, but remaining in a fresh state which offer consumers high nutritional and convenience value while still maintaining freshness (Manolopoulou & Varzakas, 2015). Due to the modern busy lifestyle, as well as the increasing demand for healthy, fresh-like and easy to prepare products, the consumption of minimally processed fruits and vegetables has remarkably increased in recent years (Rico et al., 2007) and a wide variety of minimally processed fruits and vegetables has been developed in order to conform to consumers' demands (Allende et al., 2006; Froeder et al., 2007), such as ready-to-eat, ready-to-use fruits and vegetables products.

2.1 Minimally processed green plums

Green plum (*Prunus mume*), also known as Wumei in China, Maesil in Korea and Japanese apricot in Japan, is a deciduous tree of the Rosaceae family (Chen et al., 2017). It originates in southern China and has been widely cultivated in Eastern Asian

countries (Gao et al., 2004). The fruit is rich in organic acids, edible fiber, minerals, and phenolic compounds (Gao et al., 2004) and very popular because of its pleasant flavour and odour, wide availability, high nutritional and most importantly, therapeutic benefits (Yan et al., 2014). It has been regarded as both food and medicine material since 1985 by the Ministry of Health of the People's Republic of China because of its therapeutic benefits, which include antitussive, expectoration, antiemetic, antidiarrheal, anthelmintic, and antipyretic activities (Yan et al., 2014). It has been traditionally used as herbal medicine for coughs and dyspepsia (Jung et al., 2010), stomach and intestine disorders (Miyazawa, Yamada, & Utsunomiya, 2003), fatigue, diarrhea, and fever (Shirasaka, Takasaki, & Yoshizumi, 2005).

However, because of the presence of cyanoglycoside, prunasin, amygdalin and high organic acids content, the fruit usually has an unpleasant taste defined as sour, bitter and astringent (Bolarinwa, Orfila, & Morgan, 2014), thus, it needs to be processed before consumption. One of the most popular green plum products on the market in Eastern Asian countries is candied green plum. According to “the white paper on the development of Chinese candied fruit industry in 2016”, the annual output value of candied fruit in China is about $23.5 billion, with an annual average growth rate of 21.78% in the last decade. Candied green plum is one of the main products in this industry.

Osmotic dehydration (OD) is the principal procedure used for obtaining candied green plums, being an effective way to partially remove water from the fruit tissue and increasing its soluble solid content by its immersion in hypertonic aqueous sugar solutions (Nowacka et al., 2014). However, OD is a relatively slow procedure (Souraki, Tondro, & Ghavami, 2015), and the tissue of green plum is innately tight, the traditional process includes the fruits perforation with needles or cutting before the OD procedure (Lin et al., 2013). Even in this case, it takes 5 or more days to saturate them with sugar. To speed up the candying process, heating is therefore a widely applied pre-treatment in industry. However, because it may induce damages to the quality of the final product,

a non-thermal pretreatment able to improve mass transfer could be preferable.

Paper I, II, and III studied the application of high Hydrostatic Pressure (HHP) as a pre-treatment prior to OD of green plums. HHP effectively accelerated the OD of green plums, although not as efficiently as heating (**Paper II**). HPP was shown to promote changes in the water distribution within the tissue related to damages to cell membranes and caused loss of viability at the highest level applied (**Paper I**). Furthermore, HHP pre-treated green plums retained higher overall quality and bioactive compound content when compared to fruits subjected to the traditional thermal treatment.

2.2 Minimally processed potatoes

Potato (*Solanum tuberosum*) is the third largest food crop in the world following rice and wheat (Camire, Kubow, & Donnelly, 2009; Wang et al., 2015). It is an important source of carbohydrates in the human diet (King & Slavin, 2013), also containing a lot of nutrients, including minerals, protein, vitamins, phenolic acids, anthocyanins, and carotenoids in its peel and flesh (Burlingame, Mouillé, & Charrondiere, 2009; Ezekiel et al., 2013; Hamouz et al., 2007; Love & Pavek, 2008). These nutrients play an important role against many chronic diseases, such as atherosclerosis and cancers (McGill, Kurilich, & Davignon, 2013; Williams et al., 2013).

The consumption of potato is very high all over the world, with the average consumption per capita of 26 kg in Asia, 96 kg in Europe and 58 kg in North America (Camire et al., 2009). Thanks to the large amount consumed, potatoes represent the third highest source of total phenolics among fruits and vegetables, listed after orange and apple (Camire et al., 2009; Song et al., 2010).

The industry of minimally processed potato began around 1933, when pre-peeled potatoes were delivered to restaurants and institutional food service distributors in metal containers containing water or brine, and not too long after that, minimally processed potatoes were destined to retail sale (Doan & Davidson, 2000). Nowadays,

they have a huge market share because of the popularity of products such as potato chips, *French fries* and baked potatoes.

An important problem related to potato-based product is the formation of acrylamide during high temperature processing. Acrylamide (C_3H_5NO) is a colourless, odourless processing contaminant in many cooked foods (Riboldi, Vinhas, & Moreira, 2014). The formations of acrylamide in food take place in the Maillard reaction during high temperatures (>120 °C) processing, like cooking, frying, toasting, roasting or baking of foods that are rich in carbohydrates (Tareke et al., 2002). Free asparagine and reducing sugars are the precursors for the formation of acrylamide through deamination and decarboxylation actions, in fact, the carbon skeleton of asparagine is derived entirely from asparagine (Muttucumaru et al., 2017).

Cooked potato products, coffee and cereal products are the major contributors of acrylamide intakes in the adult population of Europe (EFSA Panel on Contaminants in the Food Chain, 2015). It is reported that crisps contribute 0.6% to 6.6% of adult dietary acrylamide intake and other fried potato products account for between 9.6% and 49% in Europe (Dybing et al., 2005; Elmore et al., 2015). It is classified as a Group 2A, meaning it is a probable carcinogen (Muttucumaru et al., 2017) and has caused worldwide concerns (Elmore et al., 2015).

As a result, in 2011 food manufacturers were suggested to take efforts to decrease the acrylamide content in foodstuff to reduce the risk for dietary exposure by the Joint FAO/WHO Expert Committee on Food Additives (WHO, 2011). Recently, the Commission Regulation (EU) 2017/2158 of 20 November 2017 has established a new regulation on “mitigation measures and benchmark levels for the reduction of the presence of acrylamide in foods”, which aims to ensure that food businesses put in place steps to mitigate acrylamide formation (European Commission, 2017).

In **paper IV** the use of Pulsed Electric Fields (PEF) was investigated for the reduction of acrylamide content in potato crisps. The selection of the intensity of the electric field applied and of the further dipping time allowed to obtain a reduction of around 30% of

acrylamide, significantly higher if compared to the reduction obtained with blanching.

Sensorial properties such as flavour and aroma are among the most important factors influencing the purchase decision of minimally processed fruits and vegetables by consumers (Toivonen & Brummell, 2008) and often indicate their shelf life from the point of view of the consumers (Beaulieu & Lea, 2003).

In **Paper V,** Vacuum Impregnation (VI) was used for the enrichment of potato sticks with a rosemary essential oil solution in order to obtain an innovative aromatic minimally processed product. Quality and sensorial properties of the product were evaluated during a refrigerated storage for 14 days.

3 Quality improvement by non-thermal technologies

The deterioration and loss of quality of minimally processed fruits and vegetables occur faster compared to those of the intact whole products, because of the loss of cellular compartmentalization at the cutting surface, the contact between enzymes and substrates and an overall increase of metabolic activity. Further processing, packaging and storage conditions may have ulterior consequences on the tissue physiology, product quality and stability.

Traditional thermal treatments usually cannot be applied in the minimal processing industry, as they will alter the fresh state of the products. The term 'non-thermal processing' is referred to processing technologies that are effective at ambient or slightly higher temperatures. In the last decades, the interest of food scientists, industries and consumers has been attracted by novel non-thermal processing methods aimed at extending shelf life or increasing product functionality with a minimal impact on the nutritional and sensory properties of foods. Moreover, they may help industries in obtaining added-values products, new market opportunities and added safety margins (Morris, Brody, & Wicker, 2007)

During this doctoral research, different emerging non-thermal processing were applied to different products for different purposes including dehydration, acrylamide reduction and aromatic enrichment.

All of the investigated technologies could be coupled with high temperatures to enhance their effect. However, in the present research, temperature was not increased in order to maintain quality and nutritional characteristics of the product intact.

3.1 Dehydration of minimally processed fruits and vegetables

Fruits and vegetables are often dehydrated to enhance storage stability, minimize packaging requirement and reduce transport weight (Sagar and Kumar, 2010). It is

reported that about 20% of continuously growing world fruits and vegetables are dehydrated to increase shelf-life and promote food security (Grabowski et al. 2003). Dehydration is still the most typical postharvest operation for fruits and vegetables throughout the world (Grabowski et al. 2003).

Osmotic dehydration (OD) is one of the commonly applied dehydration processes for fruits and vegetables, being an effective method to develop intermediate moisture products with moisture content of 20–50%, which is imparted by solute gain and water loss (Ahmed et al., 2016). It can be operated at ambient temperature, so heat damage to texture, color, and flavor of food is minimize (Chandra and Kumari, 2015). Besides, it can improve the nutritional and sensorial characteristics of the final products (Tappi et al., 2017) and is a less energy consumption process as compared to other drying techniques (Ahmed et al., 2016). However, OD is a relatively slow process (Souraki et al., 2015), the combination with several new treatments during or before OD have been proposed to further enhance mass transfer.

High Hydrostatic Pressure (HHP) has been proved to be effective to improve mass transfer during OD progress in several fruits such as pineapple, strawberry, banana etc. (Nuñez-Mancilla et al., 2011; Rastogi and Niranjan, 1998; Verma et al., 2014) by damaging the cells structure (Rastogi et al., 2002). The use of OD and HHP for dehydration of minimally processed fruits and vegetables has been discussed in this section.

3.1.1 Osmotic dehydration (OD)

OD is widely applied for horticultural products to reduce the water content while increasing soluble solid content (Kaymak-Ertekin & Sultanoğlu, 2000), being an effective way to partially remove water from fruit tissue by its immersion in hypertonic aqueous sugar solutions (Nowacka et al., 2014). The final products still have relatively high moisture content (20–50%), hence they are classified as intermediate moisture

foods (IMFs) (Panagiotou, Karathanos, & Maroulis, 1998).

The driving force of the OD process is the osmotic pressure differences across cell membranes (Fernandes, Gallão, & Rodrigues, 2009). The semi-permeable cell membrane only allows the movement of low molecular molecules, such as water or sucrose. The reduction of water content up to 50% of the food material significantly decrease the water activity (Bekele & Ramaswamy, 2010) improving the microbial stability with little energy consumption (Sagar & Kumar, 2010). Moreover, a counter flux of solutes can occur from the solution into fruits tissue and can be exploited to improve the nutritional and sensorial characteristics of the final products (Chandra & Kumari, 2015; Tappi et al., 2017; Yadav & Singh, 2014). However, this process is slow, up to several hours and even days, depending on the cell membrane permeability and cell architecture (Amami et al., 2007).

3.1.1.1 Mechanism of osmotic dehydration

When food materials are immersed in a hypertonic solution, a multi-component transfer process occurs, driven by the osmotic pressure differences, resulting in drying, leaching and impregnation of the food materials.

The rate of water loss is higher in the early stage of OD and decreases along the process (Kowalska & Lenart, 2001; Santagapita et al., 2013; Tylewicz et al., 2011). This behaviour is due to the higher difference in the osmotic pressure between the food materials and the hypertonic surrounding medium at the beginning. The same trend is also found for the diffusion of the solutes into the food tissue, although the higher solids content at the beginning is mainly due to the loss of moisture (Phisut, 2012).

After the water reaches the outside of the cell, it moves to the surrounding spaces mainly by diffusion and capillary flow in porous materials (Shi & Xue, 2008), such as fruits and vegetables. The cavities, capillaries and cell walls, as well as intracellular and extracellular spaces provide the pathways for the mass transfer (Shi & Le Maguer,

2002). The cell membrane plays a dominant role in the resistance of mass transfer phenomenon between the biological materials and the surrounding environment (Rastogi & Raghavarao, 2004). The mass transfer phenomenon during OD process is represented in Figure 1.

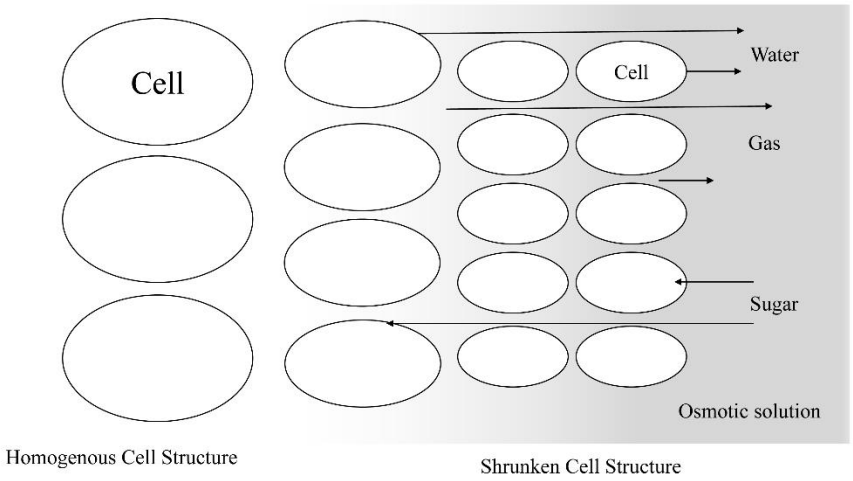

Figure 1. mass transfer phenomenon during OD process (adapted from Shi and Xue, 2008)

In many early studies, the state of the cell membrane was considered unchanged during the OD process (Rastogi et al., 2002); therefore, the diffusion coefficient of water was regarded as a constant throughout the process. However, as shown in Figure 2, after the early stage of the OD process, the physical structure of cell membranes starts to change, leading to cell membrane disintegration in the dehydrated region. With the movement of the dehydration front (Δx) into the center of the material, water is diffused across three different regions with different characteristic properties: diffusion of water from the core of the material to the dehydration front (D_1), diffusion of water across the front (D_2) and diffusion of water through the osmotically treated material into the surrounding medium (D_3).

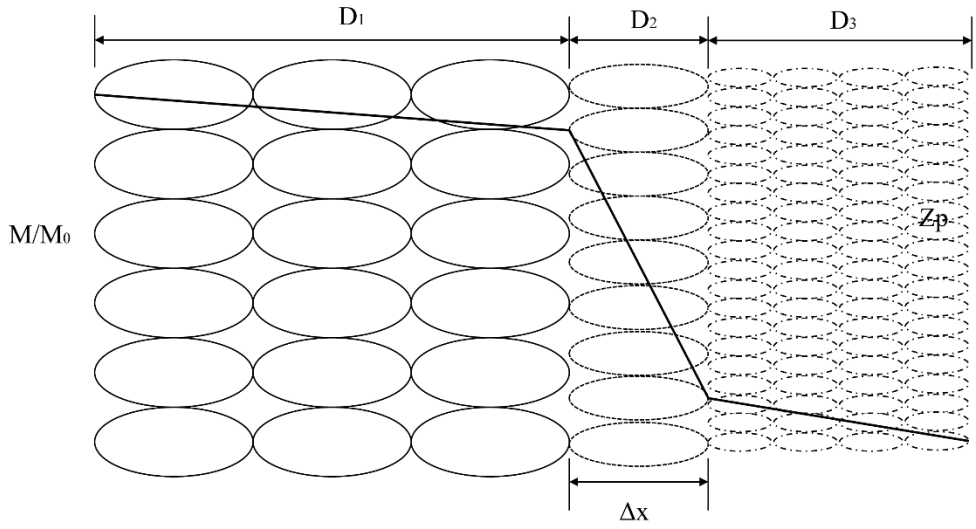

Figure 2. Mechanism of osmotic dehydration of biological material. Zp: cell disintegration index; M/M0: relative moisture content; D_1, D_2 and D_3: the diffusion coefficients of water from the core of the material to the dehydration front, across the front and through the osmotically treated material into the osmotic solution, respectively; Δx: thickness of moving dehydration front. (Figure adapted from Rastogi, Angersbach, and Knorr, 2000a).

After immersing a food material into a hypertonic solution, the cells in the layer directly in contact with the hypertonic solution begin to lose water because of the concentration gradient, leading to the shrinkage of the cells, and to the increase of the concentration of cells in this layer. As the osmotic pressure reaches a critical value, the cell membranes rupture; the diffusion coefficient of water from these ruptured cells is D_3.

As the osmotic dehydration proceeds, the cells behind the first layer start to lose water and shrink, with a diffusion coefficient of D_2. The cells in the center of the material are still intact, the diffusion coefficient (D_1) of water from these cells is much lower than D_2 and D_3.

3.1.1.2 Kinetics of osmotic dehydration

Different models have been proposed to monitor and predict the mass transfer kinetics during OD process. Generally, these models can be classified as empirical and semi-

empirical, mechanistic and phenomenological based on different theories (Assis, Morais, & Morais, 2016).

Among empirical and semi-empirical models, Peleg's equation (Peleg, 1988) is one of the most commonly used and it's based on the experimental data of water loss (WL) and solids gain (SG). It includes multivariable regressions, response surface analysis and mass balances with the advantage to estimate the initial rate and the equilibrium values of mass transfer. However, like other empirical and semi-empirical models, it does not take into account the underlying simultaneous transport phenomena of water and solutes in the process (Herman-Lara et al., 2013; Mercali et al., 2011; Sereno, Moreira, & Martinez, 2001).

On the contrary, phenomenological models take into account the phenomenological mechanisms and the shape of samples in the process. Among many phenomenological models, the most used one was proposed by Crank (1979), which is based on the Fick's second law and includes a series of mathematical solutions to describe the diffusional phenomenon during OD process.

1) *Peleg's model*

Palou et al. (1994) modified Peleg's model (Peleg, 1988) as Equation (1):

$$SG \text{ or } WL = \frac{t}{k_1 + k_2 \times t} \qquad (1)$$

k_1 and k_2 are the Peleg's constants. Solids Gain (SG) and Water Loss (WL) can be determined through the following equations (Corrêa et al., 2010):

$$SG = \frac{w_s M_t - w_{s0} M_0}{M_0} \qquad (2)$$

$$WL = \frac{w_{w0} M_0 - w_w M_t}{M_0} \qquad (3)$$

Where, w_s is the solids content at time t, M_t is the weight in time t, w_{s0} is the initial solids content of sample, M_0 is initial weight, w_{w0} is the initial moisture content, and w_w is the moisture content at time t, all expressed in g.

The constant k_1 relates to the initial rate of the mass transfer, and the constant k_2 relates to equilibrium values, as can be calculated below:

$$\left[\frac{d(SG\ or\ WL)}{dt}\right]_{t\to 0} = \lim_{t\to 0}\left[\frac{d\left(\frac{t}{k_1+k_2\times t}\right)}{dt}\right] = \frac{1}{k_1} \qquad (4)$$

$$\lim_{t\to\infty}(SG\ or\ WL) = \lim_{t\to\infty}\frac{t}{k_1+k_2\times t} = \frac{1}{k_2} \qquad (5)$$

This kinetic model offers the advantage that by calculating the inverse of the constant (k_1 and k_2) it is possible to obtain the initial rate and the values at the equilibrium of mass transfer parameters (Sacchetti, Gianotti, & Dalla Rosa, 2001).

2) Crank's model

For plant materials with high moisture content, it is often assumed that mass diffusion is dominated by external conditions of mass transfer (Sinha et al., 2010). Crank provided Equation (6) to calculate the effective diffusivity (D_{eff}) for various regular geometry materials (rectangular, cylindrical, and spherical):

$$\frac{\partial X}{\partial t} = D_{eff}\frac{\partial^2 X}{\partial z^2} \qquad (6)$$

Where X is the amount of water or solids at instant, t is time (s), D_{eff} is effective diffusivity (m^2/s) and z is a generic directional coordinate.

With the appropriate initial and boundary conditions, the equation can be modified as Equation (7) for finite cylinder samples:

$$W_{w\ or\ s} = \frac{4}{f}\cdot\frac{8}{\pi^2}\sum_{i=0}^{\infty}\frac{1}{(2i+1)^2}exp\left[-(2i+1)^2\left(\frac{f}{r^2}+\frac{\pi^2}{4l^2}\right)\cdot D_{eff}\cdot t\right] \qquad (7)$$

Where, f depends on shape of sample, for cylinder $f = 5.783$, i is the number of series terms, r is the radius, l is the characteristic length (m), and $W_{w\ or\ s}$ is the dimensionless water or solid content, which can be calculated by Equation (8):

$$W_{w\ or\ s} = \frac{M-M_e}{M_0-M_e} \qquad (8)$$

Where M is moisture or solids content at time t, M_0 is initial moisture or solids content and M_e is equilibrium moisture or solids content which is measured after 6 h of OD.

For long periods of OD process, when the Fick number, $(\frac{f}{r^2}+\frac{\pi^2}{4l^2})\cdot D_{eff}\cdot t$ is less than 0.1 or $W_{w\,or\,s}$ is less than 0.6, only the first term of the series terms is used (Sinha et al., 2010). Although it is less rigorous in the calculation, it is a good approximation of reality ($i = 0$) (Simpson et al., 2015). Equation (7) could be simplified as:

$$\mathrm{W}_{w\,or\,s}=\frac{4}{f}\cdot\frac{8}{\pi^2}exp(-(\frac{f}{r^2}+\frac{\pi^2}{4l^2})\cdot D_{eff}\cdot t) \qquad (9)$$

Take natural logarithms on both sides of the equation, it becomes:

$$ln(\mathrm{W}_{w\,or\,s})=ln\left(\frac{4}{f}\right)+ln\left(\frac{8}{\pi^2}\right)-\left(\frac{f}{r^2}+\frac{\pi^2}{4l^2}\right)\cdot D_{eff}\cdot t \qquad (10)$$

The effective diffusivity D_{eff} could be calculated by equation (10) by plotting $ln(W_{w\,or\,s})$ curve versus time t.

3.1.2 High hydrostatic pressure (HHP)

High Hydrostatic Pressure (HHP) is one of the most successfully commercialized non-thermal processing technology in the food industry (Huang et al., 2017). It has been applied on industrial scale to inactivate microorganisms and enzymatic activities with the objective to improve the stability of the products during storage while avoiding the degradation of nutritional and organoleptic properties of products (Deliza et al., 2005; McInerney et al., 2007).

Moreover, when compared with traditional thermal treatments, HHP requires lower energy. For example, the increase of pressure up to 500 MPa requires lower energy consumption than heating to 100 °C (Pereira & Vicente, 2010). The average cost of HPP is approximately 0.05–0.5 US$ per liter or kilogram depending on the processing conditions and is lower than the cost of thermal processing (Bermúdez-Aguirre &

Barbosa-Cánovas, 2011). The main cost involved is the equipment, which has decreased considerably in recent years due to the wide application of this technology (Wang et al., 2016).

Apart from the application on microbial and enzymatic inactivation, HHP can also be applied to accelerate mass transfer processes on account of the permeabilization of the cell structure due to cell disintegration (Eshtiaghi, Stute, & Knorr, 1994; Farr, 1990; Rastogi, Angersbach, & Knorr, 2000b).

3.1.2.1 HHP pre-treatment to improve dehydration rate

The application of HHP (100–800 MPa) can damage the cell membranes and increase the cell disintegration index (Zp), causing permeabilization and disruption of cell structure. For this reason, HHP can be used as a pretreatment prior to OD in order to improve the mass transfer rate (Núñez-Mancilla et al., 2014).

Verma, Kaushik, and Rao (2014) applied HHP (100–500 MPa) as a pretreatment on banana slices and found a remarkable increased of water loss and solids gain during OD process, caused by the rupture of cell structure evidenced by scanning electron micrographs of the banana tissue. George, Selvan, and Rastogi (2016) obtained similar results, finding higher moisture and solid transfer in osmo-dehydrated apples pre-treated with HPP (50-350 MPa). Cell permeabilization was revealed by microstructure analysis. Likewise, Rastogi and Niranjan (1998) showed that HHP pretreatment (100–700 MPa for 5 min at 15–35 °C) significantly enhanced the mass transfer rates of pineapples during OD, resulting in significant removal of water.

Results relative to the effect of the increased working pressure of HHP pre-treatment on the mass transfer during OD are inconsistent in the literature. Nuñez-Mancilla et al. (2013) found that at the range of applied pressure (100-500 MPa), there was no significant difference in the moisture content of strawberries after OD process. Dermesonlouoglou, Boulekou, and Taoukis (2008) found that increasing the pressure

from 200 MPa to 600 MPa did not increase the solids gain and water loss of cherry tomatoes osmotically dehydrated for 6 h. On the other side, in other studies, an increment in the SG and/or WL of candied plant materials during OD pretreated with HHP was observed by increasing the working pressure (Rastogi et al., 2000a; Rastogi et al., 2000; Rastogi & Niranjan, 1998; Verma et al., 2014).

Paper I evaluated the effects of High Hydrostatic Pressure (HHP) pre-treatment (50-400 MPa) on the mass transfer kinetics and on the water diffusivity of green plums fruit during OD and investigated the effect on water distribution and cell viability aspects. The results of **Paper II** showed that HHP increased initial rate and effective diffusivity of mass transfer values compared to non-treated samples. However, increasing pressure from 50 to 400 MPa and time from 1 to 30 min did not increase significantly the mass transfer rate.

Time Domain Nuclear Magnetic Resonance (TD-NMR) revealed that, upon HHP treatment, the water redistributed in vacuole, cytoplasm/extracellular spaces and cell wall/membrane. The application of 400 MPa probably caused some irreversible damages to the cell membranes the cell viability study determined by fluorescein diacetate (FDA) staining showed a loss of cell viability at pressures higher than 200 MPa.

3.1.2.2 Influence of HHP on physico–chemical properties of fruit and vegetables

HHP treatment has a limited effect on the pigments of fruits and vegetables (Oey et al., 2008), however, immediately after the HHP treatment, samples usually present a deeper and darker colour mainly due to the damage to the cell membranes, which causes the leakage of cellular content and then lead to a variation of the refractive index of the tissue. For example, Nuñez-Mancilla et al. (2013) found an increment of non-enzymatic browning of strawberry pre-treated by HHP during OD process. Similarly, Krebbers et al. (2002) observed remarkable changes in greenness of green beans by HHP treatment.

However, according to other authors, minimal colour changes are observed after HHP treatment (Ahmed & Ramaswamy, 2006; Ahmed, Ramaswamy, & Hiremath, 2005; Butz et al., 2003). These discrepancies are probably due to the different applied pressure and to the structural characteristics of the treated products.

Regarding the effects of HHP on texture of fruits and vegetables, Basak and Ramaswamy (1998) observed a rapid loss of firmness of fruits and vegetables such as apple, pear, orange, pineapple, carrot, celery, green pepper and red pepper upon HHP treatment (100–400 MPa/5–60 min), caused by the contact between pectinmethylesterase and its substrates. A similar result was observed for cherry tomatoes (Tangwongchai, Ledward, & Ames, 2000), however, the influence of HHP on texture is minimal when compared with traditional thermal treatments (De Roeck et al., 2008).

A positive effect have been observed for flavour attributes of fruits and vegetables. Navarro et al. (2002) observed a higher hexanal content on strawberry purée after HHP treatment (400 MPa for 20 min at room temperature). A 40% increase in hexanal concentration was detected after 300 MPa for 30 min treatment (25 °C). An enhanced volatile compound concentrations was found in mulberry and carrot juices after HHP treatment in comparison with the control (Wang et al., 2017; García et al., 2001).

Paper II reported that green plums pre-treated with HHP (50 MPa for 1 min) were charcaterized by higher titratable acid content, firmness and greener colour compared samples subjected to heating. Moreover, in **Paper III** it was observed that HPP promoted the release of various volatile components resulting in a richer volatile profile compared to both un-treated and heat-treated samples. Sensory results confirmed that HHP allowed to better preserve the typical odor and flavour of the final products.

3.1.2.3 Effect of HHP on nutritional properties of fruit and vegetables

The nutritional composition of osmotic dehydrated product can be influenced by HHP pretreatment (Ahmed, Qazi, & Jamal, 2016; Núñez-Mancilla et al., 2014). It is generally assumed that these changes are usually not directly altered by HHP processing, but caused by the disruption of cell structure, which induces the leakage of cellular content, and promotes undesirable enzymatic or non-enzymatic reactions due to the increased contact between enzymes and their substrates, and/or the oxidation actions by the oxygen in atmosphere.

Many researchers detected an increased content of bioactive compounds, such as ascorbic acid, phenols, flavonoid and anthocyanin, after HHP treatment (Corrales et al., 2008; Keenan et al., 2010; Patras et al., 2009). The authors attributed the increment to the higher extraction rate caused by rupture of cell structures, rather than a real increment in the fuits tissue.

Paper III investigated the antioxidant content and activity in osmotically dehydrated green plums subjected to HPP and thermal pre-treatments. The application of 50 MPa for 1 min promoted a higher retention of antioxidant compounds (ascorbic acid, phenolics and flavonoids content) and activity compared to the heating treatment.

3.2 Acrylamide reduction

Acrylamide (C_3H_5NO) is a harmful contaminant classified as a Group 2A, 'probably carcinogenic to humans' carcinogen and has caused worldwide concerns (International Agency for Research on Cancer, 2014). Its presence has been reported in a range of fried and oven-cooked foods (Elias et al., 2017), and it is commonly generated during heat treatment as a result of the Maillard reaction between amino acids and reducing sugars (Muttucumaru et al., 2015). Asparagine, a major amino acid in potatoes and cereals, is reported as a crucial participant in the production of acrylamide (Mottram et

al., 2002). Fried potato contributes to a substantial proportion of the estimated intake of acrylamide in the European adult population (European Food Safety Authority, 2015). It is important to decease the acrylamide content in fried potato products.

Several investigations have proposed mitigation ways to be applied at different stages of the manufacturing process of fried potato products, in order to reduce the concentration of acrylamide precursors. Among conventional mitigation strategies, hot water blanching of potato slices appeared to facilitate the extraction of Maillard reaction substrates, in addition to enzyme inactivation, also improving the colour uniformity and texture, and oil absorption reduction (Mestdagh et al., 2008; Pedreschi et al., 2004). However, this thermal pre-treatment presents the drawbacks of being time-intensive, high energy-consuming and of promoting considerable modifications of sensorial properties of final product. A non-thermal pretreatment able to reduce the acrylamide content could be preferable.

Pulsed electric fields (PEF), a technology based on the permeabilization of cell membranes, may conform to this requirement by increasing the release of sugars and amino acids that represent the main substrates for the Maillard reaction (Jaeger et al., 2010).

3.2.1 Pulsed Electric Field (PEF)

Pulsed electric field (PEF) is an innovative non-thermal technique which has many different applications in food and pharmaceutical industry (Yan, He, & Xi, 2017). It has gained interests in recent years from industries and researchers because of its low energy consumption (Boussetta et al., 2013). Depending on the applied voltages, it can effectively inactivate most micro-organisms and some enzymes at room temperatures, improving the stability and quality of food and medicinal material during storage (Boulaaba et al., 2014; Puértolas et al., 2010; Wu et al., 2014; Zhao et al., 2009), or improve the extraction rate during processing (Yan et al., 2017), as well as to enhance

mass transfer processes such as drying and frying in different fruits and vegetables (Amami et al., 2008; Donsì, Ferrari, & Pataro, 2010; Janositz, Noack, & Knorr, 2011; Palgan et al., 2012; Traffano-Schiffo et al., 2016).

In the present research, PEF was investigated only for the treatment of solid foods, exploiting its ability to improve mass transfer in potato slices during a further dipping.

3.2.1.1 Mechanisms of PEF

PEF is considered as a very effective treatment to cause the permeabilization of cell membrane through a process known as electroporation that leads to the inactivation of microbe or plant cells (Donsì et al., 2010). PEF treatment is based on the generation of short duration pulses (μs to ms) of high voltage electric fields between two electrodes, that can alter the transmembrane potential causing temporary or permanent permeabilization of cell membranes (Zimmermann, 1986).

The mechanisms of damage on cell membrane by PEF are explained by the electroporation effect (He et al., 2014; Yin et al., 2007), that is shown in Figure 3 (Donsì et al., 2010).

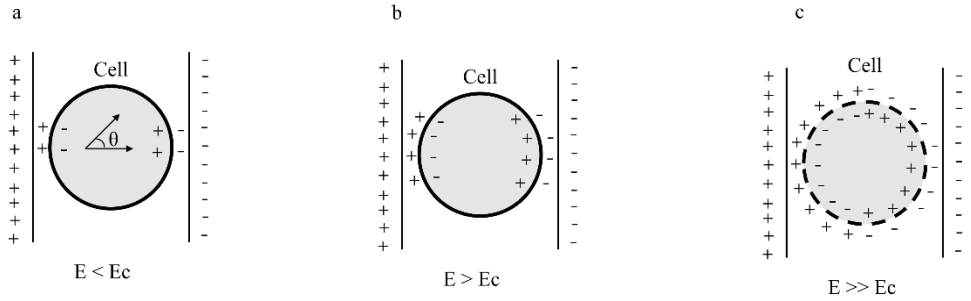

Figure 3. reversible or irreversible damages on the membrane for biological cell. E: electric field intensity; Ec: critical electric field intensity for permeabilization of cell membrane; r: radius of cell; θ: the angle between the site on the membrane and the direction of the electric field. Dashed line of the cell membrane means electroporated area. Figure adapted from Donsì et al. (2010).

When the cell is exposed to high intensity electric field pulses, the formation of pores

occurs in the lipid bilayer of cell membrane (Castro, Barbosa-Canovas, & Swanson, 1993).

Several theories based on model systems have been proposed in order to explain the reversible or irreversible damages on cell membrane (Chang & Reese, 1990; Kinosita et al., 1991; Zimmermann, 1986). One of the common facts in these theories is that the membrane will amplify the applied electric field, because the conductivity of membrane is much lower than the one of extra cellular medium and cell cytoplasm (Weaver & Chizmadzhev, 1996). As the example shown in Figure 3, when a spherical shaped biological cell is placed in electric field, the transmembrane potential increases because of the charging process at the membrane interfaces (Donsì et al., 2010). The increased transmembrane potential can be estimated by Equation (11) based on Maxwell's equation assuming several simplifying restrictions (Neumann, 1996):

$$u_m = 1.5r \cdot E \cdot \cos\theta \qquad (11)$$

Where, u_m is increased transmembrane potential, r is radius of cell, E is the applied external electric field and θ is the angle between the site on the membrane and the direction of the electric field.

According to Equation (11), the highest electric field on cell membrane is reached at θ = 0 or π, while the lowest electric field at θ = ±π/2. Because the membrane thickness (about 5 nm) is much lower than plant cell radius (about 100 μm), the transmembrane electric field (E_m) at different sites on membrane will be created when an extra electric field is applied (Vorobiev & Lebovka, 2009; Weaver & Chizmadzhev, 1996) according to Equation (12):

$$E_m = \frac{u_m}{h} \qquad (12)$$

Where, h is the thinness of the cell.

The transmembrane electric field can be as much as 10^5 times higher than the applied field strength (Donsì et al., 2010). Therefore, a relatively low critical value of the applied field strength E_c (0.2–1.0 V) can cause a strong transmembrane electric field

which lead to reversible or irreversible damages on the membrane for most plant cells. The cell disintegration index, (Zp) can be calculated by electro-physical measurement based on electrical impedance analysis (Knorr & Angersbach, 1998).

If the applied electric field is lower or close to the critical value E_c, or only a few pulses are applied, the membrane damage is reversible as the cell can recover its structure and functionality. On the contrary, if the applied electric field is much larger than critical value E_c, irreversible breakdown occurs causing perpetual mechanical destruction of the cell membrane, and further inactivation of cells. Determination of cell viability before and after PEF is therefore often used to observe the occurrence of irreversible damages to the cell membrane (Janositz & Knorr, 2010).

The whole PEF process consists in the charging and polarization of the membranes, creation of pores, expansion and aggregation of the pores, and resealing of the pores (Kandušer & Miklavčič, 2009; Vorobiev & Lebovka, 2009). The formation and development of pores during and after the treatment is a dynamic process (Angersbach, Heinz, & Knorr, 2002; Donsì et al., 2010).

In **Paper IV**, Zp was calculated after the application of a 1.5 kV treatment on whole tubers or on slices. Results showed that higher surface dimension exposed to electric pulses, being related to slices, resulted in higher cell disintegration. Following these results, the setup of the experimental plan for acrylamide reduction in fried potato crisps was designed applying the different pre-treatments on raw potatoes directly after slicing.

3.2.1.2 Influence of PEF on physico–chemical properties

A review of the literature shows that PEF (0.4-2 kV/cm for milliseconds or seconds) promotes softening of plant tissue such as carrots, apples and potatoes (Boussetta et al., 2013; De Vito et al., 2008; Lebovka, Praporscic, & Vorobiev, 2004) mainly due to the disruption of cell membrane as the observed tissue softening is often associated with the disintegration index Zp.

The influence of PEF treatment on colour of plant tissue can be ambiguous (Wiktor et al., 2015). On one hand, the damage on cell membrane causes the leakage of intracellular content, increasing the possibility of the contact between enzymes and their substrates, further promoting the enzymatic browning, as observed by Grimi et al. (2010) for PEF treatment of 100–400 V/cm for 100 ms of apple tissue. Wiktor et al. (2015) also observed a browning effect on PEF treated apple (3 and 5 kV/cm for 100 pluses). On the other hand, PEF may promote the inactivation of enzymes responsible for tissue browning (Ohshima, Tamura, & Sato, 2007), however only at high voltages not suitable for the treatment of solid foods. For example, the application of 20 to 40 kV/cm for 200 μs was able to avoid colour changes on green tea extract (Zhao et al., 2009) Likewise, Odriozola-Serrano et al. (2009) observed that a 35 kV/cm PEF treatment for 1500 μs did not cause a noticeable colour change on carrot juices.

As a non-thermal treatment, the influence of PEF treatment on bioactive compounds such as vitamins and antioxidants in fruits and vegetables is limited (Vega-Mercado et al., 2004). But with the disruption of cell membranes, PEF increases the extraction of bioactive compounds. For instance, Vallverdú-Queralt et al. (2013) observed a great increment of polyphenol and carotenoid contents of tomato fruit after moderate-intensity pulsed electric field (0.4 to 2.0 kV/cm for 5 to 30 pluses).

Moreover, PEF application to fruits and vegetables seems to be able to increase the flavour of the final products, because of the rupture of cell structure. Vallverdú-Queralt et al. (2013) reported that PEF treatment (1 kV/cm using 16 monopolar pulses of 4 μs at a frequency of 0.1 Hz) increased the detected concentration of hexanal and (E)-2-hexenal flavour compounds from tomato.

3.2.1.3 Application of PEF to potato products

The application of PEF for potato snacks pre-treatment is under investigation, and many researchers have already reported high numbers of benefits that could be achieved by

applying electric pulses to raw potatoes.

For example, Jalte et al. (2009) detected the effects of PEF pre-treatment (400 V/cm for 10^{-4} to 0.3 s) on the freezing, freeze-drying and rehydration behaviour of potato samples, and found that PEF pre-treatment reduced freezing time and improved the freeze-drying rate and the quality of rehydrated samples. The increment of drying rate of potato samples pre-treated by PEF was also observed by some other studies (500 to 1500 V/cm for 0.1 to 1 s) (Arevalo et al., 2004; Lebovka, Shynkaryk, & Vorobiev, 2007).

Furthermore, Fauster et al. (2018) have recently described the impact of PEF treatment (1.0 kV/cm on a continuous flow system of 15–16 t/h) on potato structure and various potential advantages on quality and economic aspects of industrial *French fries* production, including reductions of cutting force, starch loss and oil uptake. Similar results were found by Janositz et al. (2011), who reported a reduction of fat uptake of PEF treated potato slices (1.8 kV/cm, n = 40) of 38.66% compared to untreated samples. Likewise, lower fat uptake was also observed by Ignat et al. (2015). The application of 9000 pulses at 0.75 kV/cm electric field or 810 pulses at 2.50 kV/cm on potato cubes promoted an increase of the moisture content and a softening of samples.

Moreover, PEF treatment of raw potatoes has been suggested a possible strategy for increasing the release of reducing sugars and amino acids that represent the main substrates for the Maillard reaction (Jaeger, Janositz, & Knorr, 2010; Janositz et al., 2011). A lighter colour of deep-fat fried potatoes after PEF pre-treatment was observed by Janositz et al. (2011) and by Ignat et al. (2015), but the actual content of acrylamide has never been investigated before.

Paper IV represents a first study of the reduction of acrylamide in fried potato crisps as a consequence of PEF pre-treatment. For this aim, a PEF pre-treatment (1.5 kV/cm, pulse duration 10 µs, total treatment time 10 s, pulse frequency 100 Hz) was applied before dipping and subsequent frying with the aim of mitigating the acrylamide content, considering various factors such as the shape of treated potato samples and the washing

time after PEF. Results showed that PEF allowed a reduction of acrylamide content in potato crisps of around 30%, significantly higher if compared to the reduction obtained with blanching, with only slight modifications of the final quality of the product, in terms of colour and texture.

3.3 Aroma enrichment

The odor and flavor attributes are important factors influencing the purchasing motivation of consumers (Ragaert et al., 2004), apart from the appearance characters of fresh cut fruits and vegetables, which has already been studied extensively.

The enrichment of fruits and vegetables with an external aroma could be an interesting opportunity for creating innovative and attractive products for both consumers and industries. Vacuum impregnation (VI) is an innovative technology that has been recognized as a promising tool for the introduction of solutes into the internal structure of some porous food products, due to the action of hydrodynamic mechanisms promoted by pressure changes (Tylewicz et al., 2012).

3.3.1 Vacuum Impregnation (VI)

Fruits and vegetables have a great amount of intercellular space which is occupied by gas and offer the possibility to be impregnated by external solutions (Pedro et al., 2001). VI is an operation frequently applied as a pretreatment, for example, in minimal processing, before drying, freezing or production of fruit and vegetable preserves.

The size of pores inside plant tissue plays an important role to restrict the passage of external molecules. The pore size in the walls of living plant cells through which molecules can freely pass is very small, ranging between 35 to 52 angstroms in different plant tissues (Carpita et al., 1979). However, the pores in intercellular spaces can be much larger, for instance, the diameter of intercellular spaces in mature apple tissue can

be 50–500 μm (Lapsley, Escher, & Hoehn, 1992). Therefore, many functional compounds, including large molecules and even microorganisms can be introduced into the intracellular spaces of plant tissue, inducing various functional advantages for food materials, such as nutritional enrichment (e.g. polyphenols, minerals, vitamins, probiotics and micronutrients) (Allali, Marchal, & Vorobiev, 2010; Kazunori Hironaka et al., 2011; Hironaka, Oda, & Koaze, 2014), extension of shelf life (e. g. anti-browning agents, microbial preservatives) (Panarese et al., 2014), and improvement of sensory attributes (e. g. sugar) (Allali et al., 2010).

3.3.1.1 Mechanism of Vacuum Impregnation

During VI the intracellular spaces of food materials are filled by an external solution based on mechanisms promoted by pressure changes. The process consists in two main steps:

- In the first step, the pressure is reduced in the system, gases in the product pores expand and partially flows out until mechanical equilibrium is achieved.
- In the second step, the atmospheric pressure is restored, residual gas in the pores compresses and the external solutions fill in the pores. Hydrodynamic mechanism (HDM) and deformation-relaxation phenomena (DRP) take place during the VI process, leading to the flow of external solutions into intracellular spaces of food materials.

The mechanism is shown in detail in Figure 4.

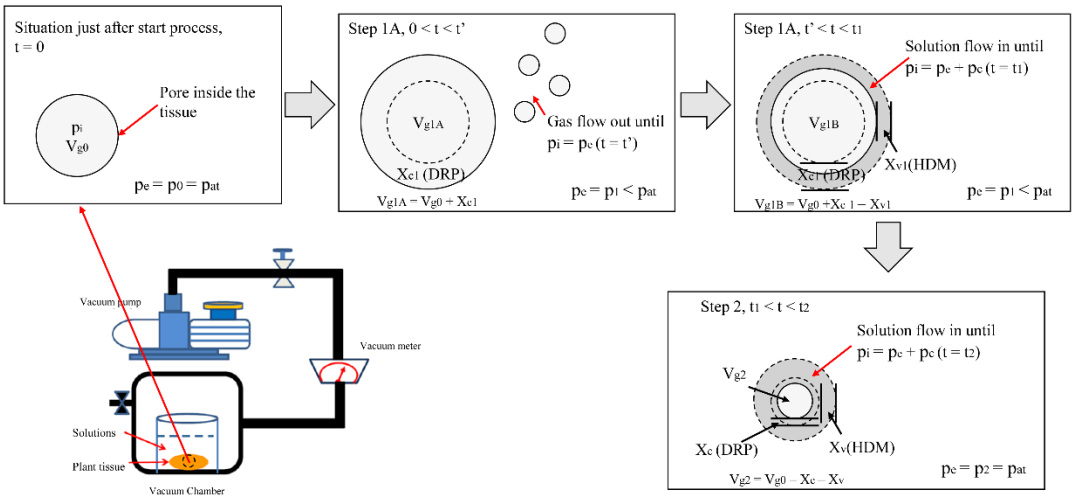

Figure 4. The whole processes of VI, adapted from Fito et al. (1996). (t: Time; t': Time when the pressure inside and outside food material become equal; t_1: Applied vacuum time; t_2: Applied relaxation time; p_0: Initial pressure; p_1: Applied vacuum pressure; p_2: Applied final pressure; p_i: Inside pressure; p_e: outside pressure; p_c: Capillary pressure; p_{at}: Atmospheric pressure; Vg_0: Initial volume of the pores; Vg_{1A}: Volume of pores in Step 1A; Vg_{1B}: Volume of pores in Step 1B; Vg_2—Volume of pores in Step 2; Xc_1—Increment of volume of pores as result of DRP; Xc—Decrement of volume of pores as result of DRP; Xv_1—decrement of volume of pores as result of HDM; Xv—Decrement of volume of pores as result of HDM).

Before the application of vacuum (t_0), the pressure inside (p_i) and outside (p_e) the food material is equal to the atmospheric pressure ($p_i = p_e = p_{at}$). The initial volume of pores inside the food materials (Vg_0) is filled with gas. When the vacuum is applied in the system (Figure 1, Step 1A and Step 1B), the pressure in the system (p_1) is decreased until it is equal to the external pressure (p_e), and lower than the pressure inside the food materials ($p_1 = p_e < p_i$). Because of the pressure changes, the gas in the pores expands and flows out, leading to the increment of the volume of the pores ($Vg_{1A} = Vg_0 + Xc_1$). This is the first part of the deformation–relaxation phenomenon (DRP), which lasts until the pressure inside (p_i) reaches the pressure outside (p_e) (Figure 1, Step 1A). Subsequently, during the vacuum time, the solutions start to fill in the pores, forced by the hydrodynamic mechanism (HDM), leading to the decrease of the volume inside the food materials ($Vg_{1B} = Vg_0 + Xc_1 - Xv_1$) and causing the increase of pressure inside (p_i

= $p_e + p_c = p_1 + p_c$) (Figure 1, Step 1B). In the second step of VI (Figure 1, Step 2), the relaxation phase, the pressure outside (p_e) returns to atmospheric pressure ($p_e = p_{at}$) and is higher than the internal pressure ($p_i < p_e$), the pores inside the food material start to shrink and, as a consequence of the capillary pressure and compression, an inflow of liquid from the outside is observed. At the end of the process, the capillary volume is reduced compared to the initial one because of the deformation–relaxation phenomenon (DRP) ($Vg_2 = Vg_0 - X_c - X_v$). The inflow continues until the inside pressure reaches the equilibrium ($p_i = p_e + p_c$).

The capillary pressure is affected by pore size, surface tension of the solution and wetting angle between the liquid and the pore walls. The pressure changes in this step also promote deformation of food because of the viscoelastic properties of the materials (Tylewicz et al., 2012).

The relaxation phase is particularly important from the practical point of view, because most of the impregnation takes place during this step. The application of vacuum should not be too fast, because the excessively rapid pressure decrease may cause closure of the capillary vessels and further inhibit the HDM (Fito et al., 1996; Salvatori et al., 1999)

The efficiency of VI can be affected by many factors that are related to the food material intrinsic characteristics or to processing parameters. Regarding the first, the capacity of impregnation induced by vacuum is limited mainly by the porosity of the food materials. Other factors include the shape and the size of the sample, the mechanical properties and the shape and size of the pores and capillary vessels. Among processing parameters, important factors include the value of applied vacuum pressure, vacuum time, the speed of the decompression process, the ratio of solutions to samples, composition, concentration and surface tension of the solution, temperature and the restoration of atmospheric pressure (Fito et al., 1996; Gras et al., 2003; Zhao & Xie, 2004).

3.3.1.2 Influence of VI on physico–chemical properties and sensory attributes

Fruits and vegetables have a great amount of intercellular spaces which are occupied by gas (Pedro et al., 2001). The filling of these intercellular spaces with external solutions may significantly change the physico–chemical properties and sensory attributes of the final products.

The use of solutions characterized by a specific colour, such as fruit juices, has a great influence on the visual quality of the final product as observed by Betoret et al. (2012) and Castagnini et al. (2015). However, VI was observed to promote changes in the colour of the samples even when using an uncoloured solution (Muntada et al., 1998; Tapia et al., 2003). The observed variation was due to the variation of the refractive index of the tissue as a result of the filling of the intercellular spaces with the impregnating solution, leading to a reduced lightless value (L* value). A similar result was reported in **Paper V**.

Besides, because of the reduction of the presence of oxygen in the tissue, VI may affect sample colour by decreasing the enzymatic browning of plant tissue during storage (Tylewicz et al., 2013; Zhao & Xie, 2004; Perez-Cabrera et al., 2011).

The filling of the intercellular spaces with an external solution has been observed to reduce the respiration rate of the tissue because of the reduced presence of oxygen. However, results about the influence of the application of VI on the respiration rate of plant materials were inconsistent in the literature. Castelló, Fito, and Chiralt (2006) compared the respiration rate of VI or non-VI treated strawberry, and showed that VI process (5 min at 5 kPa and 5 min at atmosphere pressure) did not change the respiration behavior in terms of O_2 consumption but promoted the onset of an anaerobic pathway as shown by the higher CO_2 production detected in VI treated sample, this being more notable when temperature increased (from 1 to 20 ºC). They attributed this phenomenon to the mechanical damage by pressure changes and further alterations in metabolic routes. However, Igual et al. (2008) observed a significantly decrease of respiration rate in terms of O_2, but the same production level of CO_2 of cut persimmon samples treated

by VI (5 min at 50 mbar plus 5 min at atmospheric pressure). In another study, Sanzana et al. (2011) studied the influence of VI on the respiration rate of broccoli, endive, carrot and cauliflower, using isotonic sucrose solution, and reported that the changes in the respiration rates after VI (10 min at 50 mbar and 10 min at atmosphere pressure) depended on the temperature (5 or 20°C) and were not consistent in all the investigated products. In **Paper V,** it observed a lower CO_2 consumption treated by VI with water during storage than that in dipping group.

Some other changes of physico-chemical characteristics are mainly brought along with the functional properties of the applied impregnating solutions. For example, vacuum impregnation allows to obtain the acidification of plant material using organic acids (Derossi, De Pilli, & Severini, 2010) and the improvement of textural characteristics with the introduction of calcium ions (Tappi et al., 2016). Moreover, the aromatic enrichment of apple sticks was obtained by impregnation with an artificial green apple flavouring at 280 mbar for only 5 min (Comandini et al., 2010).

Sapers, Garzarella, and Pilizota (1990) used pressure infiltration at 108 kPa on potato plugs of ascorbic- or erythorbate-based enzymatic browning inhibitors and reported an extension of shelf life by 2–4 days when compared to dipping.

Therefore, it is necessary to select the proper operational parameters and impregnation solutions in order to optimize the potential influence on the physico–chemical characteristics and sensory attributes of the final products (Zhao & Xie, 2004).

Paper V investigated the potential of VI for the enrichment of potato sticks with an essential rosemary oil. Samples were subjected to impregnation at 60 mbar for 30 min followed by a relaxation time at atmosphere pressure for 30 min. The results showed that VI allowed to effectively impregnate potato sticks with rosemary essential oil, with a weight gain in the range of 6-12%, depending on the solution concentration. The rosemary flavour, detected by GC analysis and sensorial test, was still perceivable after frying, even for the samples stored at 4 °C for 14 days.

3.3.1.3 Influence of VI on nutritional properties

VI also provides the opportunity to improve the nutritional properties of fruits and vegetables. For instance, small nutritional molecules such as minerals (calcium, iron, zinc etc.) and vitamins (vitamin C and E) can be vacuum impregnated into different fruits and vegetables tissue including apple, pear, and eggplant (Fito et al., 2001; Hironaka et al., 2015; Hironaka et al., 2014; Lin et al., 2006; Park, Kodihalli, & Zhao, 2005; Xie & Zhao, 2003). However, also large size nutritional compounds and living microorganisms can be introduced into fruit tissue using VI. Betoret et al. (2003) developed probiotic-enriched (*S. cerevisiae* and *L. casei*) dried apples using VI at 50 mbar for 10 min followed by a relaxation time at atmospheric pressure of 10 min and further air drying.

With the aim of nutritional fortification, apple slices have been impregnated also with grape juice (Joshi & Rupasinghe, 2010) and blueberry juice (Castagnini et al., 2015).

Although the low porosity of potatoes, some successful applications of VI for their nutritional enrichment have been reported. Hironaka et al. (2011) applied a vacuum pressure of 70 cm Hg for 0–60 min on whole potatoes in 10% ascorbic acid solution, followed by immersion at atmospheric pressure for 3 h. A final ascorbic acid content of 150 mg/100 g fresh weight was found at 60 min of vacuum time.

Similar experiments were conducted for the enrichment of whole potato with iron and zinc (Hironaka et al., 2015; Hironaka et al., 2014).

4. Materials and Methods

4.1 Application of HHP as a pre-treatment for osmotic dehydration of green plum

4.1.1 Dehydration kinetics and mechanism study of HHP pre-treatment for OD for green plum

Green plum (*Prunus mume* Siebet Zucc) were purchased from an orchard located in Shaoxing (Zhejiang Province, China). The altitude and latitude of the orchard was 500-600 meters and 29° North. 2 cylinders with a diameter of 0.5 cm and 1 cm length were cut from the pericarp of each fruit, using a hand-made sharp metal tube.

Before the HHP treatment, the green plum cylinders were packed in PE/PA food packaging bags together with an isotonic solution (6% sucrose solution) with a ratio of 1:20. The bags were sealed by a thermal sealer (300A, Bake Easy, China) after manual removal of the residual gas. The packed samples were subjected to HHP treatment at 50, 100, 200, 400 MPa for 10 min. Distilled water was used as pressure transmitting medium. For the control, the green plum cylinders were immersed in isotonic solution at room temperature for 10 min. HPP-treated and control green plum cylinders were dipped in 40% sucrose solution and magnetically stirred at 80 rpm at room temperature (22 °C) up to 6 h, based on the procedure described by Mauro et al. (2016), as shown in Figure 5. The concentration of sucrose was chosen to be lower compared to the highest achievable (around 61%), in order to limit the final concentration of sugar in the product, for both sensorial and nutritional reasons.

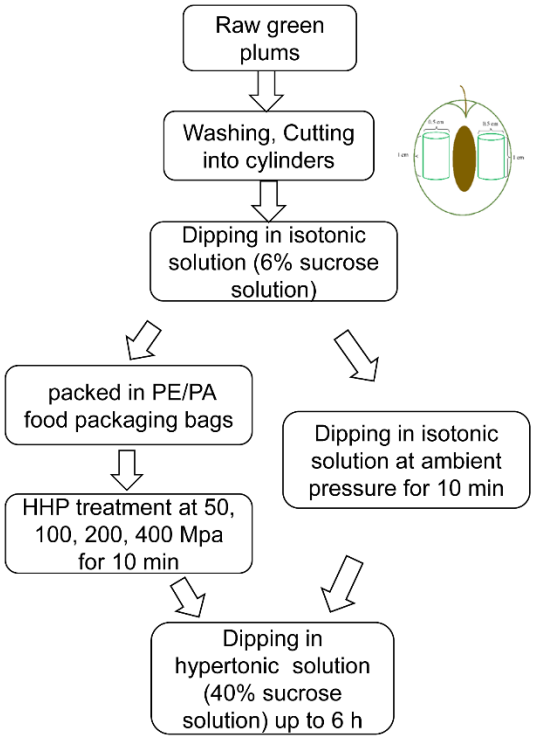

Figure 5. Flow sheet of green plum processing for the dehydration kinetic study

The analytical determinations carried out on the treated green plum samples were:

- Mass transfer kinetic modeled with Peleg's model and Crank model;
- Water state through Time Domain Nuclear Magnetic Resonance (TD-NMR);
- Cell viability using the Fluorescein diacetate (FDA) staining.

All detailed methods were reported on the results section in **Paper I**.

4.1.2 Effect of HHP pre-treatment on mass transfer and quality of osmotically dehydrated green plum

On the basis of the traditional process used for obtaining candied green plums, the fruits, still containing seeds, were perforated by a tool containing matrix of needles (1 mm of diameter, 25 needles/cm^2) in order to accelerate mass transfer based on the traditional process of candied green plum. This pre-treatment is one of the main treatments used in the industry. The perforated green plums were immersed in hypertonic sucrose solutions (40% w/w) and packed in high barrier PE/PA food packaging bags with a fruit-to-syrup ratio of 1:4 (w/w). Before the HHP treatment, the bags were sealed by a thermal sealer (300A, Bake Easy, China) after manual removal of the residual gas.

A central composite design (CCD) was applied to evaluate the singular, quadratic and interactive effects of treatment pressure (50-400 MPa) and time (1-30 min). The design with actual variables of the experiment is shown in Table 1. Solids Gain and Water Loss after 72 h were used as processing responses. Every experiment was conducted in 3 replicates. The optimized result was given by the software (Design Expert 8.0.5, Statease, U.S.A). Because from the results, no improvement in the mass transfer parameters was observed by increasing pressure or time, the lowest values of these variables (50 MPa and 1 min) were chosen for the second part of the experiment.

Table 1. Central composite design (CCD) with coded and actual variables

Run	Coded variables		Actual variables	
	Factor 1	Factor 2	Pressure (MPa)	Time (min)
1	0	-1.41	225	1
2	0	0	225	16
3	1	-1	349	5
4	-1.41	0	50	16
5	-1	-1	101	5
6	1.41	0	400	16
7	1	1	349	26
8	0	0	225	16
9	0	1.41	225	30
10	0	0	225	16
11	-1	1	101	26
12	0	0	225	16
13	0	0	225	16

The perforated samples were divided into four groups to compare different treatments on the efficiency of OD and quality of the products:

a) Untreated green plums were used as the control (C group).

b) HHP treated samples (HHP group) were obtained according to the parameters and procedure chosen in the first part of the experiment (50 MPa for 1 min).

c) Samples subjected to heating (H group) were obtained by immersion in boiling 40% sucrose solutions for 10 s followed by cooling down to room temperature (22 °C), the total process time was about 20 min.

d) The last group was treated accordingly to the traditional candied green plums procedure (T group): perforated green plums were dipped in 1% $CaCl_2$ and 5%

NaCl solution for 10 h in order to eliminate the bitter taste and harden the fruits, then immersed in boiling 40% sucrose solutions for 10 s and naturally cooled down to room temperature (22 °C).

After the pre-treatments, the plums were dipped in a 40% sucrose solution at room temperature (22 °C) for a total of 5 days.

Figure 6 exhibits the flow sheet of the green plums processing for the evaluation of mass transfer and quality parameters.

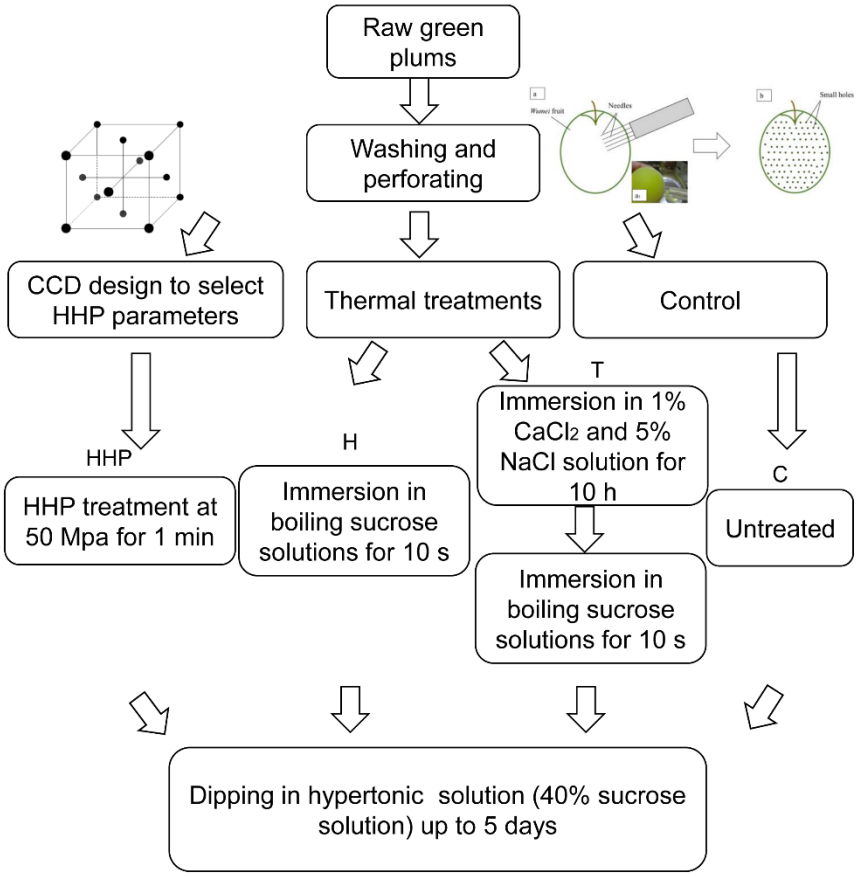

Figure 6. Flow sheet of the comparison of HHP and thermal treatments on mass transfer and quality influence of candied green plums

Treated samples were analyzed for:

- Mass transfer kinetic modeled with Peleg's model;
- Colour;
- Texture;

- Sensory profile;
- Antioxidant content (ascorbic acid, total polyphenols and total flavonoids content);
- Antioxidant activity (DPPH and ABTS methods);
- Volatile profile by SPME-GC-MS.

All detailed methods were reported on the results section in the list of papers (**Paper II and III**).

4.2 Pulsed Electric Fields (PEF) for mitigation of acrylamide content in potato chips

Potato tubers (*Solanum tuberosum* L.) of the Lady Claire variety (suitable for industrial processing), were purchased at a local market and stored in the dark at 10 ± 2 °C for a maximum of two weeks before trials. Before pre-treatment, only tubers of a similar size and shape were selected, manually peeled and sliced (1.5 ± 0.2 mm in thickness) using a stainless-steel electric slicer machine (Mod. KAFPL0922N CAD, Italy). Both tubers and slices were rinsed for 1 min in tap water (water temperature: 18 ± 2 °C) prior to pre-treatments.

Samples were subjected to PEF anf blanching pre-treatments as follow:

1) Pulsed electric fields (PEF)

An electric field strength of 1.5 kV cm^{-1} was selected in order to achieve irreversible electroporation. The treatment chamber was filled with tap water, with an initial electrical conductivity of 542 ± 2 μS cm^{-1} at 25 °C, measured using an EC-meter (Mod. Basic 30, Crison, Spain). A product–to–water ratio of around 1:5 (w/w) was used. The applied PEF treatment parameters are listed in Table 2. Temperature changes due to PEF treatments were negligible. After PEF treatment, the slices were immersed in distilled water at room temperature for 3 min with a product-to-water ratio of around 1:2 (w/w).

Table 2. Pulsed electric fields treatment parameters

PEF parameter	
Field strength E [kV/cm]	1.5
Pulse number n	1000
electrode distance d [cm]	4.7
Pulse form	rectangular monopolar
Pulse duration τ [µs]	10
Pulse frequency [Hz]	100
Total treatment time [s]	10

2) Blanching

Blanching was performed by immersing potato slices in hot distilled water at 85 °C and for 3.5 min stirring with a product-to-water ratio of around 1:2 (w/w).

Reference standard samples (Control) were treated by immersing potato slices in distilled water at room temperature for 3.5 min with a product-to-water ratio of around 1:2 (w/w).

PEF-treated, blanched and control treated potato slices were deep-fried in high-oleic sunflower oil using an electrical fryer (Mod. MFR280R, Fama Industrie, Italy) at 175 °C as initial oil temperature. Potato-to-oil weight ratio was around 1:10 and slices were fried for 3 min until a final moisture of ~2% (wet basis) was reached.

The flow sheet of potato processing is shown in Figure 7.

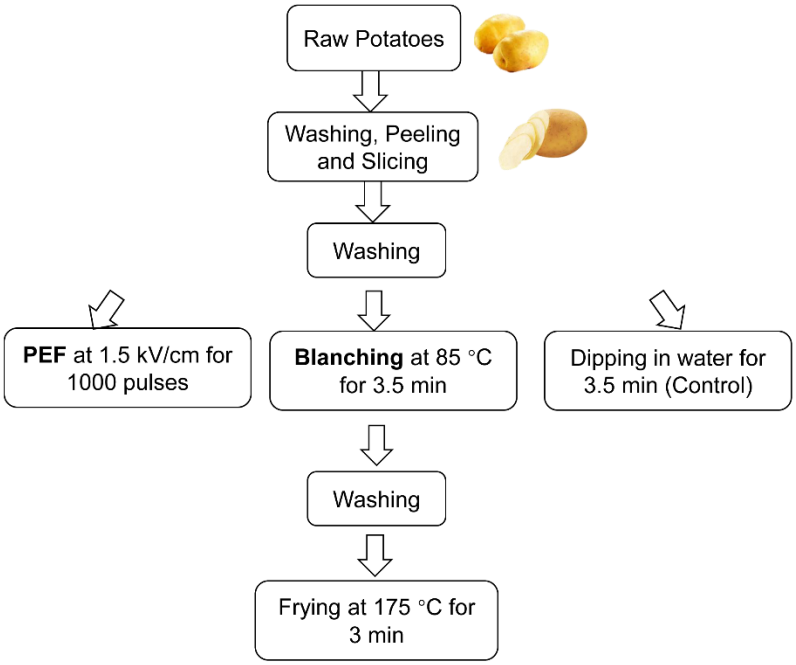

Figure 7. Flow sheet of potato processing

Fried potato slices were analyzed for:

- Colour;
- Texture;
- Acrylamide content.

All detailed methods were reported on the results section in **Paper IV**.

4.3 Vacuum impregnation for the aroma enrichment of potato sticks

The potatoes (*Solanum tuberosum* L.) used in this study were collected in January, provided by the company Pizzoli S.R.L. (Italy). Potatoes were firstly washed with tap water to remove dirt and then dipped in a 200 ppm sodium hypochlorite solution for 2 min for sanitizing the surface. The skins were peeled using a sharp knife and then cut by a manual cutter into pieces of rectangular shape of 1 x 1 x 7 cm.

Rosemary essential oil solutions were prepared at 0, 4, 8 and 12 % (w/v). Potatoes sticks were immersed in the solutions a 1: 1.5 product/solution ratio. A sub-atmospheric pressure of 60 ± 10 mbar was applied for 30 minutes followed by a relaxation time at atmospheric pressure of 30 min. Afterwards the excess liquid on the surfaces of the samples was removed lightly with absorbent paper. Reference standard samples were treated by immersing potato sticks in distilled water at atmospheric pressure for the same time (about 70 min).

The potato sticks were packed in polypropylene trays, sealed with a medium permeability polyethylene film and stored at 4 °C for 14 days. After 0, 3, 7, 10 and 14 days, 3 packages for each sample were removed for analytical determinations.

Moreover, after 0, 7 and 14 days of storage, analytical determination were carried out also on the product after frying. Frying was carried out with a home fryer (De Longhi, Italy), using peanut oil (oil/ samples ratio was 20: 1) at 180 °C for 5 min. After frying the potatoes were drained of excess oil and dried on paper towels for 5 min before the analytical determinations.

The flow sheet of the vacuum impregnation process for the enrichment of potato sticks with a rosemary essential oil solution is shown in Figure 8.

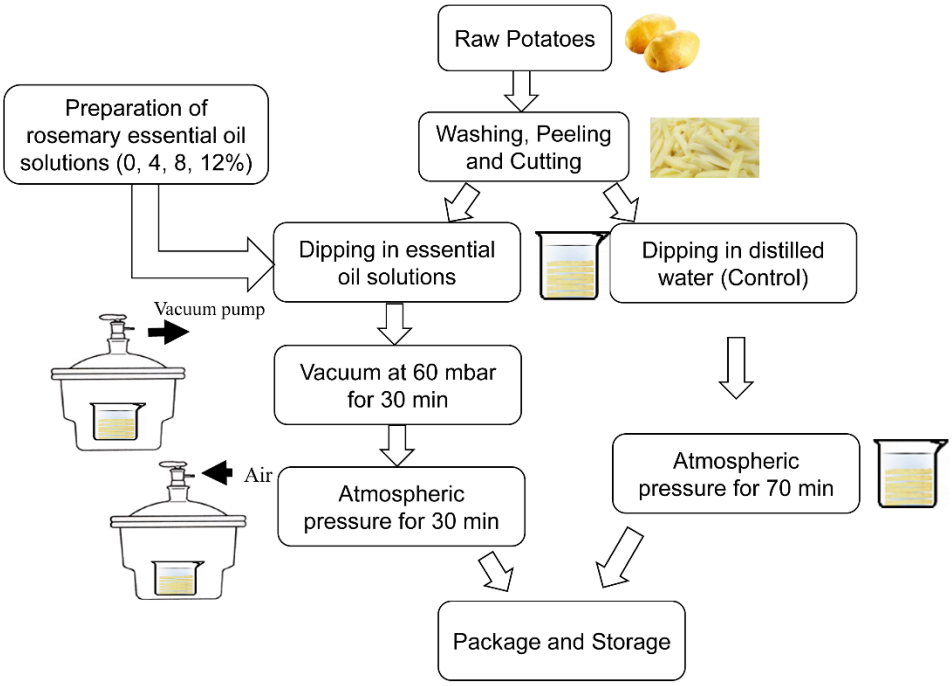

Figure 8. Flow sheet of vacuum impregnation for aromatic potato sticks.

Analytical determination carried out on the fresh samples were:

- Weight gain (%) after the treatment;
- Moisture content;
- Respiration rate;
- Colour;
- Texture;
- Volatile compositions;
- Microbiological quality.

Analytical determination carried out on the fried samples were

- Colour;
- Texture;
- Sensorial profiles;
- Oil content.

All detailed methods were reported on the results section in the paper (**Paper V**).

5. Conclusions

The research findings of this PhD activity increase the application prospect of some non-thermal technologies (High Hydrostatic Pressure, Pulsed Electric Fields, Vacuum Impregnation) on minimally processed fruits and vegetables. The results related to the applied non-thermal techniques confirmed their different potentiality in the toptic of quality improvement and product innovation.

The application of HHP resulted as a promising non-thermal technology, evaluated as an alternative to heat treatment prior to OD, as it has shown to efficiently accelerate the mass transfer, allowing to reduce the dehydration time from more than 5 days to about 2 days. Moreover, HPP allowed to obtain dehydrated fruit charcaterized by high nutritional and sensorial properties.

Pulsed Electric Fields as a pre-treatment was found to be a useful method to reduce the acrylamide content in deep-fat fried potato crisps by improving the release of acrylamide precursors in raw potatoes. By monitoring treatment parameters and other manufacturing steps, it was possible to achieve a consistent reduction of acrylamide, with only slight modifications of the final quality of the product, in terms of colour and texture.

Vacuum Impregnation was applied for the aromatic enrichment of potato sticks showing a good potentiality for product innovation. However, based on the characteristics of the raw materials, the process needs to be improved in order to guarantee the quality of the product during storage.

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6. List of papers

Paper I: Study of the effect of High Hydrostatic Pressure (HHP) on the osmotic dehydration mechanism and kinetics of wumei fruit (Prunus mume)

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ORIGINAL PAPER

Study of the Effect of High Hydrostatic Pressure (HHP) on the Osmotic Dehydration Mechanism and Kinetics of *Wumei* Fruit (*Prunus mume*)

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Abstract
Osmotic dehydration (OD) is the most important procedure for obtaining candied *wumei* (*Prunus mume*), which is a very popular snack in Eastern Asian countries. This study aims to evaluate the effects of high hydrostatic pressure (HHP) pre-treatment (50–400 MPa) on the mass transfer kinetics and on the water diffusivity of *wumei* fruit during OD and to investigate the effect on water distribution and cell viability aspects. The results showed that HHP increased initial rate and effective diffusivity of mass transfer values compared to non-treated samples. Time domain nuclear magnetic resonance revealed that, upon HHP treatment, the water redistributed in vacuole, cytoplasm/extracellular spaces, and cell wall/membrane. The application of 400 MPa probably caused some irreversible damages to the cell membranes. The cell viability study determined by fluorescein diacetate staining showed a loss of cell viability at pressures higher than 200 MPa. HHP exhibited an effective pre-treatment to increase mass transfer of *wumei* fruit during OD process.

Keywords Candied *wumei* fruit · Osmotic dehydration · High hydrostatic pressure · Water distribution · TD-NMR · FDA staining

Introduction

Wumei (*Prunus mume*) is a popular fruit because of its pleasant odor and therapeutic benefits (Yan et al. 2014). Recent studies evidenced its health benefits like anti-oxidative and free radical scavenging activities (Imahori et al. 2008), blood fluidity improvement (Chuda et al. 1999), anti-fatigue activity (Tsuji et al. 2011), and anti-cancer properties (Adachi et al. 2007). It is commonly consumed as processed product like candies, pickle, drink, or liquor (Imahori et al. 2008) rather than the fresh fruit due to its sour taste. In Eastern Asian countries, candied *wumei* is one of the favored snacks. According to "the white paper on the development of Chinese candied fruit industry in 2016," the annual output value of candied fruit in China is about $23.5 billion, with an annual average growth rate of 21.78% in the last decade. Candied *wumei* is one of the main products in this industry.

Osmotic dehydration (OD) is often used for improving sensorial characteristics of this fruit. The raw material is placed into concentrated solutions of soluble solids having higher osmotic pressure which are caused by the water and solute activity gradients across the cell membrane (Chandra and Kumari 2015). Since the cell membrane is a semi-permeable surface, the solutes in the hypertonic solution enter the free space of the tissue while water comes out from the cells (Nowacka et al. 2014; Deng and Zhao 2008). It has been widely used to improve the nutritional and sensorial characteristics of the final products (Tappi et al. 2017; Yadav and Singh 2014; Chandra and Kumari 2015). However, OD is a relatively slow process (Souraki et al. 2015), hence the combination with several other treatments during or before OD, such as pulsed electric field (Traffano-Schiffo et al. 2016), ultrasound (Nowacka et al. 2014), pulsed vacuum (Deng and Zhao 2008), and high hydrostatic pressure (Núñez-Mancilla et al. 2014) have been proposed to further enhance mass transfer.

High hydrostatic pressure (HHP) is one of the most successfully commercialized non-thermal processing technologies in the food industry (Huang et al. 2017). Compared to

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traditional thermal treatments, HHP helps the retention of nutritional and organoleptic attributes of foods (McInerney et al. 2007; Deliza et al. 2005). As reported by literature, HHP is able to improve mass transfer during OD progress by damaging the cell structure (Rastogi et al. 2002) in several fruits such as pineapple, strawberry, banana, etc. (Rastogi and Niranjan 1998; Verma et al. 2014; Nuñez-Mancilla et al. 2011). Since *wumei* fruit has relative aggregated tissue, and the market of candied *wumei* is very large, it is worth to investigate the application prospect of HHP on this product.

Time domain nuclear magnetic resonance (TD-NMR) represents a valuable tool in order to better understand the water distribution inside the cell and the internal water transports (Santagapita et al. 2013). By measuring the transverse relaxation time of protons (T_2), it is possible to separately observe the water located in extracellular spaces and cytoplasm, in vacuole, and the water tightly bound to the most rigid biopolymers (Traffano-Schiffo et al. 2017a; Santagapita et al. 2016; Dellarosa et al. 2016).

The aim of this study was to evaluate the effects of HHP pre-treatment (50–400 MPa) on the osmotic dehydration kinetics of *wumei* fruit and to investigate the HHP-induced mechanisms that affect the mass transfer by water distribution and cell viability.

Materials and Methods

Raw Materials

Wumei fruits (*Prunus mume* Siebet Zucc) were purchased from an orchard located in Shaoxing (Zhejiang Province, China). The altitude and latitude of the orchard is 500–600 m and 29° N. The fruits were harvested at commercial maturity, selected for uniform shape and size (about 20 g per fruit), and then stored at 4 °C and 100% relative humidity (RH) until use.

Sample Preparation

Two cylinders with a diameter of 0.5 cm and 1 cm length were cut from the pericarp of each *wumei* fruit, using a hand-made sharp metal tube, as shown in Fig. 1. Each cylinder weighed around 0.2 g.

The hypertonic sucrose solutions were prepared by dissolving commercial sucrose in distilled water at a concentration of 40% (*w*/*w*).

HHP Process

Before the HHP treatment, the *wumei* cylinders were packed in PE/PA food packaging bags together with an isotonic solution (6% sucrose solution) with a ratio of 1:20. The bags were sealed by a thermal sealer (300A; Bake Easy, China) after manual removal of the residual gas.

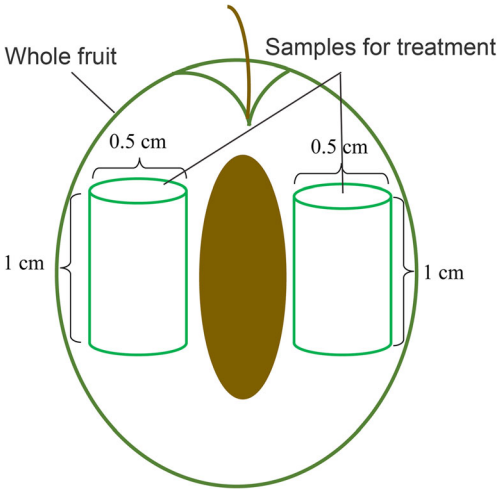

Fig. 1 Schematic representation of the samples obtained from *wumei* fruit. Two cylinders with 0.5 cm diameter and 1 cm height were obtained for each fruit

The packed samples were subjected to HHP treatment in a pressure chamber (Baotou Kefa High Pressure Technology Co., Ltd., China) with a working volume of 10 L and diameter of 120 mm and a recommended maximum working pressure of 600 MPa. Samples were subjected to pressures in 50, 100, 200, and 400 MPa for 10 min. Parameters were chosen on the basis of previous literature studies (Nuñez-Mancilla et al. 2013; Rastogi et al. 2000a; Dörnenburg and Knorr 1993). Distilled water was used as pressure-transmitting medium. The changes of temperature and pressure of the sample during HHP treatment are shown in Fig. 2 (400 MPa as an example).

For the control, the *wumei* cylinders were immersed in isotonic solution at room temperature for 10 min.

Osmotic Dehydration (OD)

HPP-treated and control *wumei* cylinders were dipped in 40% sucrose solution and magnetically stirred at 80 rpm at room temperature (22 °C) up to 6 h, based on the procedure described by Mauro et al. (2016). The fruit-to-syrup ratio was set at 1:20 (*w*/*w*) to avoid concentration changes of the solution (Duduyemi et al. 2015; Ahmed et al. 2016; Castro-Giráldez et al. 2011). After removal from the syrup, the samples were blotted with absorbent paper to remove the solution adhered on the surface.

Moisture Content

For determining the mass transfer kinetic, samples were analyzed for moisture at 0, 0.25, 0.5, 0.75, 1, 2, 4, and 6 h. Moisture content was determined by placing the samples in an oven at 105 °C until constant weight (Assis et al. 2017).

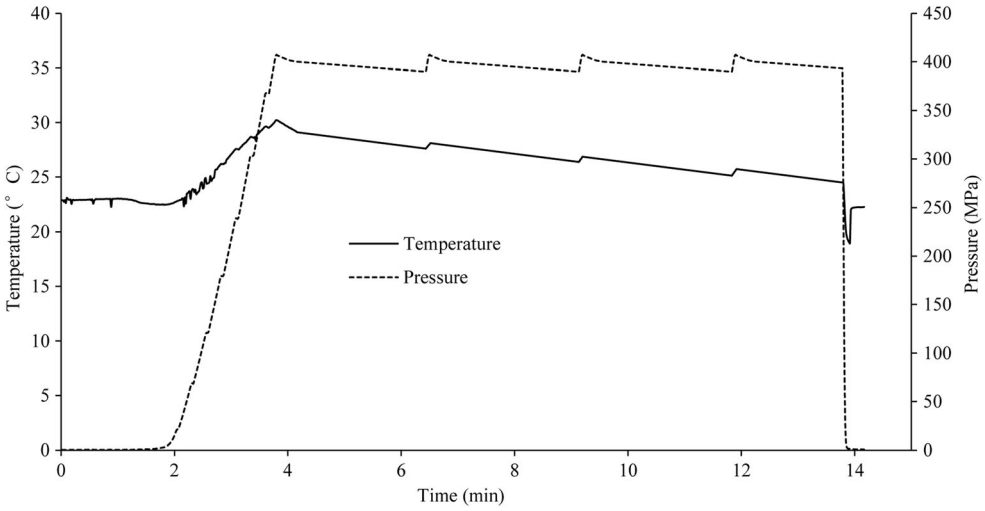

Fig. 2 Temperature and pressure changes during HHP treatment at 400 MPa for 10 min

Mass Transfer Kinetics

Different models have been proposed to predict the mass transfer kinetics during the OD process. In this research, the mathematical models proposed by Peleg (1988) and Crank (1979) were applied to fit the experimental data. Peleg's model can be classified as empirical and semi-empirical model with the advantage to estimate the initial rate and equilibrium values of mass transfer, and it is largely applied to model OD processes (Assis et al. 2016). However, it does not take into account the phenomenological mechanisms in the process (Assis et al. 2016). Based on Fick's second law, Crank proposed a model to evaluate the mass diffusivities of WL and SG (Crank 1979), considering the phenomenological mechanisms. These two models can provide useful information for the comparison of mass transfer efficiency during OD process by different treatments.

The parameters solids gain (SG) and water loss (WL) were determined through the following equations (Corrêa et al. 2010):

$$SG = \frac{w_s M_t - w_{s0} M_0}{M_0} \tag{1}$$

$$WL = \frac{w_{w0} M_0 - w_w M_t}{M_0} \tag{2}$$

where w_s is the solids content at time t, M_t is the weight in time t, w_{s0} is the initial solids content of sample, M_0 is initial weight, w_{w0} is the initial moisture content, and w_w is the moisture content at time t, all expressed in grams.

Peleg's model (Peleg 1988) was applied using the equation proposed by Palou et al. (1994):

$$SG\ or\ WL = \frac{t}{k_1 + k_2 \times t} \tag{3}$$

k_1 and k_2 are Peleg's constants for SG or WL.

The constant k_1 relates to the initial rate of the mass transfer, and the constant k_2 relates to equilibrium values, as can be calculated below:

$$\left[\frac{\mathrm{d}(SG\ or\ WL)}{\mathrm{d}t}\right]_{t\to 0} = \lim_{t\to 0}\left[\frac{\mathrm{d}\left(\frac{t}{k_1 + k_2 \times t}\right)}{\mathrm{d}t}\right] = \frac{1}{k_1} \tag{4}$$

$$\lim_{t\to\infty}(SG\ or\ WL) = \lim_{t\to\infty}\frac{t}{k_1 + k_2 \times t} = \frac{1}{k_2} \tag{5}$$

This kinetic model offers the advantage that by calculating the inverse of the constant (k_1 and k_2), it is possible to obtain the initial rate and the values at the equilibrium of mass transfer parameters (Sacchetti et al. 2001).

Crank provided Eq. (6) to calculate the effective diffusivity (D_{eff}) for various regular geometry materials (rectangular, cylindrical, and spherical):

$$\frac{\partial X}{\partial t} = D_{eff}\frac{\partial^2 X}{\partial z^2} \tag{6}$$

where X is the amount of water or solids at instant, t is time (s), D_{eff} is effective diffusivity (m^2/s), and z is a generic directional coordinate. With the appropriate initial and boundary conditions, the equation can be modified as Eq. (7) for finite cylinder samples:

$$W_{w\ or\ s} = \frac{4}{f}\cdot\frac{8}{\pi^2}\sum_{i=0}^{\infty}\frac{1}{(2i+1)^2}\exp\left[-(2i+1)^2\left(\frac{f}{r^2}+\frac{\pi^2}{4l^2}\right)\cdot D_{eff}\cdot t\right] \tag{7}$$

where f depends on shape of sample, for cylinders $f = 5.783$, i is the number of series terms, r is the radius, l is the characteristic half length (m), and $W_{w\ or\ s}$ is the dimensionless water or solid content, which can be calculated by Eq. (8):

$$W_{w\ or\ s} = \frac{M - M_e}{M_0 - M_e} \tag{8}$$

where M is moisture or solids content at time t, M_0 is initial moisture or solids content, and M_e is equilibrium moisture or solids content which is measured after 6 h of OD.

For long periods of OD process, when the Fick number $\left(\frac{f}{r^2} + \frac{f}{r^2}\right) \cdot D_{eff} \cdot t$ is less than 0.1 or $W_{w\ or\ s}$ is less than 0.6, only the first term of the series terms is used (Sinha et al. 2010). Although it is less rigorous in the calculation, it is a good approximation of reality ($i = 0$) (Simpson et al. 2015). Equation (7) could be simplified as:

$$W_{w\ or\ s} = \frac{4}{f} \cdot \frac{8}{\pi^2} \exp\left(-\left(\frac{f}{r^2} + \frac{\pi^2}{4l^2}\right) \cdot D_{eff} \cdot t\right) \tag{9}$$

Take natural logarithms on both sides of the equation, it becomes:

$$\ln(W_{w\ or\ s}) = \ln\left(\frac{4}{f}\right) + \ln\left(\frac{8}{\pi^2}\right) - \left(\frac{f}{r^2} + \frac{\pi^2}{4l^2}\right) \cdot D_{eff} \cdot t \tag{10}$$

The effective diffusivity D_{eff} could be calculated by Eq. (10) by plotting $\ln(W_{w\ or\ s})$ curve versus time t.

Water State by Time Domain Nuclear Magnetic Resonance (TD-NMR)

The proton transverse relaxation time (T_2) of the samples after treatments and 6 h of OD was measured by a nuclear magnetic resonance (NMR) spectrometer (PQ001; Suzhou Niumag Analytical Instrument Corporation, China). The Carr–Purcell–Meiboom–Gill (CPMG) pulse sequence was used at 22.907 MHz and 25 °C. About 0.5 g *wumei* samples were placed in the NMR tubes. The parameters were set as follows to avoid sample heating: repetition time, 10 s; echo time, 200 μs; echo count, 16,000.

The acquired CPMG data were analyzed by the inversion of decay data software named T2_InvfitGeneral given by the instrument company (Suzhou Niumag Analytical Instrument Corporation, China) and normalized by the sample weights to obtain the proton transverse relaxation time (T_2).

Cell Viability Test by Fluorescein Diacetate (FDA) Staining

The cell viability test was performed on the samples after treatments using fluorescein diacetate (FDA, Sigma-Aldrich, USA, $\lambda_{ex} = 495$ nm, $\lambda_{em} = 518$ nm), as described by Tylewicz et al. (2013) with some modifications. After the treatments, *wumei* samples were manually cut into slices using a scalpel and placed on microscope slides; one to two drops of 100 μM FDA solution were added to cover each slice which were followed by incubation for 2 h in darkness at room temperature. Nikon upright microscope (Eclipse Ti-U; Nikon Co., Japan) equipped with a Nikon digital video camera (digital sight DS-Qi1Mc; Nikon Co., Japan) under a fluorescent light was used to observe the cells. Viable cells could be easily identified by a bright fluorescence.

Statistical Analysis

The statistical significance of differences was analyzed with Statistics 22.0 (SPSS Inc., Chicago, IL, USA) by one-way analysis of variance (ANOVA) using the Tukey post hoc test ($p < 0.05$).

The regression coefficient (R^2) and root mean square error (RMSE) were applied to evaluate the fitting efficiency of the applied models. R^2 was calculated by Eq. (11), and RMSE was calculated by Eq. (12):

$$R^2 = 1 - \frac{SSE}{SST} \tag{11}$$

$$\mathrm{RMSE} = \sqrt{\frac{\sum\left(y_{obs} - y_{pre}\right)^2}{n}} \tag{12}$$

where SSE is the error sum of squares, SST is the total sum of squares, y_{obs} is the observed value, y_{pre} is the predicted value given by the model, and n is the number of observations.

The parameters of the applied models were calculated by Origin Lab 8.0 (Massachusetts, USA). All determinations were carried out in triplicate. Pearson's analysis was performed to evaluate the correlation between kinetic and water state data.

Results and Discussions

Osmotic Dehydration (OD) Kinetics

Moisture and solids content during the OD process are reported respectively in Fig. 3a and b. During OD, HHP-treated samples showed lower moisture content and higher solid content compared to the control at all processing times. However, there were no significant differences among the HHP samples. After 6 h of dehydration, water content was around 0.63 and 0.60 g/g for respectively control and HPP-treated samples.

The fitted parameters of Peleg's models are shown in Table 1. In general, the model showed a good fit to experimental data, as high R^2 values (0.95–0.99) and low RMSE were found, confirming its suitability for describing mass transfer phenomena as already reported by Peleg (1988) and on other studies successively (Palou et al. 1994; Sacchetti et al. 2001; Tappi et al. 2017).

Initial rates and equilibrium values of WL were greater than the values of SG. This behavior occurs commonly in plant

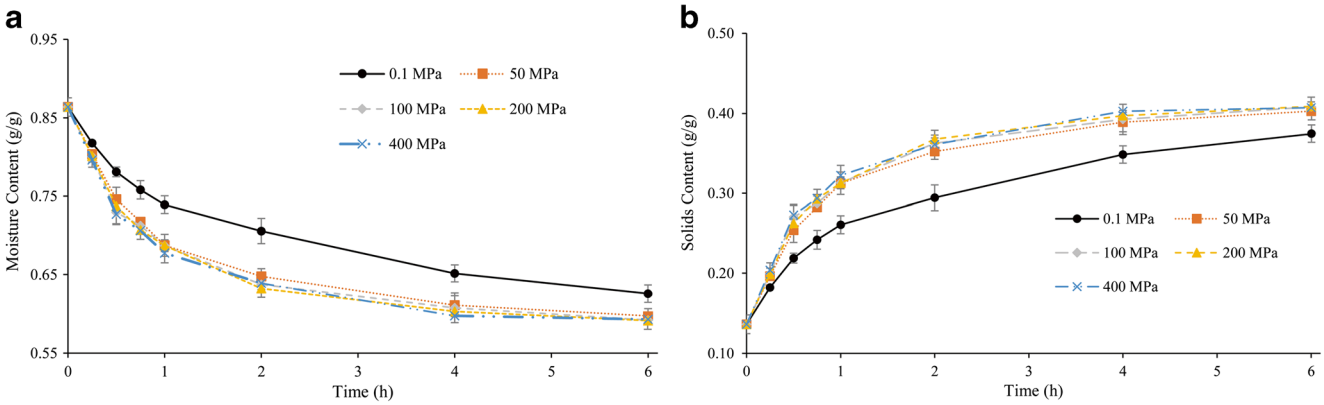

Fig. 3 Moisture and solids content during the OD process

tissue (Tappi et al. 2017; Silva et al. 2014; Yu et al. 2017; Assis et al. 2017) because of the selective permeability of the cell membranes that allows the transport of small molecules such as water, but restrict the transport of larger molecules such as sucrose, and hence reduces the diffusion of sucrose through the cell tissue (Silva et al. 2014).

For both SG and WL, the initial rate of dehydration was increased in 50, 100, and 200 MPa samples compared to the control, and then further increased at the higher pressure applied (400 MPa), as higher $1/k_1$ values were observed.

The equilibrium rate ($1/k_2$) followed an analogous tendency for WL, while for SG no differences were observed among all samples.

Generally, it has been observed that a pre-treatment with HHP increases the mass transfer during OD; however, according to previous literature studies, the effect is not always proportional with the level of applied pressure, while in some studies an increment in the SG and/or WL during OD was observed by increasing the working pressure (Rastogi et al. 2000a, b; Verma et al. 2014; Rastogi and Niranjan 1998). But Nuñez-Mancilla et al. (2013) found that in the range of applied pressure (100–500 MPa), there was no significant difference in the moisture content of strawberries after OD. Also, Dermesonlouoglou et al. (2008) found that increasing the pressure from 200 to 600 MPa did not increase the SG and WL of cherry tomatoes osmotically dehydrated for 6 h.

To better understand the influence of the HPP pre-treatment on the mass transfer kinetics, effective diffusion coefficients by Crank's equation were calculated (Table 1). Diffusive mass transfer phenomena occur in OD processes. The effective diffusivity is a useful parameter to evaluate the rate of mass transfer (Yadav and Singh 2014). The high determination coefficients (R^2) and low RMSE suggest a good fit to the experimental data. The D_{eff} of the control sample was $1.53 \pm 0.09 \times$

Table 1 Fitted parameter of Peleg's and Crank's model*

Pressure (MPa)	0.1 (control)	50	100	200	400
Peleg's model					
Water loss					
R^2	0.98	0.95	0.96	0.98	0.98
RMSE	0.0075	0.018	0.016	0.012	0.0099
$1/k_1$	0.35 ± 0.07a	0.53 ± 0.02b	0.58 ± 0.02bc	0.55 ± 0.04bc	0.60 ± 0.03c
$1/k_2$	0.36 ± 0.01a	0.40 ± 0.01b	0.39 ± 0.004b	0.39 ± 0.01b	0.40 ± 0.02b
Solids gain					
R^2	0.98	0.97	0.96	0.97	0.95
RMSE	0.0058	0.0087	0.0094	0.0086	0.010
$1/k_1$	0.18 ± 0.03a	0.32 ± 0.01b	0.35 ± 0.02bc	0.36 ± 0.02bc	0.39 ± 0.03c
$1/k_2$	0.23 ± 0.01a	0.24 ± 0.01a	0.25 ± 0.01a	0.25 ± 0.02a	0.24 ± 0.02a
Crank's model					
R^2	0.94	0.95	0.92	0.96	0.98
RMSE	0.03	0.07	0.17	0.08	0.06
D_{eff} ($\times 10^{-10}$)	1.53 ± 0.09a	2.68 ± 0.15b	3.16 ± 0.24bc	3.16 ± 0.16bc	3.76 ± 0.14c

* Different letters indicate significant differences among samples ($p < 0.05$)

R^2 regression coefficient, *RMSE* root mean square error, $1/k_1$ initial rate of the mass transfer, $1/k_2$ equilibrium values of mass transfer, D_{eff} effective diffusivity

10^{-10} m^2/s, which is similar to the value indicated in the literature for other fruits (Sinha et al. 2010). The application of the HHP pre-treatment promoted an increase of the diffusivity, as higher pressure resulted in higher value of D_{eff}. The higher increment was observed initially while at higher pressures it tended to flatten as the same as reported by Rastogi and Niranjan (1998); according to these authors, the application of HHP facilitated the diffusion, thanks to the creation of a more open structure of the tissue. The leveling of the effective diffusivity is due to a limit in the openness of the structure promoted by HHP. However, as observed also by Nuñez-Mancilla et al. (2011), a further increase was observed at 400 MPa.

The cell membranes of fruits exert high resistance to mass transfer and slow down the OD rate (Allali et al. 2010). Application of HHP may cause damages to cell membranes altering their permeabilization (Rastogi et al. 2000a; Farr 1990; Eshtiaghi et al. 1994) and significant changes in the tissue structure resulting in increased mass transfer rates during OD (Rastogi et al. 2002). In particular, the increase of the initial rate of SG and WL and of the effective diffusivity in the 400 MPa treated sample may indicate a more pronounced damage on the cell structure.

Water State of *Wumei* Tissue by HHP and/or OD

The proton transverse relaxation time spectra (T_2) were obtained by TD-NMR in order to have a deeper understanding of the water diffusion affected by HHP and/or OD. Because the T_2 of the protons depends on chemical exchange among water, solutes, and biopolymers (Panarese et al. 2012; Nowacka et al. 2014), representing an index of the mobility of the water molecule (Traffano-Schiffo et al. 2017b), it is possible to separately observe the water in different locations (cell wall, extracellular space, cytoplasm, and vacuole) of plant cells (Mauro et al. 2016). The water molecules combined with cell wall and cell membranes are bonded by the electrical adsorption forces that show the lowest T_2 and intensity. The water in the vacuole displays high mobility and quantity in plant cell, showing the highest T_2 and intensity. The remaining group of water subjected to the plasmolization process, located in the cytoplasm and extracellular space, shows intermediate T_2 and intensity (Traffano-Schiffo et al. 2017a).

Figure 4 and Table 2 show the distribution of T_2 on *wumei* tissue before (Fig. 4a) and after 6 h OD (Fig. 4b) as a function of HHP treatments. In the fresh samples, the distribution of water in vacuoles, cytoplasm plus extracellular spaces, and cell wall plus cell membrane was respectively about 86, 11, and 3%, with the T_2 values of 534, 28, and 4 ms (Fig. 4a and Table 2). The values were slightly different compared to other studies regarding different plant materials (Hills and Duce 1990; Marigheto et al. 2004; Panarese et al. 2012; Tylewicz et al. 2011), possibly due to the different diameter of plant cells (Panarese et al. 2012) and different NMR measurement parameters applied. Actually as Hills and Duce (1990) pointed out, different measurement parameters (pulse spacing time in their work) resulted in T_2 and signal intensity modifications.

Before OD, the HHP pre-treatment caused a redistribution of water among the different compartments, characterized by a decrease of the vacuole proton pool intensity of about 18–20% and an increase of the cytoplasm/extracellular space one of about 2–2.5 times, indicating the water flowed from vacuole to these spaces.

Despite the loss of water from the vacuole, the protons' T_2 significantly decreased only in cytoplasm/extracellular space and increased significantly in samples subjected to higher pressure (400 MPa). On the opposite, T_2 of the cytoplasm/extracellular space proton pools increased significantly in sample subjected to 50, 100, and 200 MPa treatment in a similar way, while the increase for the 400 MPa sample was lower although still significant.

Butz et al. (1994) observed severe damage to the vacuoles of onions' cell after 300 MPa treatments at 25 °C. A similar membrane damage caused by high pressure (higher than 300 MPa) was also observed by other studies (Prestamo and Arroyo 1998; Luscher et al. 2005; Schlüter et al. 2009;

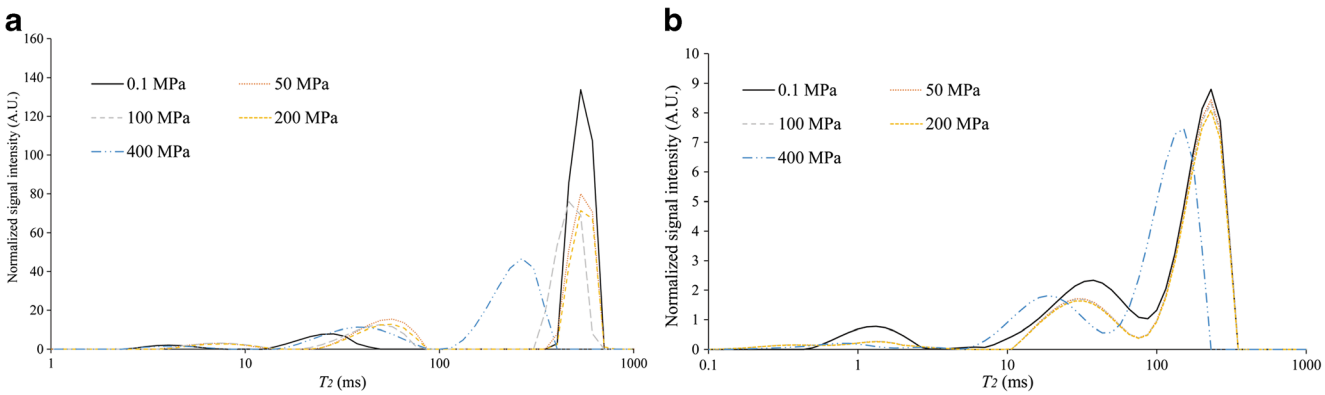

Fig. 4 The distribution of transverse relaxation times (T_2) of proton pools in different cellular compartments before (**a**) and after 6 h OD (**b**) as a function of HHP treatments

Table 2 Effect of HHP on the total and relative intensity and T_2 of proton pools in different cellular compartments before and after OD*

Cell compartments	Pressure (MPa)	Before OD			After 6 h OD		
		Total signal intensity (peak area)	RI (%)	T_2 (ms)	Total signal intensity (peak area)	RI (%)	T_2 (ms)
Vacuole							
	0.1	327 ± 23a	86 ± 3a	533 ± 51a	46 ± 3a	61 ± 3a	230 ± 18a
	50	273 ± 18b	72 ± 4b	513 ± 33a	44 ± 4a	68 ± 4b	229 ± 20a
	100	269 ± 22b	71 ± 3b	464 ± 43a	43 ± 3a	69 ± 3b	225 ± 12a
	200	258 ± 31b	68 ± 1b	518 ± 38a	44 ± 4a	71 ± 4b	226 ± 16a
	400	250 ± 25b	66 ± 2b	265 ± 22b	46 ± 3a	73 ± 3b	150 ± 15b
Cytoplasm/extracellular space							
	0.1	42 ± 2a	11 ± 1a	28 ± 3a	24 ± 3a	31 ± 2a	36 ± 3a
	50	76 ± 7b	20 ± 2b	57 ± 5c	15 ± 1b	23 ± 3b	31 ± 2a
	100	83 ± 7b	22 ± 3b	49 ± 6c	15 ± 2b	24 ± 2b	31 ± 5a
	200	91 ± 8b	24 ± 2b	57 ± 5c	15 ± 2b	24 ± 3b	32 ± 2a
	400	99 ± 7b	26 ± 2b	37 ± 3b	16 ± 3b	25 ± 2b	19 ± 2b
Cell wall/membrane							
	0.1	11 ± 1a	3 ± 0a	4 ± 0a	6 ± 1a	8 ± 1a	6 ± 0a
	50	30 ± 2b	8 ± 1b	7 ± 0b	6 ± 0a	9 ± 1a	1 ± 0c
	100	27 ± 1b	7 ± 1b	7 ± 1b	4 ± 0b	7 ± 0b	1 ± 0c
	200	30 ± 1b	8 ± 0b	8 ± 0b	3 ± 1b	5 ± 1b	1 ± 0c
	400	30 ± 1a	4 ± 0a	4 ± 1a	1 ± 0c	2 ± 0c	3 ± 0b

* Different letters indicate significant differences among samples at the same OD time ($p < 0.05$)

HHP high hydrostatic pressure, *OD* osmotic dehydration, *RI* relative intensity, T_2 proton transverse relaxation time

Gonzalez and Barrett 2010; Kato et al. 2002). Because of the damage of the membrane, the solutes previously inside the cytoplasm may flow into the vacuole, decreasing the mobility of the related water proton pools, as it is clearly showed for 400 MPa samples by Fig. 4a.

As evidenced by Dörnenburg and Knorr (1997), the application of pressure levels of 175 MPa or higher led to an increase in the amount of proteins and phenols in the medium, which constitutes a clear sign of irreversible permeation of the tonoplast surrounding the vacuole. Besides, HHP is able to induce the structural and profile changes of biomacromolecule such as proteins (Boonyaratanakornkit et al. 2002; Mozhaev et al. 1996; Baier et al. 2015; Balasubramaniam et al. 2015) and fatty acids (Tauc et al. 1998; Canto et al. 2015), increasing the exposition of hydrophilic active groups in the solution leading to a decrease in the water mobility in the 400 MPa sample.

Regarding the water bound to cell wall and membrane, the total signal intensity increased 2.33–2.56-fold in 50, 100, and 200 MPa groups compared to the untreated fruit, together with an increase of T_2 by about 3–4 ms, suggesting that the water bound to cell wall and membranes exhibited higher mobility. However, no significant difference was observed between control and 400 MPa sample.

After 6 h of OD at 40% sucrose solution, the total signal intensity dramatically dropped both in the vacuole and cytoplasm/extracellular space, due to the cellular shrinkage caused by the osmotic process.

Moreover, compared to the control samples, a different distribution of water among the cell compartments was observed. In particular, a higher proportion of water was found in vacuoles and a lower one in cytoplasm/extracellular space and cell wall. Nevertheless, for 50, 100, and 200 MPa HPP treatments, the relaxation times of vacuole and cytoplasm proton pools did not change compared to the control. Only in the 400 MPa sample T_2 was significantly lower in both compartments.

According to the study of Kato et al. (2002), pressures lower than 220 MPa caused reversible modification to a lipid bilayer model system, decreasing the fluidity of the membrane by changing it from a liquid crystalline to a gel phase. At higher pressures, irreversible damages on membrane due to protein unfolding and interface separation were observed. As mentioned previously, these conformational changes may contribute to increased water interaction with the macromolecules of the membranes, thus decreasing its mobility. Obtained results suggest that 400 MPa caused irreversible damages to the cell structures.

Table 3 Correlation matrix among OD kinetic parameters ($1/k_1$ and $1/k_2$) and water state parameters (relative intensity of water populations of vacuole, cytosol, and cell wall)

	$1/k_1$ (WL)	$1/k_2$ (WL)	$1/k_1$ (SG)	$1/k_2$ (SG)	$D_{eff,w}$	RI (vac)	RI (cyt)	RI (wall)	RI (vac) OD	RI (cyt) OD	RI (wall) OD
$1/k_1$ (WL)	–										
$1/k_2$ (WL)	0.9173*	–									
$1/k_1$ (SG)	0.9874**	0.9074*	–								
$1/k_2$ (SG)	0.7721	0.5819	0.7638	–							
$D_{eff,w}$	0.9691**	0.8575	0.9842**	0.6911	–						
RI (vac)	− 0.9701**	− 0.9174*	− 0.9948**	− 0.7450	− 0.9713**	–					
RI (cyt)	0.9664**	0.8793*	0.9945**	0.7401	0.9871**	− 0.9944**	–				
RI (wall)	0.9262*	0.9685**	0.9404*	0.7167	0.8749	− 0.9593**	0.9243*	–			
RI (vac) OD	0.9530*	0.8807*	0.9875**	0.6945	0.9852**	− 0.9915**	0.9976**	0.9204*	–		
RI (cyt) OD	− 0.8847*	− 0.9292*	− 0.8628	− 0.7821	− 0.7661	0.8700	− 0.8200	− 0.9558*	− 0.7993	–	
RI (wall) OD	− 0.5432	− 0.3728	− 0.6251	− 0.2369	− 0.7332	0.6234	− 0.6911	− 0.4073	− 0.7191	0.1572	–

Note: * and ** indicate significant differences at $p < 0.05$ and 0.01, respectively

WL water loss, *SG* solid gain, *RI* relative intensity, *vac* vacuole, *cyt* cytoplasm, *wall* cell wall

A Pearson correlation was performed in order to evaluate the possible correlation among kinetic data and water state parameters. Results are reported in Table 3. The effective diffusivity of water showed a strong positive correlation to the initial rate ($1/k_1$) of dehydration ($p < 0.01$). Both kinetic parameters were also correlated to the distribution of water in the different compartments after the application of HPP. Indeed, $1/k_1$ and D_{eff} were positively correlated to the relative intensity in the cytoplasm and negatively to the one in the vacuole. After 6 h of OD, a good correlation ($p < 0.05$) was observed also between the Peleg parameters and the relative content of water in vacuoles and cytoplasm.

These results confirm the strong correlation between the water state in a vegetable tissue and the alteration to the membrane that influence the kinetic of dehydration, in agreement with previous findings (Cheng et al. 2014; Traffano-Schiffo et al. 2017a).

Cell Viability

HHP can cause damages to the cell membranes that may subsequently lead to cell death. Through the detection of cell viability, it is possible to have an indirect assistant observation of the influence on cell membrane by HHP. Figure 5 shows slides of *wumei* tissue pre-treated with different pressure levels followed by staining with FDA.

Fluorescein diacetate is known for its ability to passively penetrate the protoplast and to be hydrolyzed by cytoplasmic esterases that produce the fluorescein. This charged form is accumulated intracellularly in viable cells because it is unable to cross cellular membranes that remain intact (Saruyama et al. 2013).

By increasing pressure, a progressive loss of cell viability was observed until 100 MPa. After 200 and 400 MPa HPP treatment, no viable cells were observable. These results are consistent with Dörnenburg and Knorr (1993) that observed that working pressure at around 50 MPa reduced cell viability of *Morinda citrifolia* by about 20%, and pressure higher than 100 MPa caused complete absence of viable cells. The loss of viability of plant cells caused by HHP was also observed by other previous studies (Denoya et al. 2016; Gonzalez et al. 2010; Techakanon et al. 2016) and attributed to the changes in cell permeability due to the loss of membrane integrity (Gonzalez et al. 2010; Gonzalez and Barrett 2010).

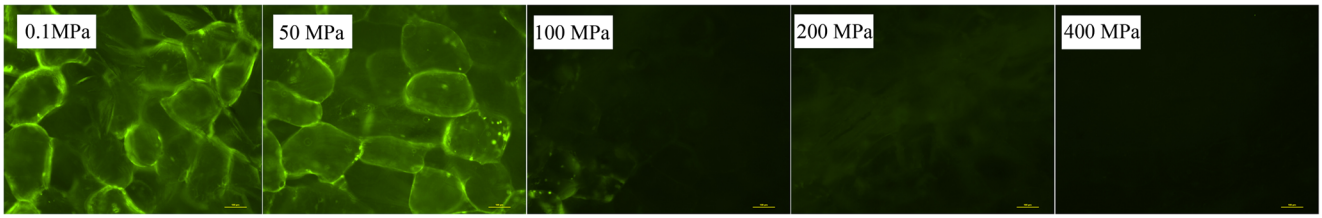

Fig. 5 Micrographs of *wumei* tissue by fluorescence detection microscope, using fluorescein diacetate to identify viable cells. Viable cells are distinguished by a bright fluorescence

Conclusions

In this study, HHP has shown to be an efficient way to accelerate the mass transfer of *wumei* fruit during OD process, as it increased the initial rate and equilibrium value of both solids gain and water loss, although the effect was not proportional to the applied pressure.

The TD-NMR results revealed that the HPP treatment promoted a flow of water flowed from the vacuole to cytoplasm/extracellular space, more pronounced in the 400 MPa treatment. After OD, the application of HPP caused a different distribution of water in the cell compartments. Moreover, results showed that the application of 400 MPa probably caused some irreversible damages to the cell membranes. Microscopic observation allowed to evaluate the loss of cell viability that was complete after 200 MPa treatment.

Generally, the HPP processing technology resulted as a very promising technique in order to obtain OD *wumei* fruit of high quality and stability. Further studies are in due course in our laboratory in order to study the effect of the treatment in real conditions and compare it to traditional heating treatments.

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Paper II: Study and optimization of High Hydrostatic Pressure (HHP) to improve mass transfer and quality characteristics of candied green plums (Prunus mume)

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ORIGINAL ARTICLE

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Study and optimization of high hydrostatic pressure (HHP) to improve mass transfer and quality characteristics of candied green plums (*Prunus mume*)

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Abstract

Osmotic dehydration (OD) is the most important procedure for producing candied green plums. This study investigated high hydrostatic pressure treatment (HHP) to improve OD process. Central composite design was applied to optimize HHP parameters with pressure ranging from 50 to 400 MPa and time from 1 to 30 min. Innovative HPP procedure was compared with traditional heating treatments on the acceleration efficiency by Peleg's model. The physico-chemical and sensorial properties were compared. The results showed that HHP was effective to accelerate the OD process, but was not as efficient as heating methods. HHP promoted higher titratable acid content, firmness and greener color than heating on the obtained product. Sensory results revealed that HHP allowed to better preserve the typical odor and flavor of the final products. Generally, HHP treatments show high potentiality to improve the OD performances of candied green plums, together with the overall quality of the final product.

Practical application

Candied green plum is a popular snack in Eastern Asian countries. The traditional process includes a heating assisted treatment in order to accelerate the candying progress, which may induce decrease to the quality of the final product. Non-thermal high hydrostatic pressure could improve the osmotic dehydration process performances, minimizing the thermal damages on the final products, promoting the improvement of its overall quality.

1 | INTRODUCTION

Green plum (*Prunus mume*) originates in southern China and has been widely cultivated in Eastern Asian countries (Gao et al., 2004). It is a very popular fruit because of its pleasant flavor and odor, wide availability, high nutritional and most importantly, therapeutic benefits (Yan et al., 2014). Green plum has been traditionally used as herbal medicine for coughs and dyspepsia (Jung, Cho, Koh, Han, & Lee, 2010). Recent studies have proved its health benefits, such as anti-oxidative and free radical scavenging activities (Yan et al., 2014), blood fluidity improvement (Chuda et al., 1999), anti-fatigue activity (Tsuji, Koizumi, & Fujiwara, 2011) and anti-cancer properties (Adachi et al., 2007). The Ministry of Health of the People's Republic of China has thus regarded it as both food and medicine since 1985. However, because the taste of fresh green plum is very sour, it is usually processed into different products, one of the most popular

products on the market in Eastern Asian countries is candied green plum. According to "the white paper on the development of Chinese candied fruit industry in 2016," the annual output value of candied fruit in China is about $23.5 billion, with an annual average growth rate of 21.78% in the last decade. Candied green plum is one of the main products in this industry.

Osmotic dehydration (OD) is the principal procedure used for obtaining candied green plums, being an effective way to partially remove water from fruit tissue by its immersion in hypertonic aqueous sugar solutions (Nowacka, Tylewicz, Laghi, Dalla Rosa, & Witrowa-Rajchert, 2014). But the OD procedure is relatively slow (Souraki, Tondro, & Ghavami, 2015), and the tissue of green plum is innately tight. The green plums are usually perforated with needles or broken before OD procedure (Lin et al., 2013). Even in this case, it takes 5 days or more to saturate them with sugar. The heating method is therefore widely applied in industry in order to speed up the candying process, although it may induce damages to the quality of the final product. A non-thermal pretreatment able to improve mass transfer could be preferable.

High hydrostatic pressure (HHP) conforms to these requirements, being an innovative, emerging non-thermal technology with the capability of promoting the retention of nutritional and organoleptic attributes of foods (Deliza, Rosenthal, Abadio, Silva, & Castillo, 2005; McInerney, Seccafien, Stewart, & Bird, 2007). It has been proved that application of HHP causes permeabilization of the cell structure because of the cell disintegration (Eshtiaghi, Stute, & Knorr, 1994; Farr, 1990; Rastogi, Angersbach, & Knorr, 2000), which leads to significant changes in the tissue structure resulting in increased mass transfer rates during OD as compared to untreated samples (Rastogi, Raghavarao, Niranjan, & Knorr, 2002). HHP has been successfully used to improve the mass transfer during OD in several fruits such as pineapple (Rastogi & Niranjan, 1998), strawberry (Nuñez-Mancilla et al., 2011) and banana (Nuñez-Mancilla et al., 2011; Rastogi & Niranjan, 1998; Verma, Kaushik, & Rao, 2014), but, to our knowledge, no studies on green plum have ever been carried out.

Besides, when compared with traditional thermal treatments, energy requirements are lower. Indeed, the increase in pressure to 500 MPa requires lower energy consumption than heating to 100°C (Pereira & Vicente, 2010). The average cost of HPP is approximately 0.05–0.5 US$ per liter or kilogram depending on the processing conditions, and is lower than the cost of thermal processing (Bermúdez-Aguirre & Barbosa-Cánovas, 2011). The main cost involved is the equipment, which has decreased considerably in recent years due to the wide application of this technology (Wang, Huang, Hsu, & Yang, 2016). Because of the large market of candied green plum, it is worthwhile to study the application potential of HHP on this industry.

The aim of this work was to: (a) optimize the HPP process as an assistant treatment to improve OD of green plums; (b) compare with heating treatments on efficiency of OD and the effect on the quality of the final products.

2 | MATERIALS AND METHODS

2.1 | Raw materials

Green plums (*Prunus mume* Siebet Zucc) were purchased from an orchard located in Shaoxing (Zhejiang Province, China). The altitude and latitude of the orchard is 500–600 m and 29° North. Fruits harvested at commercial maturity and with uniform shape and size (about 20 g per each fruit) were selected and stored at 4°C and 100% relative humidity until use.

2.2 | Sample preparation

The hypertonic sucrose solutions were prepared by dissolving commercial sucrose in distilled water at a concentration of 40% (w/w).

On the basis of the traditional process of candied green plums, the green plums, still containing seeds, were perforated by a matrix of needles (1 mm of diameter, 25 needles/cm^2) in order to accelerate mass transfer.

2.3 | HHP process and experimental design

The perforated green plums and hypertonic sucrose solutions were packed in high barrier PE/PA food packaging bags with a fruit-to-syrup ratio of 1:4 (w/w). Before the HHP treatment, the bags were sealed by a thermal sealer (300A, Bake Easy, China) after manual removal of the residual gas.

The packaged green plums were subjected to HHP treatment in a pressure chamber (Baotou Kefa High Pressure Technology Co., Ltd, China) with a working volume of 10 L and diameter of 120 mm and a recommended maximum working pressure of 600 MPa. Samples were subjected to pressures in the range of 50–400 MPa. An example of the temperature and pressure profile of the sample during HHP treatment at 400 MPa for 10 min is shown in Figure 1. Distilled water has been used as pressure transmitting medium.

The experiment was divided into two parts as follows:

1. Optimization of HHP parameter to improve the OD of candied green plum:
   A central composite design (CCD) was applied to evaluate the singular, quadratic and interactive effects of treatment pressure (50–400 MPa) and time (1–30 min). The design with actual variables of the experiment is shown in Table 1. Solids Gain (SG) and Water Loss (WL) after 72 hr were used as processing responses. Every experiment was conducted in three replicates.
2. Comparison of different pre-treatments on the efficiency of OD and quality of the products:
   The perforated samples were divided into four groups to compare different treatments on the efficiency of OD and quality of the products:

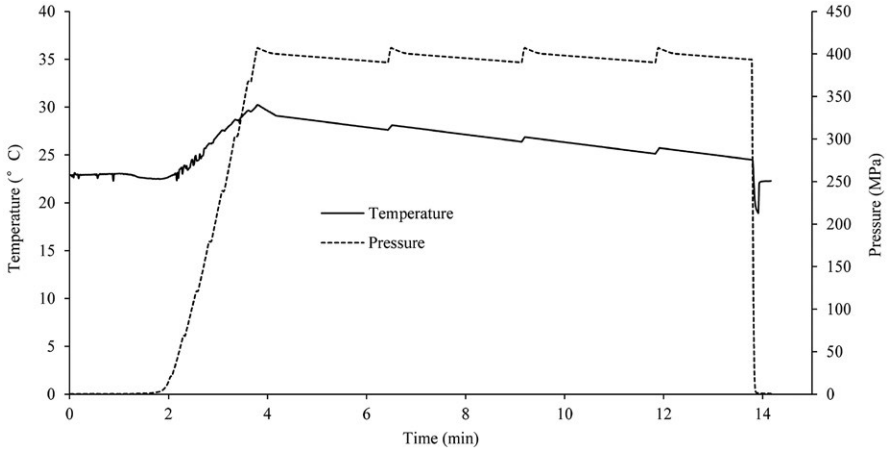

FIGURE 1 Temperature and pressure changes during HHP treatment (400 MPa for 10 min as an example)

a Untreated green plums were used as the control (C group).
b HHP treated samples (HHP group) were obtained according to the parameters and procedure chosen in the first part of the experiment (50 MPa for 1 min).
c Samples subjected to heating (H group) were obtained by immersion in boiling 40% sucrose solutions for 10 s followed by cooling down to room temperature (22°C), the total process time was about 20 min.
d The last group was treated accordingly to the traditional candied green plums procedure (T group): perforated green plums were dipped in 1% $CaCl_2$ and 5% NaCl solution for 10 hr in order to eliminate the bitter taste and harden the fruits, then immersed in boiling 40% sucrose solutions for 10 s and naturally cooled down to room temperature (22°C).

2.4 | Osmotic dehydration

After the pre-treatments, the plums were dipped in the 40% sucrose solution at room temperature (22°C) for a total of 5 days. The fruit-to-syrup ratio was set at 1:4 (w/w) (Tylewicz et al., 2017). The beakers were stirred twice a day to mix the solutions, and syrup concentration was adjusted to 40% every day by adding sucrose to the solutions. Samples were subjected to analytical determination after 0, 1, 2, 3, 4, 5 days. After removal from the syrup, the samples were quickly washed with distilled water to remove the adhered solution on the surface and blotted with absorbent paper.

2.5 | Mass transfer kinetic model of OD

At each measuring point, the samples were weighted by a technical balance (D&T, Tianjin, China), followed by determination of moisture content by placing the samples in an oven at 105°C until constant weight (Assis, Morais, & Morais, 2017).

The parameters WL and SG were determined through the following equations (Corrêa, Pereira, Vieira, & Hubinger, 2010):

$$WL = \frac{w_{w0} - W_w}{w_0} \quad (1)$$

$$SG = \frac{w_s - w_{s0}}{w_0} \quad (2)$$

where w_{w0} is the initial moisture content, w_w is the moisture content at time t, w_0 is the initial weight of the sample, w_s is the solids content at time t, and w_{s0} is the initial solids content of sample, all in g.

Mass transfer data were modeled according to the equation proposed by Palou, Lopez-Malo, Argaiz, and Welti (1994), using the Peleg's model (Peleg, 1988):

$$SGorWL = \frac{t}{k_1 + k_2 \times t} \quad (3)$$

k_1 and k_2 are the Peleg's constants for WL or SG.

The constant k_1 relates to the initial rate of the mass transfer, and vthe constant k_2 relates to equilibrium values, WL_∞ or SG_∞, as can be seen below:

TABLE 1 Central composite design (CCD) with coded and actual variables

	Coded variables		Actual variables	
Run	Factor 1	Factor 2	Pressure (MPa)	Time (min)
1	0	−1.41	225	1
2	0	0	225	16
3	1	−1	349	5
4	−1.41	0	50	16
5	−1	−1	101	5
6	1.41	0	400	16
7	1	1	349	26
8	0	0	225	16
9	0	1.41	225	30
10	0	0	225	16
11	−1	1	101	26
12	0	0	225	16
13	0	0	225	16

$$\left[\frac{d(\mathrm{SGorWL})}{dt}\right]_{t\to 0} = t\to 0\lim\left[\frac{d\left(\frac{t}{k_1+k_2\times t}\right)}{dt}\right] = \frac{1}{k_1} \quad (4)$$

$$\lim_{t\to\infty}(SGorWL) = \lim_{t\to\infty}\frac{t}{k_1+k_2\times t} = \frac{1}{k_2} \quad (5)$$

This kinetic model offers the advantage that by calculating the inverse of the constant (k_1 and k_2) it is possible to obtain the initial rate and the values at the equilibrium of mass transfer parameters (Sacchetti, Gianotti, & Dalla Rosa, 2001).

2.6 | Physico-chemical properties of green plums

The pH values were measured using pH meter (PHS-25, Leici Instrument factory, Shanghai, China).

Titratable acidity (TA) contents were measured by titration using sodium hydroxide (0.1 mol/L NaOH) to neutralize all the titratable protons, until reaching pH 8.1. Results were expressed as g/L of malic acid (Xie, Ye, Liu, & Ying, 2011).

2.6.1 | Color measurement

A Chroma Meter CM-700d reflectance colorimeter (Konica Minolta, Japan) was used to measure the CIE L^*, a^*, b^* values of samples. The color values were expressed as L^* (whiteness or brightness/darkness), a^* (redness/greenness) and b^* (yellowness/blueness). The total color difference ΔE was calculated by the equation below (Maskan, 2001) using fresh sample as reference:

$$\Delta E = \sqrt{\left(L^*-L_0^*\right)^2+\left(a^*-a_0^*\right)^2+\left(b^*-b_0^*\right)^2} \quad (6)$$

2.6.2 | Texture measurement

The texture of the green plums was evaluated by subjecting each sample to a penetration test using a Texture Analyzer (Stable Micro Systems, Surrey, U.K.), equipped with a 25 kg load cell and a cylindrical shape stainless steel probe of 2 mm diameter. The penetration test rate was 1 mm/min with and the penetration depth 0.2 cm.

From the obtained curves of force versus time, the hardness (kg) that represents the maximum force required to penetrate of the sample was extrapolated.

2.6.3 | Sensory analysis

Sensory analysis was performed after OD by a panel of 10 trained tasters (age 21–43 years old; 50% males and 50% females), who had been selected for their reliability, consistency and discriminating ability (ISO, 2012) and had former experience in the sensory assessment of fruits products, including green plums. The evaluation sheet included six parameters related to green color, brightness, hardness, sweetness, typical green plum odor and typical green plum flavor by a 0–9 rating scale. The reference score was built as follows: whole fruit of fresh green plums were used to define 9 points for green color, brightness and hardness; the puree of fresh green plums was used to define nine points for typical flavor and odor; the puree of fresh green plums containing 40% sucrose was used to define nine points for sweetness.

Samples were presented to panelists separately and in coded dishes.

2.7 | Statistical analysis

The CCD experiment design was given by Design Expert 8.0.5 (Stat-Ease, Inc., US). All determinations were carried out in triplicate. Data were analyzed with Statistics 22.0 (SPSS Inc., Chicago, IL, USA) by one-way analysis of variance to check the statistical significance of differences using the Tukey test ($p < 0.05$), followed by model fitting. The parameters to be used in the second part of the experiment were chosen on the basis of obtained results. The parameters of Peleg's model were calculated by Origin Lab 8.0 (Massachusetts, USA).

Correlations between the results of instrumental and sensory analysis were calculated by Pearson's correlation analysis.

3 | RESULTS AND DISCUSSION

3.1 | Optimization of HHP parameter

Table 2 shows the SG and WL after 72 hr OD process treated by HHP at different pressure (50–400 MPa) and time (1–30 min). Compare to control samples (0.1 MPa), the HHP treated samples obtained higher SG and WL, while there was no statistical difference among all the HHP treatments ($p < 0.05$). The optimization results given by Design Expert 8.0.5 also suggested that the applied pressures and times have similar effects on the mass transfer of green plums. For the industrial view on energy and time saving consideration, the lowest pressure and time (50 MPa for 1 min) have been selected as the HHP processing parameter for further studies.

When comparing the mass transfer efficiency with other studies that applied HHP as a pre-treatment for candied fruits and vegetables, the results were not all consistent. Nuñez-Mancilla, Pérez-Won, Uribe, Vega-Gálvez, and Di Scala (2013) found that at the range of applied pressure (100–500 MPa), there was no significant difference in the moisture content of strawberries after OD process. Dermesonlouoglou, Boulekou, and Taoukis (2008) found that increasing the pressure from 200 to 600 MPa did not increase the SG and WL of cherry tomatoes osmotically dehydrated for 6 hr. On the other side, in other studies an increment in the SG and/or WL of candied plant materials during OD pretreated with HHP was observed by increasing the working pressure (Rastogi, Angersbach, & Knorr, 2000; Rastogi, Angersbach, Niranjan, & Knorr, 2000; Rastogi & Niranjan, 1998; Verma et al., 2014). Nevertheless, these authors verified the efficiency of OD after a significantly shorter time (6–9 hr) compared to the period of time chosen in the present experiment (72 hr).

TABLE 2 Results (solids gain and water loss) of the CCD design*

Pressure (MPa)	Time (min)	Solids gain (%)	Water loss (%)
50	16	22.13 ± 0.77[a]	30.67 ± 0.66[a]
101	5	21.25 ± 1.24[a]	30.03 ± 0.86[a]
101	26	21.23 ± 0.67[a]	30.30 ± 1.15[a]
225	1	21.47 ± 0.86[a]	30.40 ± 0.53[a]
225	16	20.97 ± 1.33[a]	29.70 ± 0.75[a]
225	30	20.59 ± 0.45[a]	30.43 ± 0.81[a]
225	16	20.74 ± 0.96[a]	29.83 ± 0.76[a]
225	16	21.43 ± 1.21[a]	29.53 ± 1.36[a]
225	16	21.35 ± 1.07[a]	30.60 ± 0.36[a]
225	16	20.94 ± 2.17[a]	29.87 ± 0.75[a]
349	5	20.62 ± 0.55[a]	30.17 ± 0.60[a]
349	26	20.67 ± 0.48[a]	29.73 ± 1.11[a]
400	16	22.33 ± 0.73[a]	30.37 ± 0.83[a]
0.1	-	17.13 ± 0.21[b]	20.68 ± 1.00[b]

*Different letters indicate significant difference at $p < 0.05$.

3.2 | Mass transfer of OD green plums

The results of the non-linear regression of WL and SG data of the OD process of green plums are shown in Table 3 and Figure 2. The Peleg's model utilized to fit mass transfer parameters data over processing time showed high values of determination coefficients (R^2) in a range of 0.89–0.99 and relatively low RESM, indicating good adaptability of Peleg's model for the data fitting.

The curves showed in all samples a higher rate of SG or WL in the early stage of OD that decreased along the process. This phenomenon was commonly observed (Kowalska & Lenart, 2001; Santagapita et al., 2013; Tylewicz et al., 2011) and is mainly due to the higher osmotic driving force between the green plums and the hypertonic surrounding medium at the beginning.

It was evident from Figure 2 and from the values of $1/k_1$, that the pre-treatments, both thermal (H and T group) and non-thermal (HHP) had positive effects on SG and WL, making it possible to reduce the drainage time from more than 5 days to about 2 days. Generally the cell membranes of fruits exert high

TABLE 3 Fitted parameter of Peleg's model

	C	HHP	H	T
Solids gain				
R^2	0.99	0.93	0.91	0.89
RESM	0.0053	0.016	0.015	0.017
k_1	199.94 ± 20.07	53.93 ± 21.10	3.29 ± 1.28	2.91 ± 1.18
k_2	3.19 ± 0.25	3.67 ± 0.35	4.36 ± 0.17	4.36 ± 0.18
$1/k_1$	0.0050	0.018	0.30	0.34
$1/k_2$	0.31	0.27	0.23	0.23
Water loss				
R^2	0.99	0.96	0.96	0.96
RESM	0.0087	0.015	0.011	0.012
k_1	197.86 ± 1.97	3.42 ± 1.12	1.63 ± 0.32	1.60 ± 0.33
k_2	23.06 ± 0.27	3.41 ± 0.10	3.33 ± 0.07	3.35 ± 0.08
$1/k_1$	0.0050	0.29	0.61	0.62
$1/k_2$	0.043	0.29	0.30	0.30

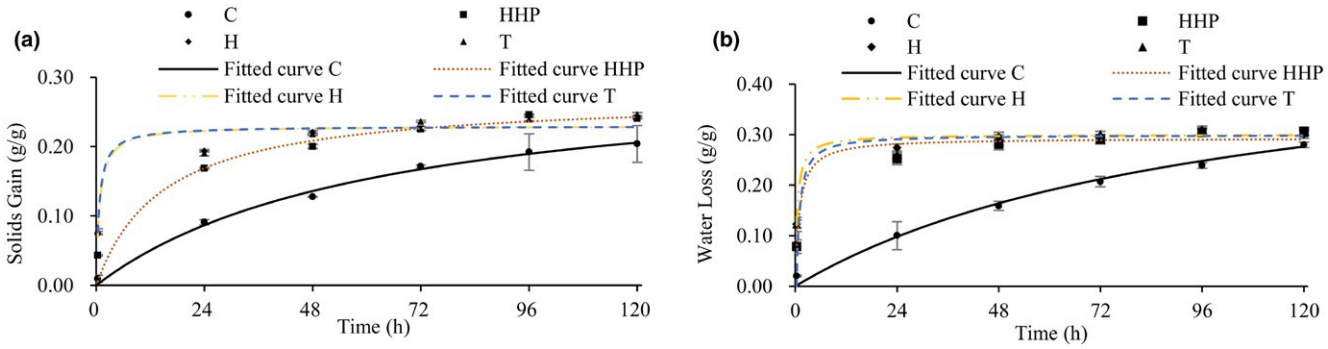

FIGURE 2 Experimental data and fitting curves of Peleg's model to SG (a) and WL (b)

resistance to mass transfer and slows down the OD rate (Allali, Marchal, & Vorobiev, 2010a). It has been reported that heating (Allali, Marchal, & Vorobiev, 2010b) and high pressure (Taiwo, Angersbach, Ade-Omowaye, & Knorr, 2001) can promote damages to the cell membranes altering their permeability, and thus, improving mass transfer. Considering the $1/k_1$ parameter, H and T samples showed similar values that were 60 times higher compared to sample C. HPP sample was characterized for a lower initial rate of mass transfer compared to C and T samples, but still, it exhibited a much higher rate compared to untreated ones.

For the $1/k_2$ value, the equilibrium rate of mass transfer parameters, the SG of $1/k_2$ in the different groups was similar, implying the heating and HHP would not affect the SG of the products. However, for what concerns about WL, the heating and HHP showed positive effect on WL of green plums.

3.3 | Physico-chemical properties of green plums

The pH and TA content changes of green plums during OD are shown in Table 4. The pH was around 3 in fresh green plums and treated ones, and basically remained stable during the OD.

The TA content dropped in all groups along with the OD. After the heating treatments (H and T), the green plums showed lower TA content compared to HPP and C samples, possibly due to the leakage of intracellular organic acid promoted by the damages to the cell membranes during the heating process. In all samples this parameter decreased during OD time, more rapidly at the beginning of the process and reaching a plateau after 48 hr for the H and T samples, and after 96 hr for the HPP and C samples. This decrease was probably due to the loss of soluble components like organic acids that can occur during osmotic processes (Bidaisee & Badrie, 2001).

Figure 3 shows the influence of the different pretreatments on the color of green plums. Immediately after the treatment, the color turned bright yellow in samples subjected to heating (H and T), bottle green in HHP sample, while remained light green in control one.

After the treatment, all of the three coordinates (L^*, a^*, and b^*) showed significantly higher value in H and T samples compared to fresh and control ones. The remarkable color change due to heating may be caused by degradation of the natural plant pigments as they are usually sensitive to heat (Delgado-Vargas, Jiménez, & Paredes-López, 2000; Shahid & Mohammad, 2013) coupled to the desegregation of tissue structure. On the opposite, the sample subjected to high pressure was characterized by a significantly lower L^* and b^* values and by a higher a^* value.

The reduced L^* value by HHP was probably caused by alteration of cell structure, which induces the leakage of cellular content and then lead to the refractive index variation of the tissue.

During the OD process, the main change in C sample was an increase of a^* value, while L^* and b^* were quite constant. The increase of the redness value in the control samples indicates a browning development that may be due to enzymatic activity, such as peroxidase and/or chlorophyllase that promote the degradation of color and the formation of dark pigments.

HPP sample showed a slight increase of L^* and b^* and a remarkable increase in a^* value. According to previous work, the application of pressures of 50 MPa was not sufficient for enzymatic inactivation (Hendrickx, Ludikhuyze, Broeck, & Weemaes, 1998) but may only cause reversible changes (Serment-Moreno, Barbosa-Cánovas, Torres, & Welti-Chanes, 2014). Some studies even found an increase in enzyme activity at low pressure (under 100 MPa) (Eisenmenger & Reyes-De-Corcuera, 2009; Yaldagard, Mortazavi, & Tabatabaie, 2008). Therefore, a browning development probably due to enzymatic activity similar to the C sample was observed in the HPP

TABLE 4 pH and titratable acidity (TA) content changes of green plums during OD*

Time (hr)	Fresh	C	HHP	H	T
pH					
0	3.05 ± 0.05[a]	3.06 ± 0.039 [Aa]	3.04 ± 0.060 [Aa]	3.07 ± 0.031[Aa]	3.04 ± 0.038 [Aa]
24		3.14 ± 0.041 [Aa]	3.08 ± 0.046 [Aa]	3.06 ± 0.027 [Aa]	3.06 ± 0.028 [Aa]
48		3.09 ± 0.072 [Aa]	3.11 ± 0.067 [Aa]	3.12 ± 0.047 [Aa]	3.08 ± 0.081 [Aa]
72		3.00 ± 0.067 [Aa]	2.99 ± 0.006 [Aa]	3.04 ± 0.022 [Aa]	3.03 ± 0.029 [Aa]
96		3.00 ± 0.017 [Aa]	2.94 ± 0.099 [Aa]	3.01 ± 0.013 [Aa]	2.99 ± 0.054 [Aa]
120		3.00 ± 0.038 [Aa]	2.95 ± 0.065 [Aa]	3.03 ± 0.033 [Aa]	2.98 ± 0.070 [Aa]
TA (g/L malic acid)					
0	5.91 ± 0.04[a]	5.55 ± 0.24 [Aa]	5.84 ± 0.14 [Aa]	4.78 ± 0.16 [Ab]	4.71 ± 0.13 [Ab]
24		4.30 ± 0.17 [Ba]	4.14 ± 0.15 [Ba]	3.10 ± 0.08 [Bb]	3.15 ± 0.12 [Bb]
48		4.04 ± 0.10 [Ba]	3.60 ± 0.17 [Ca]	2.79 ± 0.05 [BCb]	2.91 ± 0.07 [Bb]
72		3.34 ± 0.13 [Ca]	3.14 ± 0.17 [Da]	2.49 ± 0.20 [Cb]	2.52 ± 0.09 [Cb]
96		2.92 ± 0.15 [Da]	2.85 ± 0.14 [Da]	2.41 ± 0.08 [Cb]	2.53 ± 0.11 [Cb]
120		2.91 ± 0.05 [Da]	2.71 ± 0.03 [Da]	2.36 ± 0.02 [Cb]	2.42 ± 0.03 [Cb]

*Different capital letters indicate statistical difference within the same sample during storage and different lowercase letters indicate statistical difference in different samples at the same storage time ($p < 0.05$).

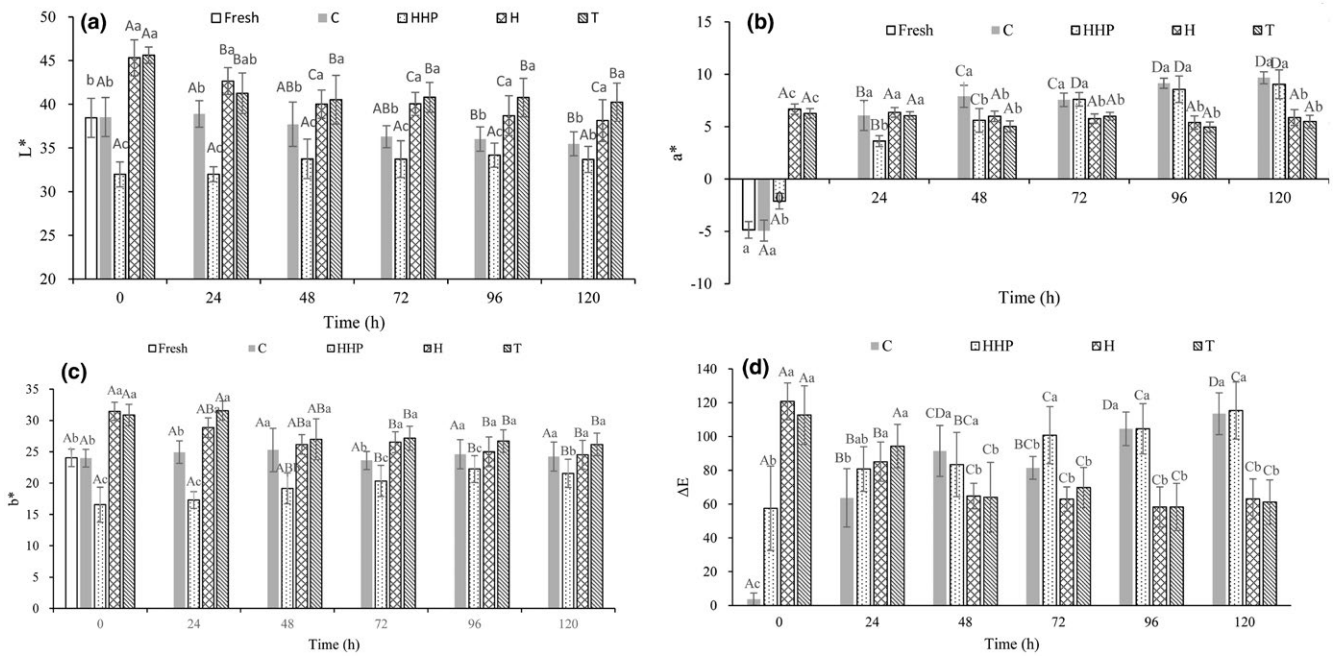

FIGURE 3 Modifications of color parameters, L^* (a), a^* (b), b^* (c) and ΔE (d) of green plums during OD. Bars labeled with different capital letters indicate statistical difference within the same sample during storage, and different lowercase letters indicate statistical difference in different samples at the same storage time ($p < 0.05$)

sample. At the end of the OD process, no significant differences were observed for all three color parameters between C and HPP samples.

On the opposite, in H and T samples the redness parameter was maintained constant throughout the OD process, probably for the enzymatic inactivation promoted by the heat treatment.

The total color change was calculated using fresh sample as reference. Immediately after the treatment, HHP showed a lower value compared to H and T samples. However, during the OD process, the total changes decreased in heating groups (H and T) because of the decrease in L^* and b^* value, while they increased in control and HHP groups. After 48 hr HPP and C samples showed higher values compared to H and T, and afterward, the gap widened. No significant differences were observed between HPP and C samples starting from 24 hr onwards.

Figure 4 reports the firmness changes of green plums during osmosis process. Heating showed a strong negative influence on the firmness of this fruit (sample H); although the addition of calcium salt in the dipping solution promoted a better retention of texture (sample T), firmness was still considerably lower compared to the control after heating. No differences compared to C were observed after the HHP treatment.

After the first 24 hr of the process, firmness significantly decreases in HHP and C samples but then was maintained stable until the end. Texture decrease along with the OD process was also found in previous studies (Castelló, Igual, Fito, & Chiralt, 2009; Torreggiani, Forni, Maestrelli, & Quadri, 1999).

No significant differences were observed between HHP and Control groups at all measurement points, suggesting that the application of 50 MPa of pressure for 1 min does not influence the firmness of green plums.

In H and T samples, firmness values were constant throughout the osmotic process and always lower compared to C and HPP samples.

3.4 | Sensory tests

Figure 5 shows the results of sensory analysis performed after the 5th day of OD of the candied green plums.

Appearance was evaluated through the parameters green color and brightness. Compared to the control, HPP sample scored similar values for green color and slightly but significantly higher for brightness. H and T samples instead appeared remarkably less green and bright.

The panel perceived a strong decrease in firmness in the sample subjected to heating (H), while no differences were observed in the sample subjected to high pressure (HPP). The addition of calcium prior to the heat treatment was found to increase the perceived firmness in sample T compared to sample H.

As expected, the heat treatment seemed to have promoted also a loss of the typical green plum odor and flavor, both in H and T samples. On the contrary, the application of HPP seemed to indicate a good preservation of these parameters, showing sensory score values similar to the control.

The perception of sweetness was found higher in the T sample that scored a value almost at the top of the range. H and HPP samples had slightly lower values but non-significantly different, while C samples was perceived as less sweet. This result was explained by

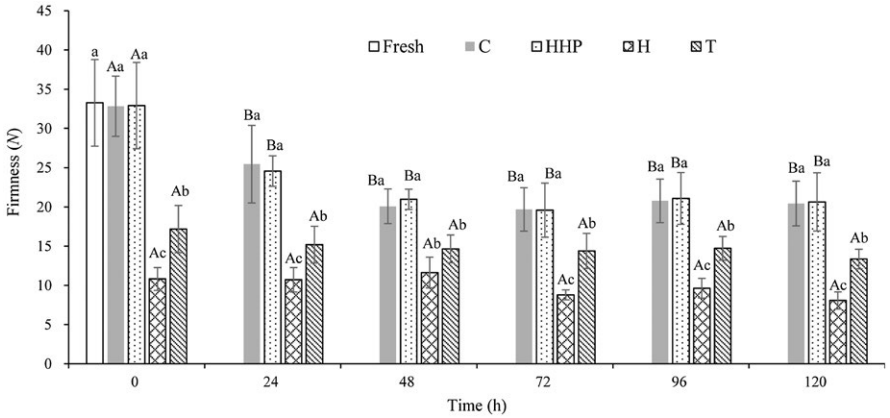

FIGURE 4 Variations of firmness values of green plums during OD. Bars labeled with different capital letters indicate statistical difference within the same sample during storage, and different lowercase letters indicate statistical difference in different samples at the same storage time ($p < 0.05$)

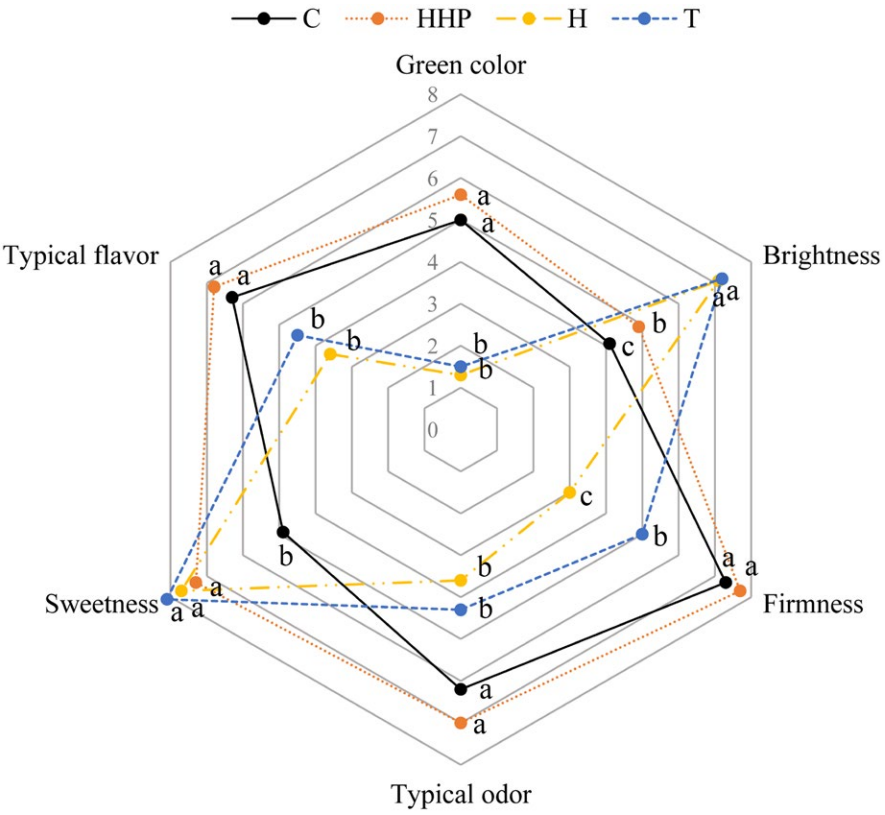

FIGURE 5 Sensory scores of candied green plums after OD for 120 hr. Each point labeled with different letters indicated the statistical difference from each other

the lower SG of the untreated green plums compared to the treated ones.

The Pearson correlation analysis was performed to correlate results obtained with instrumental and sensory analysis (Table 5).

Titratable acidity was found positively with color, firmness, typical odor and flavor and negatively to sweetness. Solid gain was positively correlated to the perception of sweetness. Concerning color parameters, only L^* and a^* were found correlated to the sensorial perception, while b^* showed no significant value. The changes in color were indeed mainly related to a loss of brightness and green color.

The measurement of firmness with the two methods was positively correlated, showing that changes due to the considered treatments were well perceived by sensory tests. Moreover, instrumental firmness was also positively correlated to the sensorial parameters of green color, typical odor, and flavor. This result suggested a close

TABLE 5 Pearson correlation coefficient between instrumental and sensory analysis

	Sensory analysis					
Instrumental analysis	Green color	Brightness	Firmness	Typical odor	Sweetness	Typical flavor
Titratable acidity	0.86*	−0.51	0.83*	0.79*	−0.77*	0.75*
Solids gain	−0.64	0.54	0.48	0.58	0.78*	−0.51
L^*	−0.48	0.73*	−0.45	−0.62	0.45	−0.54
a^*	0.89*	−0.81*	0.77*	0.59	0.61	0.58
b^*	−0.45	0.34	−0.24	0.45	0.23	−0.42
Firmness	0.85*	−0.48	0.90*	0.87*	−0.51	0.77*

*Superscript labels indicate statistical significance ($p < 0.05$).

relationship among structural, physical, and aromatic characteristics of the product.

4 | CONCLUSIONS

The application of HHP was evaluated as an alternative to heat treatment prior to OD for the production of candied green plums.

Kinetic of mass transfer showed that HHP resulted an effective method to accelerate the OD process of candied green plums, allowing to reduce the dehydration time from more than 5 days to about 2 days, even though it was not comparable to the traditional heating method. Nevertheless, respect to heating, HPP allowed to maintain higher firmness values and a greener color of the final product.

Sensory analysis has confirmed the data obtained by instrumental analysis of firmness and color, also indicating that HHP treated samples retained the typical green plums odor and flavor of the fresh sample.

The results obtained in the present study suggest a potential application of HHP as a pre-treatment for obtaining candied green plums characterized by high quality level. A wider consumer test is needed for confirming the acceptability of the product compared to the traditional one. Finally, the effect of the HPP treatment on the nutritional value of the products should be investigated in future studies.

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Paper III: Effect of High Hydrostatic Pressure (HHP) on the antioxidant and volatile properties of candied wumei fruit (Prunus mume) during osmotic dehydration

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ORIGINAL PAPER

Effect of High Hydrostatic Pressure (HHP) on the Antioxidant and Volatile Properties of Candied *Wumei* Fruit (*Prunus mume*) During Osmotic Dehydration

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Abstract
Candied *wumei* fruit (*Prunus mume*) is a traditional product in Eastern Asian countries generally obtained by a long osmotic dehydration (OD) process. This study evaluated the effect of the application of high hydrostatic pressure (HHP) and heating pre-treatments to accelerate the OD process on some bioactive compound content, antioxidant activity, and volatile profile of *wumei* fruit. Whole fruits were subjected to HPP treatment (50 MPa for 1 min) and heating (100 °C for 1 min) and then to OD in a 40% sucrose solution for 5 days. Results showed that both heating and HPP pre-treatment increased mass transfer in a comparable way; however, HPP allowed a significant higher retention of antioxidant compounds and activity compared with the heating treatment, resulting in similar values to the untreated product. Moreover, HPP promoted the release of various volatile components resulting in a richer volatile profile compared with both control and heat-treated samples. Hence, HPP showed good potentiality as an alternative non-thermal pre-treatment for the production of candied *wumei* fruit characterized by high nutritional and sensorial properties.

Keywords *Prunus mume* · Antioxidant · Volatile · High hydrostatic pressure · Heating · Osmotic dehydration

Introduction

Wumei (*Prunus mume*) is a deciduous tree of the Rosaceae family (Chen et al. 2017). The fruit is rich in organic acids, edible fiber, minerals, and phenolic compounds, and it is popular in Eastern Asian countries (Gao et al. 2004). It has been regarded as both food and medicine material since 1985 by the Ministry of Health of the People's Republic of China because of its therapeutic benefits, which include antitussive, expectoration, antiemetic, antidiarrheal, anthelmintic, and antipyretic activities (Yan et al. 2014). It has traditionally been used as herbal medicine for coughs and dyspepsia (Jung et al. 2010), stomach and intestine disorders (Miyazawa et al. 2003), fatigue, diarrhea, and fever (Shirasaka et al. 2005). However, because of the presence of cyanoglycoside, prunasin, amygdalin, and a very high organic acid content, the fruit usually have an unpleasant taste defined as sour, bitter, and astringent (Bolarinwa et al. 2014), thus, it is generally processed before consumption. One of the most popular *wumei* products in the market in Eastern Asian countries is candied *wumei* that is generally obtained using osmotic dehydration (OD). According to "The white paper on the development of Chinese candied fruit industry in 2016," the annual output value of candied fruit in China is about $23.5 billion, with an annual average growth rate of 21.78% in the last decade. Candied *wumei* is one of the main products in this industry.

OD is an effective process to used partially remove water from fruit tissue and to enrich it with soluble solids by its immersion in hypertonic aqueous sugar solutions (Nowacka et al. 2014). Because OD is a

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relatively slow process (Souraki et al. 2015), the traditional procedure involves a pre-treatment consisting in perforating the fruit with needles. Nevertheless, to obtain the saturation of the fruit with sugars, long times are required, up to more than 5 days. The application of heating is therefore widely applied in industrial processing in order to speed up the candying process, although it promotes a loss of quality and of bioactive compounds. The use of a non-thermal pre-treatment able to improve mass transfer as an alternative to heating could allow a better retention of the qualitative and nutritional properties of the final product.

High hydrostatic pressure (HHP) is a non-thermal technology that has been shown to increase OD rate in some fruits and vegetables such as pineapple, strawberry, banana, etc., permitting the quality improvement of the final products (Rastogi and Niranjan 1998; Verma et al. 2014; Nuñez-Mancilla et al. 2011; Núñez-Mancilla et al. 2014). Hence, it could be a promising method to accelerate dehydration of *wumei* fruit to reduce processing time.

The aim of this work was to evaluate the application of HHP as a pre-treatment to OD of *wumei* fruit and its effect on the nutritional and aromatic properties of the final product, compared with the traditional use of heating.

Materials and Methods

Raw Materials and Reagents

Wumei fruits (*Prunus mume* Siebet. Zucc.) were purchased from an orchard located in Shaoxing (Zhejiang Province, China). The altitude and latitude of the orchard is 500–600 m and 29° North. Fruits harvested at commercial maturity and with uniform shape and size (about 20 g per each fruit) were selected and stored at 4 °C and 100% relative humidity (RH) until use.

Ascorbic acid, gallic acid, catechin standard, 2,2-diphenyl-1-picrylhydrazyl (DPPH), and 2,2′-azino-bis (3-ethylbenzothiazoline-6-sulphonic acid) (ABTS) were purchased from Sigma (Sigma Corporation, Japan). The chemicals used for HPLC were of chromatographic purity grade. Other chemicals were of analytical purity grade.

Sample Preparation

The hypertonic sucrose solutions were prepared by dissolving commercial sucrose in distilled water at a concentration of 40% (*w*/*w*).

The *wumei* were perforated by a matrix of needles (1 mm of diameter, 25 needles/cm^2) in order to accelerate mass transfer, as shown in Fig. 1. According to the traditional process of candied *wumei*, cores of the fruit were not removed.

HHP and Heating Treatments

For the HHP treatment, the perforated *wumei* samples were packed in PE/PA packaging bags immersed in hypertonic sucrose solutions (40%, *w*/*w*) with a fruit-to-syrup ratio of 1:4 (*w*/*w*). Before the HHP treatment, the bags were sealed by a thermal sealer (300A, Bake Easy, China) after manual removal of the residual gas. The packed *wumei* were subjected to HHP treatment in a pressure chamber (Baotou Kefa High Pressure Technology Co., Ltd., China) with a working volume of 10 L and diameter of 120 mm and a recommended maximum working pressure of 600 MPa. Samples were subjected to pressure at 50 MPa for 1 min. These parameters were chosen on the basis of a preliminary study that showed that by increasing working pressure and time, no further gain was observed during the OD process. The maximum temperature reached by the samples during pressurization was 24 °C. Distilled water was used as a pressure-transmitting medium.

The heating treatment (*H*) was carried out by immersing the perforated *wumei* in boiling 40% sucrose solutions (fruit-to-syrup ratio of 1:4, *w*/*w*) for 1 min.

For the control, perforated *wumei* were dipped in sucrose solutions (fruit-to-syrup ratio of 1:4, *w*/*w*) at room temperature (22 °C) for 1 min.

After the pre-treatments, fruits were dipped in the 40% sucrose solution at room temperature (22 °C) for a total of 5 days. The fruit-to-syrup ratio was set at 1:4 (*w*/*w*). The beakers were stirred twice a day to mix the solutions, and syrup concentration was adjusted every day by adding sucrose to the solutions. Samples were subjected to analytical determination after 0, 1, 2, 3, 4, and 5 days of treatment. After removal from the syrup, the samples were quickly washed with distilled water to remove the adhered solution on the surface and blotted with absorbent paper.

Mass Balance

At each measuring point, the samples were weighed by a technical balance (D&T, Tianjin, China) and moisture content was determined by drying in an oven at 105 °C until constant weight. The weight and moisture content at every measurement point were determined at least in three replicates.

The parameters water loss (WL) and solid gain (SG) were determined through the following equations (Corrêa et al. 2010):

$$\mathrm{WL} = \frac{w_{w0} - w_w}{w_0} \quad (1)$$

$$\mathrm{SG} = \frac{w_s - w_{s0}}{w_0} \quad (2)$$

where w_{w0} is the initial moisture content, w_w is the moisture content at time (t), w_0 is the initial weight of the sample, w_s is the solid content at t, and w_{s0} is the initial solid content of sample.

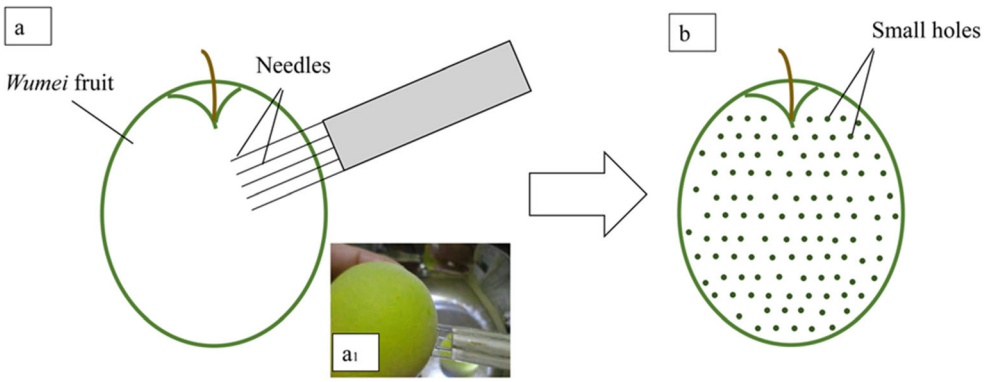

Fig. 1 **a**, **b** Schematic representation of sample preparation (a_1, operation photo)

Bioactive Compound Content

Ascorbic Acid Content

The ascorbic acid content was measured according to Vangdal et al. (2017), with minor modifications. After removal of cores, 5 g of sample was grinded with 10 mL pre-refrigerated (4 °C) 2% meta-phosphoric acid (*w*/*v*). Then sample was extracted in an ultrasonic bath (20 kHz, KQ2200DV, Kunshan Ultrasonic Instrument Co., Ltd., China) for 5 min and centrifuged for 10 min at 15,000×*g* at 4 °C. The supernatant was filtered by a 0.45-μm aqueous filter membrane before HPLC determination.

An Intersil® ODS-3 column (250 × 4.6 mm i.d., 4 μm particle size, GL Sciences Inc., Japan) was used for HPLC analysis (Agilent 1100, USA). Twenty microliters of sample or ascorbic acid standard (1–100 μg/mL) was injected with an autosampler; 0.02 M orthophosphoric acid solution was used as mobile phase, at a flow rate of 0.7 mL/min. The analysis was carried out at 25 °C. UV detector at 245 nm was used to detect ascorbic acid. The results were express as ascorbic acid per 100 g fresh weight (FW).

Preparation of Extracts

The extracts used for total phenolic and flavonoid content, radical scavenging activities for DPPH, and ABTS were prepared according to Bouayed et al. (2011). Briefly, the samples were first freeze dried (FD-1A-50, TENLIN Instrument, Jiangsu, China) for 24 h until constant weight, followed by grinding to powder. Two grams of dry sample was added to 10 mL of pre-refrigerated (4 °C) 80% methanol (*v*/*v*) into 50 mL centrifuge tubes, extracted in an ultrasonic bath (20 kHz, KQ2200DV, Kunshan Ultrasonic Instrument CO., LTD., China) for 20 min and centrifuged at 15,000×*g* at 4 °C for 10 min (Sorvall ST 16R Centrifuge, Thermo Fisher Scientific Inc., USA). The supernatant was collected, and the residue was re-extracted twice with 5 mL of 100% methanol. The obtained supernatants were combined, and the final volume was adjusted to 25 mL using 100% methanol. The extracts were stored at − 80 °C until further analysis. Three independent extractions were prepared for each sample.

Total Phenolic Content

Total phenolic content was determined with Folin–Ciocalteu's phenol reagent by a spectrophotometric method (Bouayed et al. 2011). One-milliliter extract or gallic acid standard (0–1000 μg/mL) was added into a 50-mL centrifuge tube together with 8 mL distilled water and 1 mL Folin–Ciocalteu's phenol reagent. After 5 min, 10 mL of 10% sodium carbonate solution (*w*/*v*) was added and mixed. The mixture was incubated for 60 min at room temperature (22 °C). The absorbance was read at 750 nm. The total phenolic content was expressed as gallic acid equivalents (GAE) per 100 g dried weight (DW).

Total Flavonoid Content

Total flavonoid content was evaluated according to Bouayed et al. (2011). One milliliter of extract or catechin standard (0–50 μg/mL) was added to a 15-mL centrifuge tube and mixed with 5 mL distilled water and 0.5 mL 5% sodium nitrate solution (*w*/*v*). After 5 min at room temperature, 0.5 mL of 10% aluminum chloride (*w*/*v*) was mixed and incubated for 6 min. After that, 2 mL of 1 M NaOH was added. The absorbance was evaluated at 510 nm. The total flavonoid content was expressed as catechin equivalents (CE) per 100 g DW.

Antioxidant Capacity Determined by DPPH Scavenging Activity

The DPPH scavenging activity was determined based on Du et al. (2009), with small modifications; 0.1 mL of sample extract or Trolox standard (0–1 mg/mL) was mixed with 2 mL of a 6.25×10^{-5} M solution of DPPH in methanol. The mixture was incubated in the dark at room temperature for 30 min. The absorbance was evaluated at 517 nm. The DPPH scavenging activity was expressed as Trolox equivalent capacity.

Antioxidant Capacity Determined by ABTS Radical Scavenging Activity

The ABTS scavenging activity was determined based on Du et al. (2009), with small modifications. ABTS radical was produced by the reaction of 7 mM ABTS solution with 2.45 mM potassium persulfate and incubation in the dark at room temperature for 12–16 h. Fresh solution of ABTS radical was diluted to an absorbance of 0.70 ± 0.02 at 734 nm with phosphate-buffered saline (pH 7.4); 0.1 mL of extract or Trolox standard (0–1 mg/mL) was mixed with 2 mL diluted ABTS radical solution. After 6 min incubation in the dark at room temperature, the absorbance was measured at 734 nm. The ABTS radical scavenging activity was expressed as Trolox equivalent capacity.

Volatile Profile

Extraction of Volatiles

The extraction of volatile compounds was conducted by solid phase microextraction method (SPME) as described by Gokbulut and Karabulut (2012), with minor modifications. After removal of the cores, the fruits were cut into small pieces. Three-gram samples were placed into a 15-mL headspace vial. The volatile components were extracted using a Carboxen-polydimethylsiloxane (CAR/PDMS, 75 μm) fiber for 60 min at 40 °C.

GC-MS Conditions

The analysis was carried out with an Agilent 6890N gas chromatograph system (Agilent Technologies, USA) coupled to an Agilent 5975 quadrupole inert mass selective detector.

BP-20 fused silica capillary column (60 m × 0.25 mm ID × 0.25 μm film thickness) was used to separate the aroma components. Helium was employed as the carrier gas, at a flow rate of 0.8 mL/min. The temperature of the injector and transfer line of the MS detector were set at 160 and 265 °C. The column temperature was initially set at 50 °C and maintained for 10 min, and then increased gradually to 160 °C at a rate of 5 °C/min, kept at 160 °C for 2 min, and finally brought to 280 °C at a rate of 5 °C/min. Electron ionization system of electrons with ionization at 70 eV energy was applied. The components were identified by comparison of their relative retention times and mass spectra with those of the standard (for major components)—NIST library data of the GC-MS system and the relevant literature data. The amount of each volatile compound was quantified based on their peak areas. Two analyses were performed for each sample.

Statistical Analysis

At each measurement point, mass balance parameters were determined in triplicate. Antioxidant content and antioxidant activities were measured in triplicate on three independent extracts for each sampling point (a total of nine measurements). The determination of volatile compounds was conducted in duplicates. One-way analysis of variance (ANOVA) by Tukey test ($p < 0.05$) was applied to check the statistical significance of differences among different treatments and different OD process times with Statistics 22.0 (SPSS Inc., Chicago, IL, USA).

Results

Mass Balance

Table 1 reports WL and SG measured during OD in the different samples. At all sampling times, *H* samples showed significantly higher values for both parameters compared with the control sample, in particular in the first days of the process. HPP samples showed intermediate values between *C* and *H* during the first 2 days of OD; afterwards, it reached the same levels of WL and SG of *H* samples.

At the end of the process, WL and SG for the control samples were respectively 0.26 ± 0.01 and 0.20 ± 0.03 g/g. These values were reached by HPP and heated samples already after 1 or 2 days, indicating the possibility of HHP to reduce the OD time from more than 5 days to about 2 days.

Ascorbic Acid Content

The ascorbic acid content is shown in Fig. 2. In fresh samples, a value of 7.34 ± 0.56 mg/100 g FW was observed, which was similar to values found for various other cultivars of *Prunus* fruit (Gil et al. 2002).

Immediately after the treatments, the ascorbic acid content did not show any variation in the control and the HHP sample compared with the fresh one, while heating promoted a significant drop of ascorbic acid content of 17.44%. During the 5 days of OD process, the ascorbic acid content decreased in all groups. In samples subjected to heating, the higher loss was observed during the first day, decreasing from 6.16 ± 0.54 to 3.19 ± 0.26 mg/100 g and then remained constant until the end of the process. On the other hand, for HPP and control samples, the decrease was slower and reached a stable value after 3 days. At the end of OD process, HHP and control samples showed an ascorbic acid content that was 43.78% higher compared with the *H* sample.

Table 1 Water loss (g/g) and solid gain (g/g) measured in samples subjected to different pre-treatments during OD

OD time (day)						
Samples	0	1	2	3	4	5
Water loss (g/g)						
C	0.0020 ± 0.0004c	0.10 ± 0.027c	0.16 ± 0.0089b	0.21 ± 0.010b	0.24 ± 0.0054b	0.26 ± 0.0054b
HPP	0.078 ± 0.013b	0.25 ± 0.011b	0.28 ± 0.011a	0.29 ± 0.010a	0.30 ± 0.0071a	0.31 ± 0.017a
Heating	0.12 ± 0.0078a	0.27 ± 0.017a	0.29 ± 0.0051a	0.29 ± 0.011a	0.31 ± 0.0080a	0.30 ± 0.019a
Solid gain (g/g)						
C	0.0094 ± 0.0051c	0.091 ± 0.0036c	0.13 ± 0.014c	0.17 ± 0.021b	0.19 ± 0.026b	0.20 ± 0.026b
HPP	0.043 ± 0.0011b	0.17 ± 0.0081b	0.20 ± 0.013b	0.22 ± 0.019a	0.25 ± 0.010a	0.24 ± 0.011a
Heating	0.074 ± 0.0016a	0.19 ± 0.098a	0.22 ± 0.016a	0.23 ± 0.027a	0.25 ± 0.011a	0.24 ± 0.012a

Different letters indicate statistical difference ($p < 0.05$) at the same OD time

Total Phenolic Content and Flavonoid Content

The evolution of total phenolic and flavonoid contents of *wumei* subjected to different pre-treatment during OD process is shown in Fig. 3. Fresh sample content was 354.99 ± 19.55 mg GAE/100 g DW and 45.47 ± 2.97 mg CE/100 g DW, for respectively total phenolic content (TPC) and flavonoid content (FC). Results were in a similar range found by other authors (Arion et al. 2014).

After HHP, a significant increase of TPC and FC of respectively 13.38 and 7.18%, was observed. On the contrary, the application of heating showed a negative influence on both parameters, decreased by 14.03 and 12.70% respectively compared with the control samples. During the OD process, a similar trend as ascorbic acid was observed, as a general decrease occurred in all samples. However, control and HPP were characterized at all times by similar values higher compared with *H* samples. In particular, at the end of the process, TPC and FC were 51.97 and 74.75% higher in HPP compared with *H* samples.

Antioxidant Activity

The antioxidant activity was estimated as free radical scavenging activities of DPPH and ABTS. The results are shown respectively in Fig. 4I, II. For the fresh samples, the DPPH and ABTS scavenging activities were 3004.45 ± 295.31 and 1186.15 ± 95.06 μg/g DW Trolox equivalent, respectively.

The trends of free radical scavenging activity were similar as the antioxidants described above: compared with the control, antioxidant activities measured with both methods were higher in HHP but lower in the *H* sample after the treatment. During OD, a progressive reduction was observed in all samples. At the end of the process, HHP samples were characterized by values 86.59 and 53.28% higher compared with *H* samples for respectively DPPH and ABTS scavenging activity.

Volatile Profile

An example of the characteristic GC-qMS profile of *wumei* fruit subjected to the different pre-treatment is shown in Fig. 5. After HHP treatment, a larger number of peaks appeared in the profile.

Table 2 reports the detail of identified components for each sample before and after OD, indicating the peak area and the relative amount (%). In detail, a total of 31 compounds were identified in fresh and control samples, which included 12 esters, 9 aldehydes, 3 terpenoids, 3 alcohols, 3 ketones, and 1 lactones, as reported in Table 2. The main detected

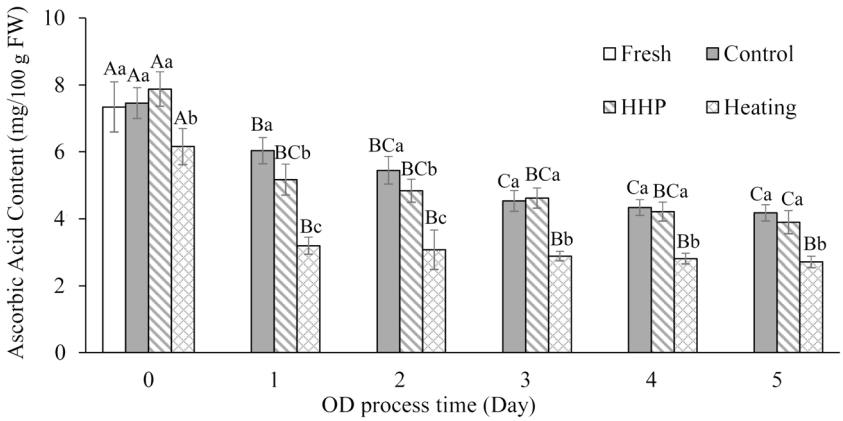

Fig. 2 Ascorbic acid content during OD process. Capital letters indicate statistical difference in a same sample in different OD sampling time; lowercase letters indicate statistical difference among different samples at the same sampling time ($P < 0.05$)

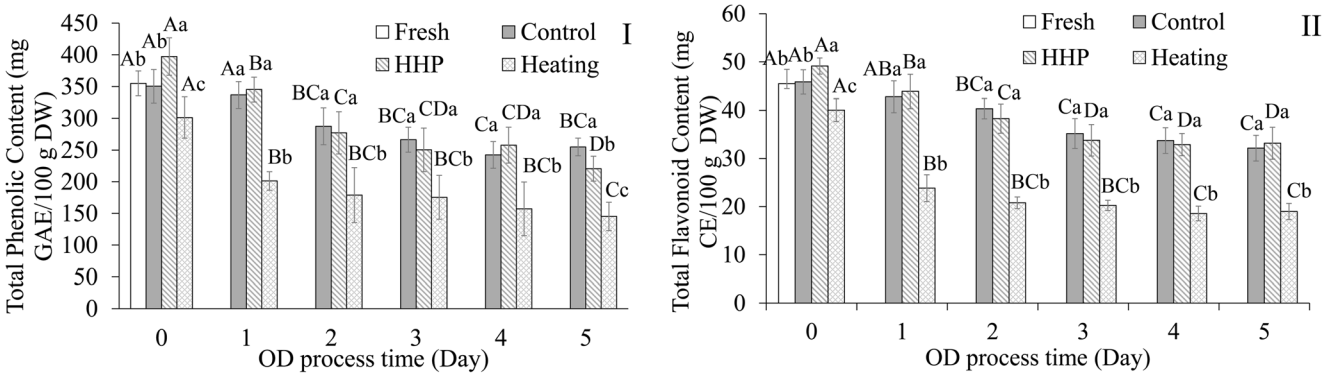

Fig. 3 Total phenolic (**I**) and flavonoid (**II**) content during OD process. Capital letters indicate statistical difference in a same sample in different OD sampling time; lowercase letters indicate statistical difference among different samples at the same sampling time ($P < 0.05$)

compounds were linalool (21.96–22.4%), hexyl acetate (14.54–17.81%), and ethyl acetate (8.92–10.25%). These findings are in agreement with Miyazawa et al. (2009).

After the HHP treatment, the number of volatile compounds increased from 31 to 64, which included 28 esters, 14 aldehydes, 9 terpenoids, 3 alcohols, 8 ketones, 1 lactone, and 1 alcane. The total amounts (peak area/weight) of compounds was increased by more than 10-fold compared with the untreated sample. The higher increase was observed for esters and aldehydes, which respectively increased by about 11-fold and 15-fold. The major detected components were 4-penten-1-yl acetate (10.95%), (*E*)-2-hexenal (10.67%), and 1-hexanol (9.68%). However, the relative amounts of some components, like thujopsene and linalool were decreased after HHP.

After the heating treatment, 27 volatile substances were detected: 7 esters, 9 aldehydes, 6 terpenoids, and 5 alcohols. The total amount of detected compounds (total peak area) was about 3-fold higher compared with the control. Also, there was a change in the relative proportion of the different compounds. First, one compound alone, 1-hexanol, accounted for 70% of the total quantity of detected volatile compounds. Some compounds were not detectable any longer, like ethyl butyrate, ethyl octoate, 1-nonanol, (*E*)-β-ionone, while many components were detected in lower amounts, like ethyl acetate, (*Z*)-3-hexenal and linalool. However, an increase of butyl butylate, hexanal, octanal, non-anal aldehyde, cuminol, and 1-hexanol was observed. Finally, components such as (*E*)-2-hexenal, D-limonene, *p*-cymene, α-damascenone, and 3-octanone, that were not detected in the control sample, appeared after the heating treatment.

The OD process promoted a change in the volatile profile of all samples, with a general decrease of the total amount (total area/weight) and of the number of identified components. In particular, 42 in HHP and 15 and 24 components were identified in HHP, *H*, and control samples, respectively. In the control group, most of the compounds were decreased along with the OD process time, but there were some compounds that stayed at the same level as before, like butyl butylate, ethyl (4*E*)-4-decenoate, decanal, and cuminol. Some compounds were even increased after the OD process, like benzaldehyde, (*E*)-2-hexenal, and 1-hexanol. After OD, HPP sample still showed the highest amount of volatiles.

Discussions

This study confirmed the ability of HPP treatment to increase mass transfer in fruit tissue during osmotic dehydration, due to

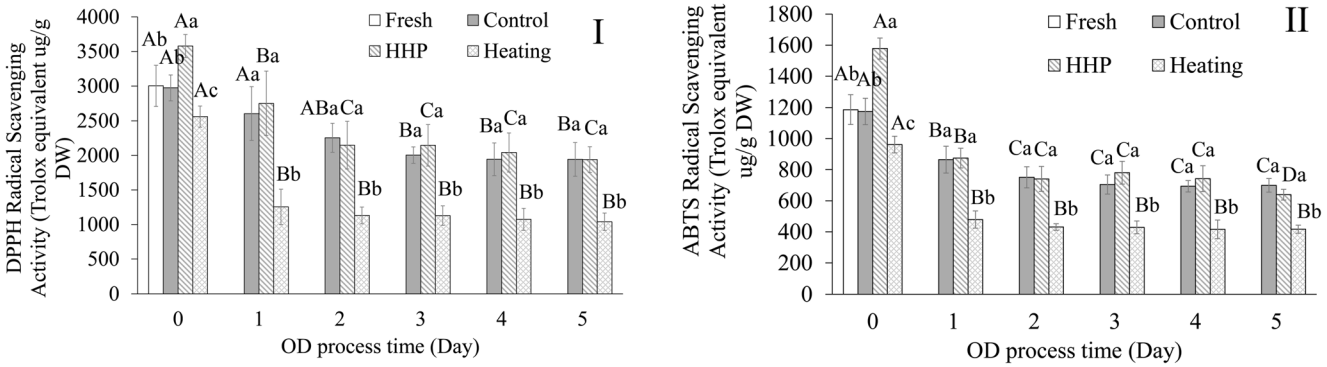

Fig. 4 Antioxidant activities for radical scavenging activity of DPPH (**I**) and ABTS (**I**) during OD process. Capital letters indicate statistical difference in a same sample in different OD sampling time; lowercase letters indicate statistical difference among different samples at the same sampling time ($P < 0.05$)

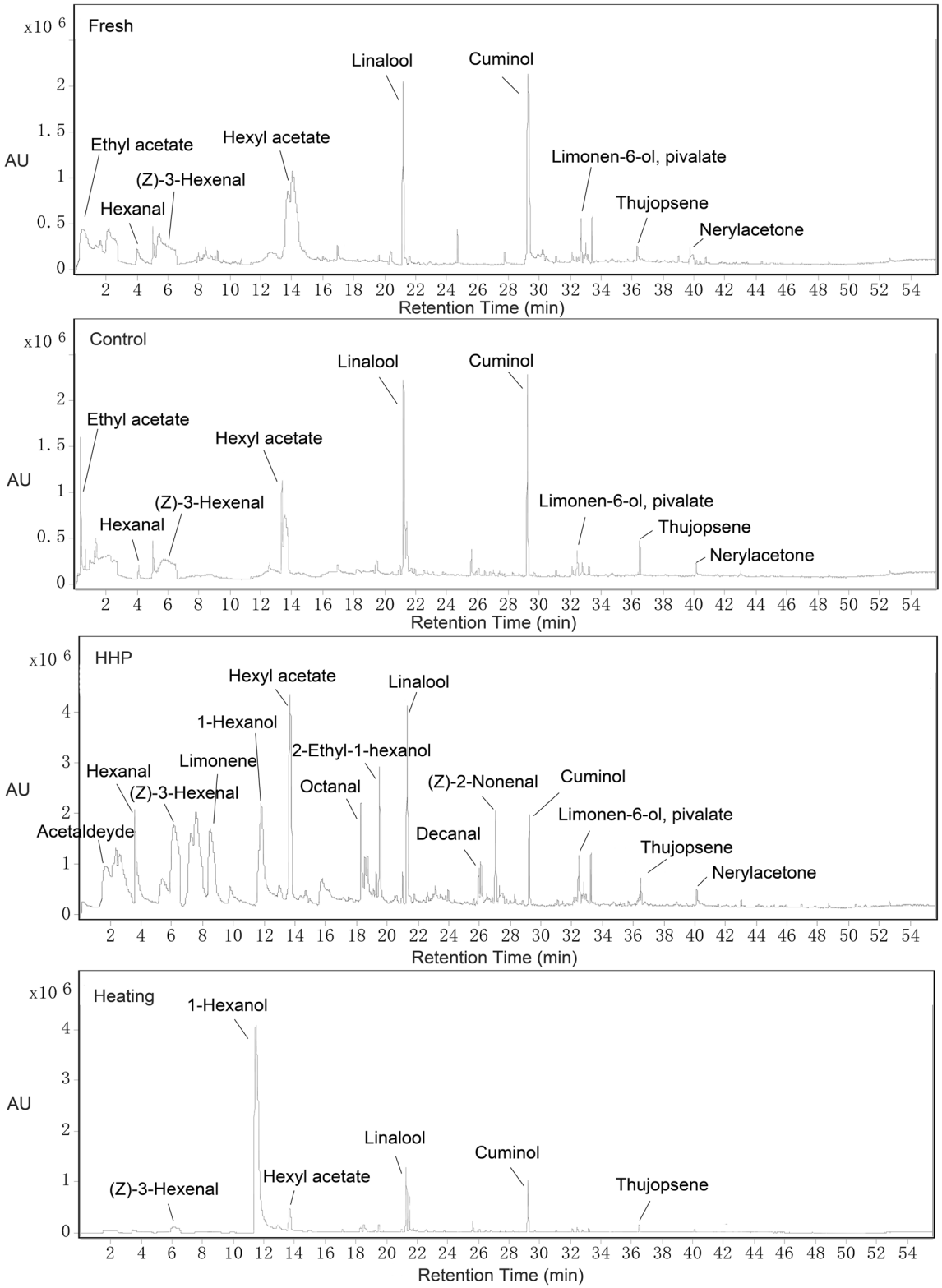

Fig. 5 Mass chromatogram of volatile compounds of *wumei* fruit by different pre-treatments; for detailed peak identifications, see Table 2

the alteration of cell membrane permeability (Taiwo et al. 2001; He et al. 2016), showing good potential for reducing processing time. Mass transfer results were similar compared with the traditional pre-treatment with heat. However, heating often leads to loss in nutritional and sensorial properties decreasing the overall quality of the final product.

Phenols and flavonoids are functional compounds in fruits and vegetables (Nayak et al. 2015; Ahmed and Eun 2017). As

Table 2 Volatile compounds of *wumei* before and after OD process expressed as mean value of two replicates of area/weight 10^{-6}; values in brackets was proportioned in percentage

Compounds	Before OD				After OD		
	Fresh	Control	HHP	Heating	Control	HHP	Heating
Esters							
Ethyl acetate	3.08 (8.92%)	3.95 (10.25%)	4.49 (1.11%)	2.31 (2.16%)	1.91 (10.02%)	2.64 (1.75%)	0.31 (1.40%)
Ethyl butyrate	2.18 (6.32%)	1.09 (2.83%)	14.53 (3.60%)	–[a]	0.12 (0.63%)	4.33 (2.87%)	–[a]
Butyl acetate	–[a]	–[a]	3.27 (0.81%)	–[a]	–[a]	1.74 (1.16%)	–[a]
3-Methyl-1-butyl acetate	–[a]	–[a]	24.99 (6.20%)	–[a]	–[a]	14.38 (9.55%)	–[a]
4-Penten-1-yl acetate	–[a]	–[a]	44.12 (10.95%)	–[a]	–[a]	24.88 (16.52%)	–[a]
Hexenyl butyrate	–[a]	–[a]	4.55 (1.13%)	–[a]	–[a]	0.27 (0.18%)	–[a]
Butyl butylate	0.22 (0.64%)	0.28 (0.73%)	2.86 (0.71%)	0.76 (0.71%)	0.24 (1.26%)	1.97 (1.31%)	0.2 (0.91%)
Hexyl acetate	5.02 (14.54%)	6.86 (17.81%)	40.06 (9.94%)	6.95 (6.51%)	1.48 (7.76%)	12.73 (8.45%)	2.29 (10.37%)
(*Z*)-4-Hexenyl butyrate	–[a]	–[a]	0.54 (0.13%)	–[a]	–[a]	–[a]	–[a]
4-Hexen-1-ol acetate	–[a]	–[a]	5.87 (1.46%)	–[a]	0.16 (0.84%)	1.72 (1.14%)	–[a]
Ethyl acetoacetate	0.18 (0.52%)	0.26 (0.67%)	0.52 (0.13%)	0.27 (0.25%)	–[a]	–[a]	–[a]
(*E*)-2-Hexen-1-ol acetate	–[a]	–[a]	0.32 (0.08%)	–[a]	–[a]	–[a]	–[a]
Heptyl acetate	–[a]	–[a]	2.59 (0.64%)	–[a]	–[a]	0.51 (0.34%)	–[a]
Butyl hexoate	–[a]	–[a]	0.22 (0.05%)	–[a]	–[a]	–[a]	–[a]
N-Benzyl-2-aminocinnamate	0.34 (0.98%)	0.22 (0.57%)	0.55 (0.14%)	–[a]	–[a]	–[a]	–[a]
Ethyl octoate	0.32 (0.93%)	0.14 (0.36%)	0.89 (0.22%)	–[a]	–[a]	0.16 (0.11%)	–[a]
(*Z*)-3-Hexenyl butyrate	0.14 (0.41%)	0.16 (0.42%)	0.37 (0.09%)	–[a]	–[a]	–[a]	–[a]
Octyl acetate	–[a]	–[a]	0.24 (0.06%)	–[a]	–[a]	–[a]	–[a]
Hexyl hexoate	–[a]	–[a]	1.14 (0.28%)	–[a]	–[a]	0.18 (0.12%)	–[a]
Butyl caprylate	–[a]	–[a]	0.2 (0.05%)	–[a]	–[a]	–[a]	–[a]
Ethyl (4*E*)-4-decenoate	0.09 (0.26%)	0.16 (0.42%)	0.79 (0.20%)	0.18 (0.17%)	0.15 (0.79%)	0.26 (0.17%)	0.04 (0.18%)
(*Z*)-3-Hexenyl hexoate	0.29 (0.84%)	0.26 (0.67%)	0.74 (0.18%)	–[a]	0.01 (0.05%)	0.04 (0.03%)	–[a]
Vinyl hexoate	–[a]	–[a]	0.25 (0.06%)	–[a]	–[a]	–[a]	–[a]
Limonen-6-ol, pivalate	0.37 (1.07%)	0.41 (1.06%)	3.7 (0.92%)	0.24 (0.22%)	0.06 (0.31%)	0.96 (0.64%)	–[a]
Isolongifololyl acetate	0.48 (1.39%)	0.51 (1.32%)	0.54 (0.13%)	0.19 (0.18%)	0.12 (0.63%)	0.38 (0.25%)	–[a]
Butyl benzoate	–[a]	–[a]	0.43 (0.11%)	–[a]	–[a]	0.1 (0.07%)	–[a]
Diisobutyl succinate	–[a]	–[a]	0.27 (0.07%)	–[a]	–[a]	0.05 (0.03%)	–[a]
Dibutyl glutarate	–[a]	–[a]	0.53 (0.13%)	–[a]	–[a]	0.09 (0.06%)	–[a]
Subtotal	12.71 (36.82%)	14.30 (37.12%)	159.57 (39.59%)	10.90 (10.20%)	4.25 (22.28%)	67.39 (44.74%)	2.84 (12.86%)
Aldehydes							
Hexanal	1.03 (2.98%)	0.61 (1.58%)	18.09 (4.49%)	0.92 (0.86%)	–[a]	2.58 (1.71%)	–[a]
(*Z*)-3-Hexenal	1.32 (3.82%)	1.9 (4.93%)	8.88 (2.20%)	0.56 (0.52%)	0.57 (2.99%)	2.62 (1.74%)	–[a]
(*E*)-2-Hexenal	–[a]	–[a]	42.99 (10.67%)	3.45 (3.23%)	2.06 (10.80%)	12.29 (8.16%)	0.84 (3.80%)
Octanal	0.34 (0.98%)	0.47 (1.22%)	11.9 (2.95%)	0.69 (0.65%)	0.88 (4.61%)	0.24 (0.16%)	–[a]
(*Z*)-2-Heptenal	–[a]	–[a]	3.64 (0.90%)	1.09 (1.02%)	0.13 (0.68%)	0.45 (0.30%)	0.09 (0.41%)
Benzaldehyde	–[a]	–[a]	1.14 (0.28%)	–[a]	1.32 (6.92%)	0.13 (0.09%)	–[a]
Nonanal aldehyde	2.48 (7.18%)	3.56 (9.24%)	20.48 (5.08%)	5.73 (5.36%)	1.91 (10.02%)	10.44 (6.93%)	1.71 (7.74%)
(*E*,*E*)-2,4-Heptadienal	–[a]	–[a]	0.48 (0.12%)	–[a]	–[a]	–[a]	–[a]
(*E*)-2-Octenal	0.36 (1.04%)	0.45 (1.17%)	1.64 (0.41%)	0.25 (0.23%)	0.01 (0.05%)	0.68 (0.45%)	0.03 (0.14%)
Decanal	0.22 (0.64%)	0.28 (0.73%)	3.97 (0.98%)	0.22 (0.21%)	0.26 (1.36%)	2.53 (1.68%)	–[a]
Benzeneacetaldehyde	0.29 (0.84%)	0.18 (0.47%)	0.86 (0.21%)	0.1 (0.09%)	0.14 (0.73%)	0.59 (0.39%)	0.04 (0.18%)
(*Z*)-2-Nonenal	0.43 (1.25%)	0.32 (0.83%)	16.41 (4.07%)	–[a]	–[a]	6.12 (4.06%)	–[a]

Table 2 (continued)

Compounds	Before OD				After OD		
	Fresh	Control	HHP	Heating	Control	HHP	Heating
Thujopsene	0.54 (1.56%)	0.53 (1.38%)	0.17 (0.04%)	–[a]	–[a]	–[a]	–[a]
2-Undecenal	–[a]	–[a]	0.47 (0.12%)	–[a]	–[a]	–[a]	–[a]
Subtotal	7.01 (20.31%)	8.30 (21.55%)	131.12 (32.53%)	13.01 (12.18%)	7.28 (38.18%)	38.67 (25.67%)	2.71 (12.27%)
Terpenoids							
D-Limonene	–[a]	–[a]	31.52 (7.82%)	0.26 (0.24%)	–[a]	15.8 (10.49%)	–[a]
p-Cymene	–[a]	–[a]	2.88 (0.71%)	0.31 (0.29%)	–[a]	1.29 (0.86%)	0.14 (0.63%)
Linalool	7.58 (21.96%)	8.63 (22.40%)	1.16 (0.29%)	0.32 (0.30%)	1.27 (6.66%)	0.33 (0.22%)	–[a]
Hotrienol	–[a]	–[a]	2.62 (0.65%)	–[a]	–[a]	0.52 (0.35%)	–[a]
Cuminol	2.41 (6.98%)	3.03 (7.87%)	6.68 (1.66%)	3.78 (3.54%)	2.63 (13.79%)	1.17 (0.78%)	0.33 (1.49%)
(*Z*)-Geraniol	0.39 (1.13%)	0.32 (0.83%)	0.42 (0.10%)	0.08 (0.07%)	0.13 (0.68%)	0.12 (0.08%)	–[a]
Limonene	–[a]	–[a]	0.09 (0.02%)	–[a]	–[a]	–[a]	–[a]
α-Damascenone	–[a]	–[a]	2.08 (0.52%)	1.68 (1.57%)	–[a]	0.08 (0.05%)	0.05 (0.23%)
Dihydro-α-ionone	–[a]	–[a]	0.1 (0.02%)	–[a]	–[a]	–[a]	–[a]
Subtotal	10.38 (30.07%)	11.98 (31.10%)	47.55 (11.80%)	6.43 (6.02%)	4.03 (21.13%)	19.31 (12.82%)	0.52 (2.35%)
Alcohols							
1-Hexanol	0.23 (0.67%)	0.12 (0.31%)	39.03 (9.68%)	75.03 (70.23%)	3.16 (16.57%)	19.9 (13.21%)	15.88 (71.89%)
2-Ethyl-1-hexanol	1.22 (3.53%)	1.08 (2.80%)	12.46 (3.09%)	0.63 (0.59%)	0.3 (1.57%)	2.54 (1.69%)	0.09 (0.41%)
1-Nonanol	0.67 (1.94%)	0.52 (1.35%)	0.44 (0.11%)	–[a]	–[a]	–[a]	–[a]
Ketones							
3-Octanone	–[a]	–[a]	1.68 (0.42%)	0.22 (0.21%)	–[a]	–[a]	–[a]
1-Hepten-3-one	–[a]	–[a]	1.37 (0.34%)	–[a]	–[a]	0.04 (0.03%)	–[a]
6-Methyl-5-hepten-2-one	–[a]	–[a]	6.24 (1.55%)	–[a]	–[a]	2.65 (1.76%)	–[a]
2,6,6-Trimethylcyclohexanone	–[a]	–[a]	0.69 (0.17%)	–[a]	–[a]	–[a]	–[a]
6,7-Dodecanedione	1.44 (4.17%)	1.25 (3.25%)	0.33 (0.08%)	0.4 (0.37%)	–[a]	–[a]	–[a]
Dihydro-β-ionone	–[a]	–[a]	0.11 (0.03%)	–[a]	–[a]	–[a]	–[a]
Nerylacetone	0.55 (1.59%)	0.65 (1.69%)	1.94 (0.48%)	0.22 (0.21%)	0.05 (0.26%)	0.14 (0.09%)	0.05 (0.23%)
(*E*)-β-Ionone	0.12 (0.35%)	0.2 (0.52%)	0.14 (0.03%)	–[a]	–[a]	–[a]	–[a]
Subtotal	4.23 (12.25%)	3.82 (9.92%)	64.43 (15.99%)	76.5 (71.60%)	3.51 (18.41%)	25.27 (16.78%)	16.02 (72.52%)
Alcanes							
Tricosane	–[a]	–[a]	0.18 (0.04%)	–[a]	–[a]	–[a]	–[a]
Subtotal	0	0	0.18 (0.04%)	0	0	0	0
Lactones							
5-Decanolide	0.19 (0.55%)	0.12 (0.31%)	0.22 (0.05%)		–[a]	–[a]	–[a]
Subtotal	0.19 (0.55%)	0.12 (0.31%)	0.22 (0.05%)	0	0	0	0
Total	34.52 (100%)	38.52 (100%)	403.07 (100%)	106.84 (100%)	19.07 (100%)	150.64 (100%)	22.09 (100%)

[a] Not detected

reported in literature, phenolic compounds play an important role in the prevention of chronic diseases (Aguilera et al. 2016) and flavonoids have anti-osteoporotic effects, antitumor effects, anti-inflammatory effects, etc. (Nijveldt et al. 2001). Since one of the most important reasons of the popularity of candied *wumei* is the healthy benefits, these components were therefore evaluated.

However, total phenols and flavonoids content were found to be easily affected by heating treatments (Yeom et al. 2000; Keenan et al. 2010; Turturică et al. 2016; Santhirasegaram

et al. 2015; Wang et al. 2017) that caused their chemical degradation. On the other side, according to other studies, total phenols and flavonoids have been found to have a high resistance to thermal treatment (Cabrera and Moon 2015; He et al. 2016; Pacheco-Palencia et al. 2009). In the present work, heating resulted in a significant reduction of total phenol and flavonoid content, exhibited a negative influence on the functional properties of the products.

It is generally believed that ascorbic acid is a typical temperature-sensitive nutrient (Yeom et al. 2000). But we observed only 17.44% of degradation of the ascorbic acid after the heating pre-treatment. As *wumei* was handled in a form of whole fruit, even though perforated by needles, the peels probably still acted as a protective layer that partially protected it from thermal degradation.

The application of HPP promoted an increase of ascorbic acid, phenolic content, and flavonoid content immediately after the treatment. This indicated that the used extraction method did not allow a full recovery of the components from the matrix. The increased extractability was probably caused by the rupture of cellular structure as previously reported (Patras et al. 2009; Keenan et al. 2010; Corrales et al. 2008), which may lead to an increased bioavailability of the components.

During OD (after pre-treatments), a significant reduction in ascorbic acid, phenolic content, and flavonoid content was observed in all samples. The migration of these bioactive compounds with water to the osmosis solutions was probably the main reason (Turturică et al. 2016; Polydera et al. 2003). Another possible reason could be the chemical degradation by enzymes and/or oxygens. Ascorbic acid is easily oxidized to dehydroascorbic acid, which is further oxidized to 2,3-diketogulanic acid, a compound with no biological activity (Panyoyai et al. 2016). The oxidation of phenols is mainly associated with polyphenol oxidase (PPO) and peroxidase (POX) (Wills et al. 2015) and represents the main cause for the color change of the fruit. The degradation of flavonoids is not directly associated with the PPO activity, since they are not direct substrates of the oxidases (An et al. 2018). The hydroxyl and ketone groups and unsaturated double bonds are responsible for the stability of flavonoids (Qiao et al. 2014). In samples subjected to heating pre-treatment (*H* samples), bioactive compound content showed a significant decrease immediately after the treatment and during the first day of OD process while values remained stable thereafter, possibly due to the inactivation of degradative enzymes like ascorbate oxidase, PPO, POX, etc.

On the contrary, in samples subjected to HPP pre-treatment, values of ascorbic acid, phenols, and flavonoids were similar to the untreated products and always significantly higher compared with the heat-treated samples. According to previous works, the application of pressures of 50 MPa for 1 min is not sufficient for the inactivation of PPO, POX, and many other enzymes (Hendrickx et al. 1998; Yi et al. 2015; Crelier et al. 1999), leading to the similar performance as the untreated samples during OD.

Similarly, the in vitro antioxidant activity of the fruit extract showed similar trends being highly correlated with the total phenolic and flavonoid content (Díaz-Mula et al. 2011).

The volatile profile evaluated by SPME-MS method was highly affected by the different pre-treatment and by the osmotic process. Both HPP and heat-treated sample showed a higher total amount of volatiles compared with the untreated sample, probably because of the release of different compounds due to the disruption of cell structure. Nevertheless, a remarkably higher number of components were identified in HPP samples compared with heating (64 and 27, respectively). Many volatile components are highly sensitive to heat that could have caused their degradation (Boff et al. 2003; Chen et al. 2015; Wang et al. 2017). Also, a higher leakage of components in the solution may have been caused by the heating pre-treatment.

HHP pre-treated samples showed a considerable higher relative amount of ester and aldehyde content. Esters are considered to be the most important aroma constituents of fruits, contributing to fruity and floral notes (Nunes et al. 2008; Wang et al. 2009), indicating the positive application prospect of HHP on this kind of fruit.

OD has been shown to promote the increase of some volatile compounds, due to the shrinking of the plant cell (Chandra and Kumari 2015) and the concentration of volatile molecules. The cell shrinkage can also increase some enzyme activities leading to the synthesis on new components (Zhao et al. 2016; Chiralt and Talens 2005).

In the present study, the osmotic process generally exhibited a negative influence on the volatile profile of all samples, probably because of the long processing time (5 days) that caused a high release of the compounds in the atmosphere or into the osmotic solution (Yadav and Singh 2014; Rahman et al. 2018). However, HPP pre-treated fruits still showed a richer volatile profile compared with both control and *H* samples.

Conclusions

This study evaluated the use of HPP as a pre-treatment for accelerating osmotic dehydration of *wumei* fruit compared with the traditional heating treatment.

Results showed that although the mass transfer parameters were not significantly improved compared with the use of heat, the application of HPP allowed to increase noticeably the antioxidant compound content (ascorbic acid, total phenols, and total flavonoids) and the antioxidant activity and to improve the aromatic profile (in terms of total amount and number of volatile components) of the final product.

HPP resulted in a promising method for the production of candied *wumei* fruit with high nutritional and sensorial properties. Further studies should focus on other quality characteristics such as color, texture, and on the sensorial perception of the product by consumers.

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Paper IV: Important factors to consider for acrylamide mitigation in potato crisps using pulsed electric fields, *manuscript submitted to Innovative Food Science and Emerging Technologies*

Important factors to consider for acrylamide mitigation in potato crisps using pulsed electric fields

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Abstract

The aim of this study was to evaluate the application of pulsed electric field (PEF) as a pre-treatment before frying for the mitigation of acrylamide content, considering various factors such as the shape of treated potato samples and the washing time after PEF. Measuring the degree of cell disintegration index (p_o) and the changes in water electrical conductivity during washing of potato slices, PEF protocol and sample preparation scheme were optimized. Peeled potato slices (thickness 1.5 ± 0.2 mm) were subjected to PEF pre-treatment (1.5 kV cm^{-1}, pulse duration 10 µs, total treatment time 10 s, pulse frequency 100 Hz) and to blanching (85°C for 3.5 min) and then to washing in water for 5, 10, 15 and 20 min. After frying (175°C, 3 min), product quality, in terms of colour, texture and acrylamide content were evaluated. Results showed that PEF allowed a reduction of acrylamide content in potato crisps of around 30%, significantly higher if compared to the reduction obtained with blanching, with only slight modifications of the final quality of the product, in terms of colour and texture.

Industrial relevance: Recently, the Commission Regulation (EU) 2017/2158 of 20 November 2017 has established new "mitigation measures and benchmark levels for the reduction of the presence of acrylamide in foods", which aim to ensure that food businesses put in place steps to mitigate acrylamide formation. The traditional blanching treatment present several practical drawbacks and lead to undesirable changes of the product quality.

Results of this study confirmed the high potentiality of the application of PEF as a pre-treatment to reduce the acrylamide content in deep-fat fried potato crisps. Moreover, important indications regarding the possibility of industrial application of PEF pre-treatment for the production of potato crisps were given.

Keywords:
Potato crisps; Acrylamide; electroporation; mass transfer; colour, texture

1. Introduction

Acrylamide has been identified as a contaminant in a range of fried and oven-cooked foods (e.g. French fries, potato crisps, bread and cereal) and drinks (e.g. coffee); and its classification as probably carcinogenic in humans has caused worldwide concerns (International Agency for Research on Cancer, 2014). Although most epidemiologic studies examining the relationship between estimated dietary consumption of acrylamide and specific cancer resulted inconclusive, experimental animal studies identified neurotoxicity, carcinogenicity, adverse effects on male reproduction, as possible critical endpoints for acrylamide toxicity (European Food Safety Authority, 2015).

Certain foods, more specifically certain food components such as asparagine and reducing sugars, could lead to the formation of acrylamide during heat treatment at temperatures above 120 °C as a result of the Maillard reaction (Mottram, Wedzicha, & Dodson, 2002). Among fried carbohydrate-rich foods, potato crisps contribute to a substantial proportion of the estimated intake of acrylamide in the European adult population (European Food Safety Authority, 2015). Statistical data revealed that in Europe the consumption of salty snacks, in particular potato chips/crisps, stands to an average of 1.5 kg per capita in 2018 (Statista, 2018).

National authorities, together with research institutes and food industries reported many strategies of controlling and minimizing the formation of acrylamide, with particular concern to fried potato-based products, due to the presence of large concentration of acrylamide precursors in the potato. In fact, levels up to 4000 ppb of this contaminant have been detected in potato crisps (Becalski, Lau, Lewis, & Seaman, 2003).

Recently, the Commission Regulation (EU) 2017/2158 of 20 November 2017 has established new "mitigation measures and benchmark levels for the reduction of the presence of acrylamide in foods", which aim to ensure that food businesses put in place steps to mitigate acrylamide formation (European Union, 2017).

Powers, Mottram, Curtis, & Halford (2017) analysed European manufacturers' data on acrylamide in potato crisps from 2002 to 2016, and the study showed that, even though acrylamide levels in potato crisps in Europe have been levelled off in recent years, more than 5% of samples exceeded the regulated benchmark level for potato crisps (750 ng g^{-1}).

Several investigations have proposed mitigation ways to be applied at different stages of the manufacturing process of potato crisps, in order to reduce the concentration of acrylamide precursors. Among conventional mitigation strategies, hot water blanching of potato slices appeared to facilitate the extraction of Maillard reaction substrates, in addition to enzyme inactivation, also improving the colour uniformity and texture, and oil absorption reduction (Pedreschi, Kaack, & Granby, 2004; Mestdagh et al.,

2008). However, this thermal pre-treatment presents the drawbacks of being time-intensive, high energy-consuming and of promoting considerable modifications of sensorial properties of final product.

Recent studies have proposed alternative non-thermal technologies in food processing and preservation. Pulsed electric fields (PEF) have been widely described as one of the most promising non-thermal novel technologies in the last decades, stimulating intensive research in several fields such as biotechnology, medicine and food processing. PEF treatment is based on the application of an external electric field applying short and intensive electric pulses. The application of PEF for potato snacks pre-treatment is under investigation, and many researchers have already reported high numbers of benefits that could be achieved by applying electric pulses to raw potatoes. Fauster et al. (2018) have recently described the impact of PEF treatment on potato structure and various potential advantages on quality and economic aspects of industrial French fries production, including reductions of cutting force, starch loss and oil uptake. Furthermore, the application of pulsed electric field treatments above a specific critical value of field strength is well known to enhance mass transfer from plant tissues, increasing the cell membrane permeabilization (Donsì, Ferrari, & Pataro, 2010). Following this principle, Jaeger, Janositz, & Knorr (2010) have stated that PEF treatment of raw potatoes could assist and increase the release of sugars and amino acids that represent the main substrates for the Maillard reaction, consequently leading to formation of lower amounts of acrylamide. Moreover, Janositz, Noack, & Knorr (2011) have reported a significant increase in the release of reducing sugars (fructose, glucose) and sucrose in potato slices after PEF application.

However, the effective reduction of acrylamide content in deep-fat fried potato products is still unclear, although many researchers observed a significant increase of acrylamide precursors extractability on PEF treated potatoes, reducing browning during frying (Ignat, Manzocco, Brunton, Nicoli, & Lyng, 2015). Besides, lab-scale investigations followed different trials' schemes, applying PEF as a pre-treatment of whole potato tubers before or after peeling, concentrating on the treatment itself, with low attention to the combination of other process operational units that could influence the outcome. It is necessary to understand how the rate of mass transfer promoted by applying certain electric field strengths could be influenced by other processes operations, and how the quality of the final product will be preserved.

This study aimed to evaluate the effect of the application of PEF as a pre-treatment of whole tuber or potato slices, and the effect of time modulation of subsequent washing steps of treated potato slices, on the final acrylamide content and quality of fried potato crisps. Measuring the degree of cell disintegration index (p_o) (Angersbach, Heinz, & Knorr, 1999) and the changes in water electrical conductivity during washing of potato slices, it is possible to optimise either PEF protocol and sample preparation scheme, in order to maximise the release of acrylamide precursors from the raw tissue. The efficiency of PEF as a non-thermal pre-treatment on the acrylamide reduction was compared to a conventional blanching in hot water usually used as a pre-treatment in the fried potato industrial lines.

2. Materials and Methods

2.1 Sampling

Potato tubers (*Solanum tuberosum L.*) of the Lady Claire variety (suitable for industrial processing), were purchased at a local market and stored in the dark at 10 ± 2 °C for a maximum of two weeks before trials. The initial moisture content of potato tubers was 81.92 ± 0.58 %, evaluated by drying 5g of fresh potato tissue in a convection oven at 105 °C until a constant weight was achieved. Before pre-treatment, only tubers of a similar size and shape were selected, manually peeled and sliced (1.5 ± 0.2 mm in thickness) using a stainless-steel electric slicer machine (Mod. KAFPL0922N CAD, Italy). Both tubers and slices were rinsed for 1 min in tap water (water temperature: 18 ± 2 °C) prior to pre-treatments.

2.2 Pre-treatments

2.2.1 Blanching

Blanching was performed by immersing potato slices in hot distilled water at 85 °C and for 3.5 min stirring with a product-to-water ratio of around 1:2 (w/w), according to the method used by Pedreschi, Mariotti, Granby, & Risum (2011).

2.2.2 Pulsed electric fields (PEF) treatment

PEF pre-treatments were performed using a lab-scale PEF unit delivering a maximum output current and voltage of 60 A and 8 kV, respectively (Mod. S-P7500, Alintel, Italy). The generator provides monopolar rectangular-shape pulses and adjustable pulse duration (5-20 μs), pulse frequency (50-500 Hz) and total treatment time (1-600 s). The treatment chamber (50 mm length x 50 mm width x 50 mm height) consisted in two parallel stainless-steel electrodes (3 mm thick) with a 47 mm fixed gap. Output voltage and current were monitored using a PC-oscilloscope (Picoscope 2204a, Pico Technology, UK). Samples were treated at room temperature in tap water, with an initial electrical conductivity of 542 ± 2 μS cm^{-1} at 25 °C, measured using an EC-meter (Mod. Basic 30, Crison, Spain). Temperature changes due to PEF treatments were negligible. An electric field strength of 1.5 kV cm^{-1} was selected in order to achieve irreversible electroporation. Faridnia, Burritt, Bremer, & Oey (2015) reported that E > 1 kV cm^{-1} promoted high amount of ion leakage from tissues due to the irreversible electropermeabilization of cell membranes. The applied PEF treatment parameters are listed in Table 1. The same PEF protocol was applied on both whole tubers and potato slices, in order to understand if the different exposed surface-to-volume ratio could have affected the efficiency of mass transfer. In both cases, the treatment chamber was filled with a product–to–water ratio of around 1:5 (w/w).

2.3 Determination of cell disintegration index (p_o)

Cell disintegration index (p_o) was analysed, according to Angersbach, Heinz, & Knorr (1999), for PEF treated whole tubers and slices. The method is based on changes in the electrical properties of an intact and

permeabilized biologic membrane (considered equal to a resistor and a capacitor). The electrical conductivity (σ) for intact and processed samples was obtained by impedance measurements at low (1 kHz) and high (5 MHz) frequencies. The impedance spectra were acquired using a precision impedance meter (Mod. LCR-8105G, GW Instek, Taiwan) connected to a parallel plate measuring cell with adjustable gap. The cell disintagration index (p_o) was calculated by the following equation:

$$p_o = \frac{\left(\sigma_h^i/\sigma_h^s\right)\sigma_l^s - \sigma_l^i}{\sigma_h^i - \sigma_l^i} \quad (1)$$

where σ_l^i and σ_l^s indicate the electrical conductivity of untreated and treated cell material at low frequency (1 kHz), respectively; and σ_h^i and σ_h^s indicate the electrical conductivity of untreated and treated material at high frequency (5 MHz), respectively. The parameter p_o ranges from 0 for intact tissue to 1 for complete disrupted tissue.

2.3 Washing in water

PEF-processed potato slices were soaked and stirred in tap water (18 ± 2 °C), with an initial electrical conductivity of 319 ± 4 μS cm^{-1} at 25 °C, and with a product-to-water ratio around 1:1.5 (w/w). In order to select the optimal soaking time as a result of maximum release of intracellular compounds into the external aqueous phase due to PEF-induced electroporation, washing times of 5, 10, 15, 20 min were tested before frying. For each dipping time, changes in water electrical conductivity were registered using an EC-meter (Mod. Basic 30, Crison, Spain). The selected washing times, based on the highest water electrical conductivity variation recorded, were applied also to untreated (control) and blanched potato slices before frying, in order to obtain comparable results for further analysis.

2.4 Frying conditions

Untreated (control), blanched and PEF-treated potato slices were deep-fried in high-oleic sunflower oil using an electrical fryer (Mod. MFR280R, Fama Industrie, Italy) at 175 °C as initial oil temperature. Potato-to-oil weight ratio was around 1:10 and slices were fried for 3 min until a final moisture of ~2% (wet basis) was reached. Temperatures of frying oil and frying potatoes were monitored using thermocouples sensors type K connected to a data logging system (Mod. 9211A, National Instruments™, Texas).

2.5 Analysis of fried potato crisps

2.5.1 Computer Vision System (CVS) for colour determination

The surface colour of potato crisps was measured using a Computer Vision System (CVS) consisting of an illumination source, a colour digital camera (CDC), and an image processing software. Potato crisps

samples were placed inside a dark box to exclude external light, and RGB images were acquired by a CDC (Mod. D7000, Nikon, Japan) with a 105 mm lens (Mod. AF-S Micro Nikkor), located vertically over the sample at a distance of 35 cm and connected to a PC. The lighting system consisted of four daylight fluorescent lamps (60 cm in length) connected to an electronic ballast to ensure uniform illumination, with a colour temperature of 6500 K and sited at an angle of 45° with the CDC. For each sample, untreated, blanched and PEF treated, 12 images were captured, each of one side of potato crisps. The pre-processing of RGB images, segmentation and colour quantification were performed with ImageJ analysis software (NIH, USA). The average value of the segmented pixels in the CIE L^* a^* b^* colour space was registered as the colour of the sample. From numerical values of a^* (green/red) and b^* (yellow/blue) chromatic parameters, hue angle (H°) was calculated by the following equation and used to describe colour variations between samples:

$$H° = tan^{-1}(b^*/a^*) \quad (2)$$

2.5.2 Texture

Texture analysis of crisps were performed at room temperature (~20 °C) using a Texture Analyser TA-XT2 (Mod. HDi 500, Stable Micro System, Surrey, UK) equipped with a 5 kg load cell. A puncture test was selected to evaluate samples firmness and crispness. Crisp samples selected on the basis of uniform size and shape, were placed on a support rig (HDP/CFS, Crisp Fracture Support Rig and corresponding Heavy Duty Platform) and compressed for 3 mm distance using a spherical probe (P/0.25S) of ¼ - inch diameter (Salvador, Varela, Sanz, & Fiszman, 2009). Force *vs* distance curves were obtained using a test speed of 1.0 mm s^{-1} and the results obtained from 12 slices for each sample were expressed as firmness, calculated by means of maximum force values and as crispness, calculated from means of linear distance (the length of a line joining all fracture points in the force-deformation curve) between the first and the last facture peaks registered.

2.5.3 Acrylamide

Sample extraction and SPE purification. Potato chips samples were finely ground before the extraction; 1 g of sample was weighted into a polypropylene conical tube, and 100 µL 10µg/mL internal standard solution ($^{13}C_3$-labelled acrylamide in MeOH) followed by 10 mL 0.1% (v/v) formic acid were added. After mixing with Vortex for 10 min, the extract was centrifuged at 4500 rpm for 15 min. A 2 mL portion of clarified solution was removed, avoiding collection of top oil layer when present, and filtered through paper filter. A solid-phase extraction (SPE) was performed using C18 cartridges (1000mg, 6mL; Phenomenex). Cartridges were first conditioned with 5 mL methanol followed by 5 mL water; 1 mL of filtered sample was loaded and washed with 1 mL water. Eluition was performed with 1 mL acetone. Acetone was removed

under nitrogen flow and sample was dissolved in 1 mL 0.1% (v/v) formic acid before injection. SPE clean-up was performed on 3 extracts for each sample.

HPLC-ESI-MS/MS analysis. LC-ESI-MS/MS in positive ion mode (ESI^+) analyses were performed by an Agilent 6420 triple quadrupole (Agilent, Santa Clara, United States) coupled to an Agilent 1290 Infinity LC Pump equipped with an autosampler and a thermostated column oven, according to Calbiani, Careri, Elviri, Mangia, & Zagnoni (2004) with some modifications. The analytical column was a Poroshell 120 C18, 3.0 x 100 mm, 2.7 µm (Agilent, USA) maintained at 20 °C. The elution was in isocratic mode using a mixture of 0.1% (v/v) aqueous formic acid and methanol (99.5/0.5, v/v) as mobile phase at a flow rate of 0.3 mL/min. The sample injection volume was 10 µL. Full-scan analyses were performed in the 40-100 Da mass range, acquiring the following transitions: extracted ion at m/z 55, due to the transition 72 > 55, and at m/z 58, due to the transition 75 > 58 were used for the quantitative analysis. A calibration curve was made for the quantification diluting stock solution of acrylamide with water in the 1.5-200 µg/L range. For acquisition and processing data, the Agilent MassHunter Workstation software was used.

2.6 Statistical analysis

Significant differences between results were calculated by paired samples Student's t-test, parametric analysis of variance (ANOVA) and Tukey multiple comparison, with a significance level of 95% ($p < 0.05$). If Shapiro-Wilk test for normality and Levene's test for homoscedasticity of data resulted statistically significant ($p < 0.05$), non-parametric multiple range test Kruskal-Wallis and Holm stepwise adjustment were used, with a significant level of 95% ($p < 0.05$) (R Foundation for Statistical Computing, Austria). All treatments were conducted in triplicate and results were expressed as mean ± standard deviations of replications.

3. Results and discussion

3.1 Experimental design set up

3.1.1 Effect of PEF treatment on cell disintegration index and degree of mass transfer

As reported by Angersbach et al. (1999), impedance measurements of plant tissues allow the evaluation of cell membrane permeabilization after applying PEF treatments. It is well established that measurements of the changes in electrophysical properties of untreated and treated cell tissues represent a reliable method to correlate PEF processing protocol and the cell damage degree in biological systems. Moreover, it has been widely reported that PEF-induced membrane permeabilization has the potential to effectively enhance mass transfer from vegetable tissues, increasing the diffusion of cell compounds/metabolites (Donsì et al., 2010).

To understand the efficiency of selected PEF protocol (Table 1) on the degree of cell disruption, and so on the level of mass transfer, cell disintegration index of PEF-treated potato tubers and potato slices was calculated from Eq. 1. Results are shown in Fig. 1; as expected, higher surface dimension exposed to electric

pulses, being related to slices, resulted in higher cell disintegration, explaining the better efficiency of the pre-treatment if applied after the slicing step of the experimental scheme. Following these results, the setup of the experimental plan for acrylamide reduction in fried potato crisps was designed applying the different pre-treatments on raw potatoes directly after slicing.

Another useful method to assess the intactness of cell membranes, reported by many researchers, is the measurement of ions/small molecules leakage from intracellular compartments. Faridnia et al. (2015) reported that by suspending a PEF-treated potato tuber into an isotonic solution of mannitol (0.2 M) it was possible to monitor the electrolytes leakage from cell tissue by measuring changes in electrical conductivity of the surrounding media. Furthermore, it has been demonstrated that cell fluid leakage due to electroporation is function of time, as many transition processes induced by PEF (e.g. moisture and air redistribution among microscopic extracellular channels, mass transfer, partially or completely resealing of cell membranes) could last from seconds to hours (Oey, Faridnia, Leong, Burritt, & Liu, 2017). On the basis of this concept, it is clear that subsequent unit operations for potato crisps manufacturing need to be assessed in order to maximise mass transfers. Washing of potato slices is a common practice in potato crisps production which allows to remove any surface starch residual prior to frying. In this work, different washing times of PEF-treated potato slices were evaluated, measuring changes in electrical conductivity of residual washing water. Results are shown in Fig. 2; the maximum water conductivity variation was after 5 min of washing, highlighting the period of time subsequent to PEF-treatment that permitted the highest release of cell fluid into the aqueous media. Thanks to the aforementioned preliminary studies, in this work the experimental plan for the production of lab-scale deep-fat fried potato crisps was designed by selecting 5 min as the preferred time for potato slices washing step.

3.2 Deep-fat fried potato crisps analysis

3.2.1 Colour

Colour is one of the most important parameters to control during frying, being strongly related to consumer perception (Scanlon, Roller, Mazza, & Pritchard, 1994). Visual quality is associated with physical, chemical and sensorial evaluation and it is an important driver for buying being associated by consumers with flavour, safety, storage time, nutritional aspects and taste (Pedreschi, Kaack, & Granby, 2006).

On the other side, development of colour during frying is the result of the Maillard reaction, that involves reducing sugars and the amino acid asparagine and has been related to the formation of toxic compounds such as acrylamide. The extent of the Maillard reaction depends on the presence of reaction substrates and on frying parameters such as temperature and time (Romani, Bacchiocca, Rocculi, & Dalla Rosa, 2008; Romani, Rocculi, Mendoza, & Dalla Rosa, 2009).

Colour of potato crisps is often measured using a colorimeter in L*a*b* units. According to Pedreschi, León, Mery, & Moyano (2006), the use of a computer vision system (CVS) technique instead of the conventional colorimeter for monitoring the development of colour in potato crisps has different advantages,

such as the possibility to analyse the whole surface of the product and to identify the presence of brown spots and other defects.

Fig. 3 shows images of control (3A), blanched (3B) and PEF treated (3C) potato crisps after frying. After blanching, little changes are noticeable in the appearance of potato slices compared to the control. On the contrary, the slices pre-treated by PEF showed a more uniform and lighter surface colour. Images have been converted from RGB into L*a*b* channels, as shown by Fig. 4. The calculated values of L* and h° (Eq. 2) of the threes samples are reported in Fig. 5.

Development of colour during frying is generally indicated by a decrease in L* and/or an increase of the redness parameter (a*) and of the hue (H°). Variation of colour parameters observed in the present study allowed the discrimination among samples. The three samples did not show significant differences in terms of L*. On the other side, hue values increased for both pre-treated samples compared to the control, the highest values being related to the PEF pre-treated sample. Ignat et al. (2015) found similar results comparing the colour development during frying of blanched and PEF-treated potato slices. The possibility of the release of the Maillard substrates, in particular of reducing sugars, from potato tissue has been observed by various authors (Jaeger et al., 2010; Janositz et al., 2011). The increase of mass transfer upon PEF treatment is due to the effect of electroporation and cell permeabilization, that allow an increasingly diffusion of intracellular components across the membranes, as confirmed by the increase of conductivity of the washing water observed above.

3.2.2 Firmness & crispness

Texture is one of the main characteristics that influence the sensorial properties of potato-based products, and a delicate and crispy texture is recommended for potato crisps (Kita, 2014). Texture changes in potato crisps during frying result in the initial tissue softening and further crust development (Pedreschi, Moyano, Santis, & Pedreschi, 2007). These changes can be influenced by many factors, e.g. firmness depends on the degree of starch gelatinization, on changes in the cell walls structure, mainly related to an increase in their permeability and on the reduction of intercellular adhesion between neighbouring cells (Moyano, Troncoso, & Pedreschi, 2007). Crispness instead is influenced mainly by dry matter content and oil uptake during frying (Abong, Okoth, Imungi, & Kabira, 2011).

The changes of firmness and crispness of untreated, blanched and PEF treated potato crisps subjected to frying are shown in Fig. 6A and Fig. 6B, respectively. A slight, but significant reduction of both parameters was observed for blanched and PEF treated samples in comparison to the untreated one. In fact, blanching at high temperature (80-100 °C), contrary to that at low temperature, has been already reported to promote potato tissue softening, by starch modification (hydration, swelling and gelatinization) along with β eliminative cleavage and pectin solubilization (Botero-Uribe, Fitzgerald, Gilbert, & Midgley, 2017). Moreover, high temperature blanching decreases polyphenol oxidase activity, responsible for enzymatic browning (Bingol, Wang, Zhang, Pan, & McHugh, 2014).

Recently, some studies have been performed showing the effect of PEF pre-treatment on texture and other quality parameters of potato tissue before frying. Fauster et al. (2018) observed softening of the potato tissue and therefore the improvement of cutting behaviour (smoother surface and lower feathering). PEF treatment (0.3- 1.2 kV/cm) caused also a significant softening of the ground tissues of sweet potato, resulted into lower force necessary for cutting (Liu et al., 2017). The softening of the tissue upon PEF treatment is probably due to the cell structure modification, mainly the increase in the membrane permeability and the irreversible cell breakdown, that in turn increase the water transfer, which is very important during potato-based product frying (Botero-Uribe et al., 2017). However, there is a lack of information in the literature about the effect of PEF pre-treatment on final products texture. Ignat, Manzocco, Brunton, Nicoli, & Lyng (2015) observed no differences in texture of PEF treated potato cubes (0.75 kV/cm and 2.50 kV/cm) in comparison to blanched and untreated fried ones. In our work, PEF-treated samples presented lower firmness and crispness in comparison to the untreated one, while no significant differences were observed between PEF-treated and blanched samples. The discrepancy could be due to the different shape of potato samples, indeed Ignat et al. (2015) performed their study on potato cubes (2 × 2 × 2 cm), while the present work was focused on 1.5 mm potato slices, as well as to different PEF process parameters and frying temperature.

3.2.3 Acrylamide content

Table 2 shows the acrylamide content (ng g^{-1}) of untreated, blanched and PEF-treated potato crisps. For the potato crisps pre-treated by PEF, the acrylamide content appeared lower compared with those pre-treated by conventional blanching. The PEF pre-treatment protocol and experimental conditions applied in this study resulted on a reduction of around 30% of acrylamide content compared to control (untreated), while only around the 17% of reduction was observed on blanched samples compared to control (untreated). Similar results of acrylamide reduction due to hot water blanching of potato slices were previously reported by other authors, as both acrylamide precursors, reducing sugars and asparagine, are leached out during blanching (Pedreschi et al., 2011). The cell electroporation phenomenon induced by the application of the applied PEF protocol on raw potato slices resulted in a further improvement of acrylamide reduction in fried potato crisps compared to the applied conventional pre-treatment.

4. Conclusions

Overall this study confirmed the high potentiality of the application of pulsed electric fields as a pre-treatment to improve the release of acrylamide precursors in raw potatoes and so to reduce the acrylamide content in deep-fat fried potato crisps. Moreover, important indications regarding the possibility of industrial application of PEF pre-treatment for the production of potato crisps were given. By monitoring PEF treatment parameters and other manufacturing steps, it was possible to achieve a consistent reduction of acrylamide, with only slight modifications of the final quality of the product, in terms of colour and texture.

Although PEF pre-treatment led to a significant reduction of acrylamide content in potato crisps if compared to untreated and blanched samples, the final amounts found were still higher than recommended

legislative limits (750 ng g^{-1}); in this direction other possible combined strategies need to be developed for industrial applicability. The combination of PEF and a mild blanching of raw potatoes and the monitoring of subsequent manufacturing operational units could enhance the extraction of reducing sugars and free asparagine and consequently the reduction of the acrylamide formation in potato crisps.

Figure captions

Fig. 1.

Cell disintegration index (CDI) of PEF treated potato slices and tubers (E = 1.5 kV/cm; n = 1000). Results are expressed as means ± standard deviations (error bars) of n=20. Values with different letters differ significantly ($p < 0.05$).

Fig. 2.

Variations of water electrical conductivity affected by different washing times of PEF treated potato slices. Results are expressed as means ± standard deviations (error bars) of n=5. Values with different letters differ significantly ($p < 0.05$).

Fig. 3.

Examples of RGB images of untreated (a), blanched (b) and PEF-treated (c) potato crisps.

Fig. 4.

Examples of image conversion from RGB into L*a*b* channels. Pixels areas analysed are highlighted in yellow.

Fig. 5.

Lightness (A) and hue angle (B) of untreated, blanched and PEF-treated potato crisps. Results are expressed as means ± standard deviations (error bars) of n=20. Values with different letters differ significantly ($p < 0.05$).

Fig. 6.

Firmness (A) and crispness (B) of untreated, blanched and PEF-treated potato crisps. Results are expressed as means ± standard deviations (error bars) of n=20. Values with different letters differ significantly ($p < 0.05$).

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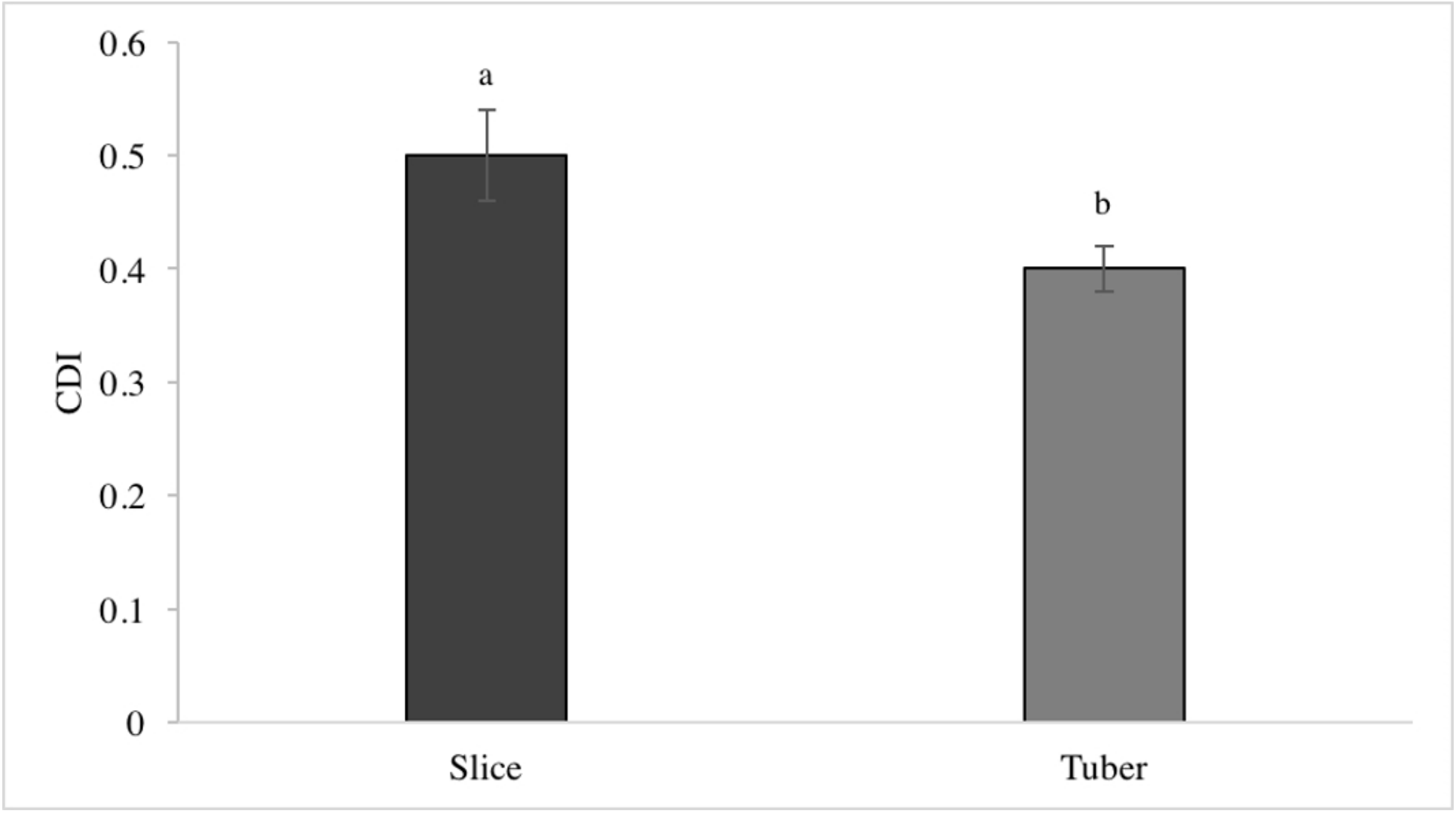
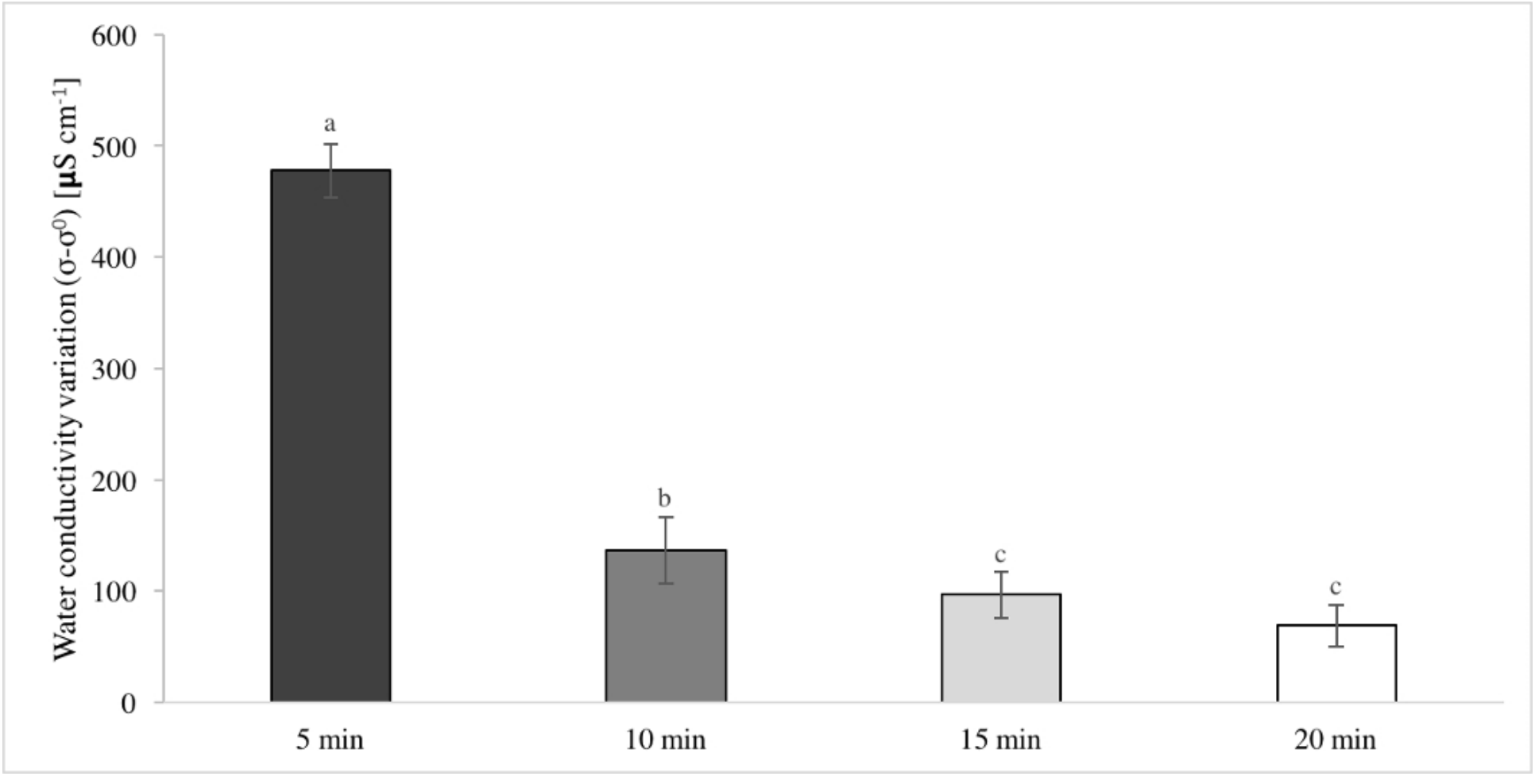

0.6
0.5
0.4
0.3
0.2
0.1
0
CDI
a
b
Slice
Tuber


Water conductivity variation (σ-σ⁰) [µS cm⁻¹]
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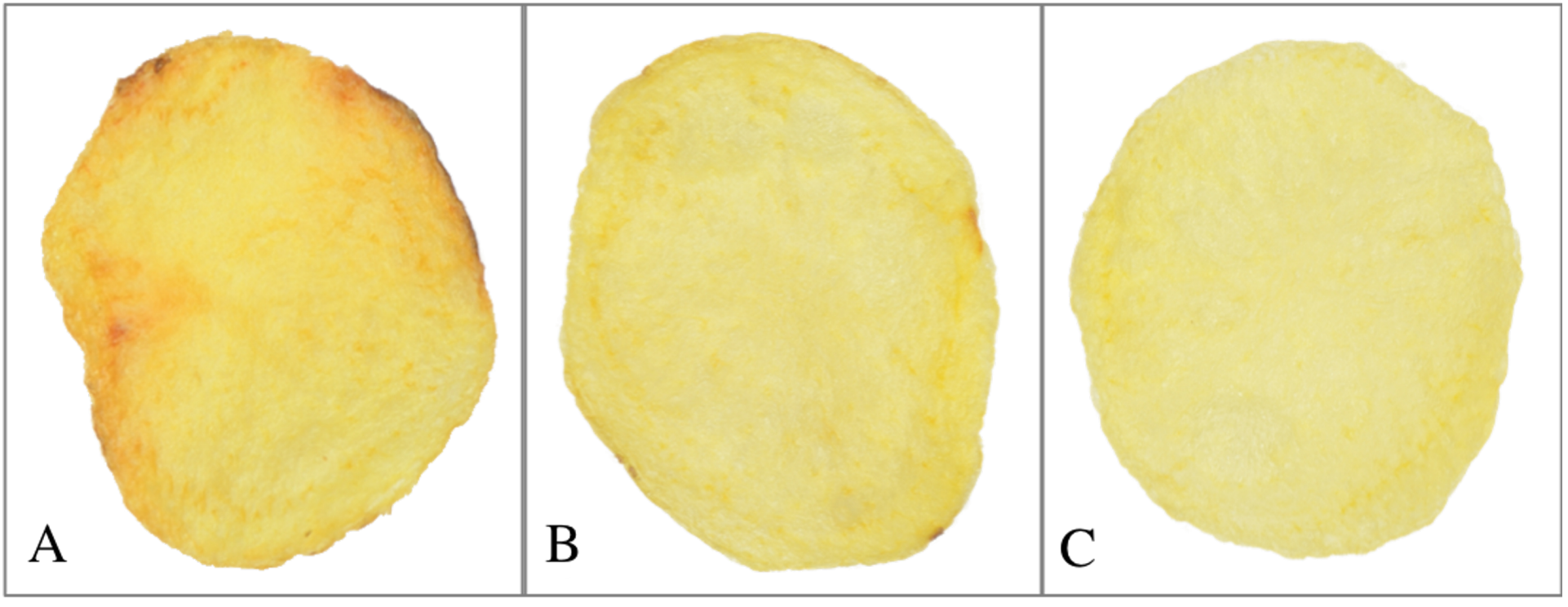
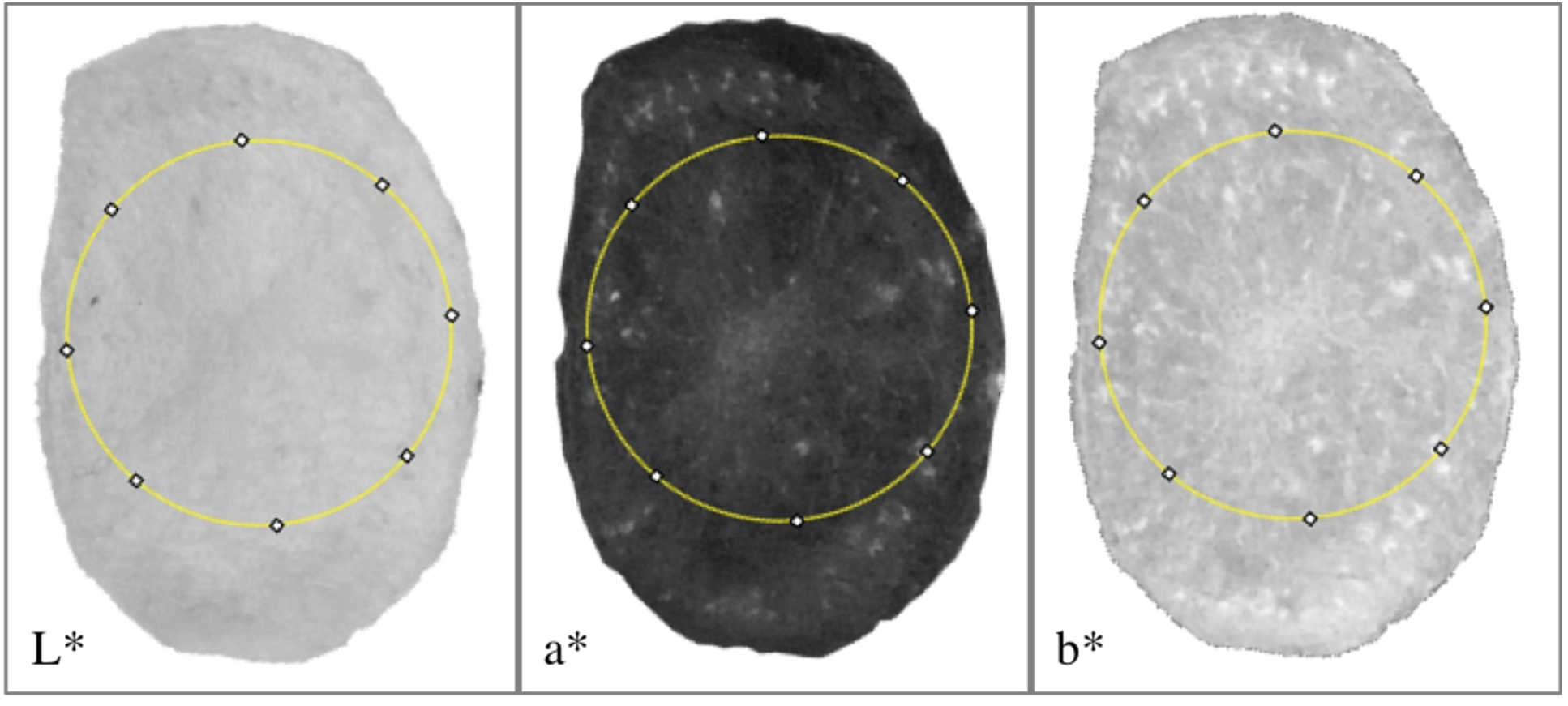
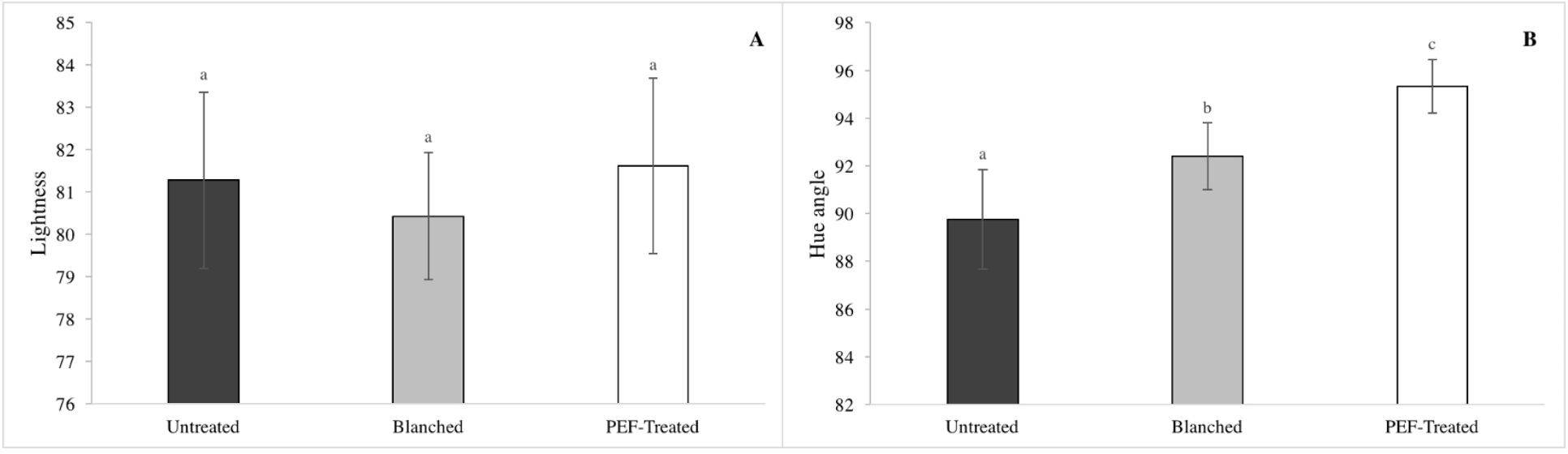
500
400
300
200
100
0
a
b
c
c
5 min
10 min
15 min
20 min


A
B
C


L*
a*
b*


A
Lightness
85
84
83
82
81
80
79
78
77
76
a
a
a
Untreated
Blanched
PEF-Treated
B
Hue angle
98
96
94
92
90
88
86
84
82
a
b
c
Untreated
Blanched
PEF-Treated

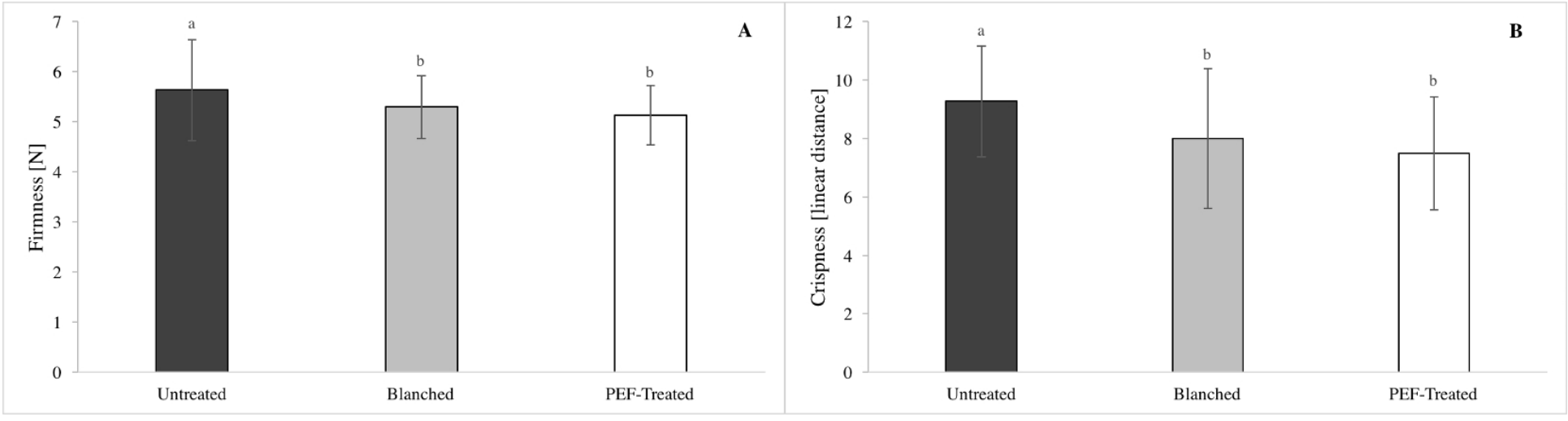

A
Firmness [N]
0
1
2
3
4
5
6
7
a
b
b
Untreated
Blanched
PEF-Treated
B
Crispness [linear distance]
0
2
4
6
8
10
12
a
b
b
Untreated
Blanched
PEF-Treated

Table 1

Parameters of pulsed electric fields (PEF) treatment

PEF parameter	
Field strength E [kV/cm]	1.5
Pulse number n	1000
electrode distance d [cm]	4.7
Pulse form	rectangular monopolar
Pulse duration ?? [µs]	10
Pulse frequency [Hz]	100
Total treatment time [s]	10

Table 2

Acrylamide content (ng g^{-1}) of untreated, blanched and PEF-treated potato crisps.

Sample	Acrylamide (ng g^{-1}) Means ± standard deviations
Untreated	1958.22 ± 64.77
Blanched	1617.02 ± 147.11
PEF-treated	1355.63 ± 101.5

Paper V: Essential rosemary oil enrichment of minimally processed potatoes by vacuum-impregnation, *manuscript submitted to International Journal of Food Science and Technology.*

Essential rosemary oil enrichment of minimally processed potatoes by vacuum-impregnation

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Essential rosemary oil enrichment of minimally processed potatoes by vacuum-impregnation

Abstract

Vacuum impregnation (VI) satisfies that requirement as it is an emerging technology that has been recognized as a promising tool for the introduction of solutes into the internal structure of some porous food products. The enrichment of minimally processed potatoes with aromatic compounds could represent an interesting method for product innovation. This study was aimed at applying VI with rosemary essential oil on a minimally processed potato product in order to obtain an innovative fresh-cut potato product, and to evaluate its influence on the physico-chemical, sensorial and microbiological properties of potato sticks during refrigerated storage and after frying. A pressure of 60 mbar was applied for 30 min followed by a relaxation time at atmospheric pressure of 30 min to potato stocks immersed in rosemary oil solutions in concentration between 0 and 12%. Obtained samples were packed and stored at 4°C for 14 days. Analytical determinations were carried out on the fresh and fried product. The weight gain of potato promoted by VI was in the range of 6-14 %, depending on the concentration of rosemary essential oil. The rosemary essential oil concentration gradients of impregnated potato sticks were detected by GC analysis and sensorial test, evidencing their persistency during storage and after frying. The treatment seemed to improve microbiological stability, not affecting the texture, moisture, but slightly deteriorating the product colour. Results obtained in the present study confirm the potentiality of vacuum impregnation for fresh products innovation.

Keywords

Minimally processed potatoes, vacuum impregnation, rosemary essential oil, refrigerated storage stability, frying

Introduction

Fresh cut fruit and vegetables are defined as any fresh fruit or vegetable or combination thereof physically altered from its original form, but remaining in a fresh state, which offer consumers high nutrition, convenience and value. Since their origin in the early 1980's, the consumption of fresh-cut fruits and vegetables has been characterized by a tremendous growth due to their health and convenience benefits (Rico et al. 2007). However, comparing to total volume of fruits and vegetables sold in Europe, the market share of fresh cut products represents only few percent points. In 2010, the fresh cut fruits and vegetables shared only 1% and 5% of total consumption of fruits and vegetables in Europe (Baselice et al. 2014). Therefore, the development of innovative products is still needed in order to improve the consumption of fresh cut fruit and vegetables.

The odor and flavor attributes are important factors influencing the purchasing motivation of consumers, in addition to the appearance characters of fresh cut fruits and vegetables, which has already been extensively studied. Essential oil is a kind of aromatic product containing more or less volatile substances with odorous impact. It is acceptable by consumers at appropriate concentration when applied in foods (Dias et al. 2015) and can improve the flavor of potato crisps when added in the oil used for frying (Chammem et al. 2015). Potato (*Solanum tuberosum*) is the third largest food crop in the world (Wang et al. 2015), and minimally processed potatoes are popular commodities due to the popularity of potatoes chips, French fries and baked potatoes. For these reasons, aromatic minimally processed potatoes could be very attractive for consumers as well as industries.

However, potato tissues are relatively tight, impermeable to water and gases. Previous study confirmed that inserting an external solution into potato tubers by simple immersion proved to be very difficult (Hironaka et al. 2011). An auxiliary technology may be therefore needed in order to impregnate potatoes with an aromatic solution.

Vacuum impregnation (VI) satisfies that requirement as it is an emerging technology that has been recognized as a promising tool for the introduction of solutes into the

internal structure of some porous food products, due to the action of hydrodynamic mechanisms promoted by pressure changes. Fruits and vegetables have a great amount of intercellular space which are occupied by gas and offer the possibility to be impregnated by external solutions (Fito et al. 2001). VI has been widely used to incorporate into the internal structure of fruit and vegetable porous matrices with various solutes. Previous studies have successfully been conducted to incorporate into the internal structure of fruit and vegetable porous matrices external solutions containing anti-browning agents, microbial preservatives, cryoprotectants (Panarese et al. 2014) or components for quality and nutritional improvements (Alzamora et al. 2005; Betoret et al. 2015; Betoret et al. 2007), including potatoes (Hironaka et al. 2011; Hironaka et al. 2014; Hironaka et al, 2015; Garzarella et al. 1990). To the best of our knowledge the odor and/or flavor enrichment of minimally processed potatoes by VI has not been studied yet.

The aim of the present research was to study the enrichment of minimally processed potato sticks with rosemary essential oil through VI in order to obtain an innovative aromatic minimally processed potato product, and to evaluate the physico-chemical, sensorial and microbiological properties of impregnated potato sticks during refrigerated storage and after frying.

Materials and methods

Raw materials

Potato tubers (*Solanum tuberosum*), cultivar Daisy, normally used for industrial processing of french-fries, were grown in Emilia Romagna region and collected in January 2017 by the company Pizzoli S.R.L. (Italy). Tubers of uniform size (40-50 g) without superficial defects and with an average dry matter content of 19.6 ± 0.3 % were selected and stored in the dark at 10 °C and 90% relative humidity (RH) for a maximum of 2 weeks, before treatment.

Aromatic solution

The rosemary oil suspension used in this work was provided by DKS AROMATIC S.R.L. (Italy). The composition of the suspension was: water, propylene glycol (E1520), hydroxy propyl distarch phosphate (E1442), octenil succinate (E1450), rosemary essential oil, xanthan gum (E415) and potassium sorbate (E202).

Vacuum impregnation process and sample preparation

The vacuum impregnation (VI) treatment was performed using a system that allowed the control of both the pressure acting on the impregnating solution during the process and the velocity of vacuum level and atmospheric pressure restoration. The potato samples, obtained as described below, and the impregnating solution were placed in a cylindrical glass chamber (10 L volume), which was connected with a rubber tube to a vacuum pump (SC 920, KNF ITALY, Milan, Italy). The system was controlled by an electro-mechanical control unit (AVCS, S.I.A., Bologna, Italy).

Potato tubers were washed with tap water and then dipped in a 200-ppm sodium hypochlorite solution for 2 min for sanitizing the surface. The skins were peeled using a sharp knife and then cut by a manual cutter into pieces of rectangular shape of 1 x 1 x 7 cm. The VI solutions were prepared at 4%, 8% and 12% (w:v) of essential oil suspensions in distilled water.

Potatoes sticks were immersed in the rosemary solutions (product weight/solution weight 1:1.5) and a sub-atmospheric pressure of 60 ± 10 mbar was applied for 30 min followed by a relaxation time at atmospheric pressure of 30 min. VI parameters (vacuum time and relaxation time) were chosen on the basis of preliminary tests (data not shown). Afterwards the excess liquid on the samples surface was removed lightly with absorbent paper. Control samples were obtained by immersing potato sticks in distilled water at atmospheric pressure for the same time of the whole VI treatment (about 70 min). The prepared samples were the following: control (dipped in distilled water); 0% (subjected to VI in distilled water); 4% (subjected to VI in 4% rosemary oil suspension); 8% (subjected to VI in 8% rosemary oil suspension) and 12% (subjected to VI in 12% rosemary oil suspension). Each VI treatment has been

replicated at least three times.

After the treatment, potato sticks were packed in polypropylene trays, sealed with a medium permeability polyethylene film of 200 μm thickness. 40 packages with about 100 g of potatoes sticks for each sample were prepared. The samples were stored at 4 °C for 14 days and sampled regularly for analytical determinations. Analyses of minimally processed potato samples were carried out after 0, 3, 7, 10 and 14 days on sticks from at least three packages for each sampling time.

Moreover, after 0, 7 and 14 days of storage, analytical determinations were carried out also on the product after frying. Frying was carried out with a home fryer mod. F989 (De Longhi, Italy), using peanut oil (w:w oil:sample ratio 20:1) at 180 °C for 5 min. After frying the potatoes were drained of excess oil and gently dried on paper towels for 5 min before the analytical determinations.

Analytical determinations

Physico-chemical properties of potatoes and rosemary solutions

Water activity (a_w) of fresh potatoes and rosemary oil solutions were measured by Aqua LAB (3TE, Decagon Devices, Inc.).

The pH value and the viscosity of the solutions were measured using a pH-meter (Cdberscan pH 510, Eutch Instruments, Singapore) and a vibrational viscometer mod. Viscolite 700 (Hydramotion Ltd., York, England).

The bulk density (ρ_b) of fresh potatoes was determined by measuring the volume of the sample (about 3 g) by displacement using a pycnometer with glycerin as the reference liquid. The solid–liquid density (ρ_s) was measured on the sample previously de-aired in order to eliminate pores and air. The porosity (ε) of fresh potato tissue was calculated from the values of ρ_b and ρ_s by the following equation (Nieto et al. 2004):

$$\varepsilon = \frac{\rho_s - \rho_b}{\rho_s}$$

145 **Mass transfer parameters**

146 To determine the total mass change due to VI, the weight of the potato sticks was
147 measured before (M_0) and after (M_t) the VI treatment. The mass changes (ΔM) were
148 calculated by the following equation (Neri et al. 2016):

149

150 $$\Delta M = \frac{M_t - M_0}{M_0} \times 100$$

151

152 **Moisture content**

153 The moisture content of the samples was determined gravimetrically by difference in
154 weight before and after drying at 70°C, until a constant weight was achieved.

155

156 **Respiratory gases in the package headspace**

157 The composition of O_2 and CO_2 (%) in the package headspace during storage was
158 determined by a gas analyser "Check Point O_2/CO_2" mod. MFA III S/L
159 (Witt-Gasetechnik, Witten, Germany). At each sampling time, measurements were
160 obtained for at least three packages for each sample.

161

162 **Color Measurement**

163 A spectrophotocolorimeter mod. Colorflex (HUNTERLAB ColorFlexTM, Reston, Virginia)
164 was used to measure surface colour of minimally processed and fried samples (D65
165 illuminant and 10° standard observer). For each piece, measurements were performed
166 on each side. The L* and a* parameters of the CIELAB scale were considered. Results
167 were expressed as average of 10 measurements for sample.

168

169 **Texture Measurement**

170 The texture of the potato samples was evaluated by subjecting each sample to a
171 penetration test using a dynamometer mod. Texture Analyzer TA. HDi500 (Stable
172 Micro Systems, Surrey, U.K) equipped with a load cell of 5 kg, using a cylindrical

shape stainless steel probe of 2 mm diameter. The penetration rate was 1 mm/min and the depth was 0.5 cm. From the graphs obtained, the parameter of hardness (N) that represents the maximum force required to make the penetration of the sample, was extrapolated.

Microbiological analysis

The total aerobic counts, psychrophilic bacteria, molds and yeasts counts, and coliforms were carried out for microbiological analysis of potato samples during 14 days of storage. 50 g of each sample were added to 50 mL of saline (0.85% sodium chloride solution) in a sterile polyethylene bag and mixed by a stomacher (Seward Stomacher 400, UK) for 2 min at high speed. Further decimal dilutions were made with sterile saline. Spread plate method was applied to enumerate the total aerobic counts, psychrophilic bacteria, molds and yeasts counts using Plate Count Agar (PCA) and Yeast Extract-Peptone-Dextrose agar (YPD) with 200 ppm antibacterial as culture medium, respectively. Coliform were determined using the pour plate method and Violet Red Bile Agar (VRBA) as medium. The storage of the plates took place at 30 °C for 48 h for total aerobic and molds and yeasts counts, at 37 ° C for 24 h for coliforms and at 10 ° C for 10 days for psychrophilic bacteria.

The microbiological analysis was conducted on the raw product after 0, 7, 14 days of storage. Each micro-organism was determined in two sample trays and two replicates for each treatment at each sampling time, and the results were transferred to $\log_{10}$ (CFU/g).

Analysis by gas chromatography with mass spectrometry (GC-MS)

The volatile compositions analysis of potato sticks after impregnation with rosemary essential oil and/or frying was performed using a VG Platform II GC-MS system equipped with a DB-5MS capillary column (30 mm x 0.25 mm i.d.; film thickness 0.25 m), both for raw potato sticks and fried ones.

The solid phase microextraction (SPME) was used to extract the volatile components

of the essential oil of rosemary. After being taken out from refrigerated storage room (4 ° C), the potato trays were maintained at 30 ° C for 20 min. The volatile components were isolated using a SPME fiber for 5 min at room temperature (22 ° C). For GC-MS detection, an electron ionization system of electrons with ionization 70 eV energy has been used. Helium was employed as the carrier gas, at a flow rate of 0.8 mL/min. The temperature of the injector and transfer line of the MS detector were set at 160 ° C and 265 ° C. The column temperature was initially set at 50 ° C and maintained for 10 min, and then increased gradually to 160 ° C at a rate of + 5 ° C/min, kept at this temperature for 2 min and finally brought to 280 ° C at the speed of +5 ° C / min. The components were identified by comparison of their relative retention times and mass spectra with those of the standard (for major components) - NIST library data of the GC-MS system and the literature data (Jiang et al., 2011). The GC-MS analysis was conducted at 0, 7, 14 days of storage, both to the minimally processed potatoes and fried ones.

Sensory descriptive analysis (DA)

Sensorial assessment of fried potato samples has been performed by a panel of 10 fully trained assessors (age between 25 and 50 years, five females and five males) recruited because of their previous experience in descriptive sensory analysis (staff and PhD students at the Campus of Food Science, University of Bologna, Cesena, Italy) and their familiarity with the product.

In order to prevent panelist fatigue, the attribute list has been minimized. After the descriptors selection, training sessions have been carried out, following the guidelines of the ISO 13299:2010. The test sessions were performed in a closed room in separate tasting booths. A final list of seven descriptors have been selected and a hedonistic scale from 0 to 8 has been used. In **Table 1** sensory terms, definitions and reference of each descriptor are reported.

Randomized blocks of fried potato samples, labelled with random three-digit codes, have been analysed. During sensorial analysis, water has been used to cleanse the

palate and between each sample analysis 2-min break has been allowed.

Sensory analysis was performed after frying and room temperature reconditioning for 3 min, until the samples reached an acceptable temperature for consumption (50 ° C).

Statistical analysis

The statistically significant differences among the treatments were analyzed by SPSS 22.0 (SPSS Inc., Chicago, IL, USA) by analysis of variance (ANOVA) using the LSD test for comparison of the data ($p < 0.05$).

Results

Physico-chemical properties of potatoes and rosemary solutions

The pH, water activity (a_w), viscosity and porosity values of raw potatoes and rosemary oil solutions are reported in **Table 2**.

The solutions used for the impregnation, with concentration of 4, 8 and 12% of essential oil were not significantly different for either pH (between 2.99 and 2.96), water activity (between 1.000 and 0.997), nor viscosity (between 1 and 0.97).

The pH of the solutions was much lower than that of the potatoes, while the water activity showed no significant differences. The porosity of the potatoes was 1.87 ± 0.45%. This finding is in agreement with previous studies (Alzamora et al. 2005; Hironaka et al. 2015).

Weight gain after treatment

After immersion in distilled water for 70 min, a weight gain of about 8% was observed. By applying vacuum to the sample immersed in water (0% solution), the weight increase was approximately 12%. The results also showed that increasing the concentration of rosemary oil proportionally decreased weight gain, until reaching values similar to the ones achieved by immersion in distilled water at atmospheric pressure. However, all weigh gain was in the range of 8-12 % (data not showed).

Physico-chemical parameters and microbial loads of minimally processed potatoes during storage

The moisture content of fresh potatoes was 80.37 ± 2.76%. This parameter did not show significant differences during storage, ranging from 78.83 ± 2.19 to 82.96 ± 1.57 % (data not reported).

The headspace gas evolution of respiratory gases is reported in **Table 3**. All the samples showed a progressive decrease of O_2 and an increase of CO_2 during storage, promoted by potato tissue respiration and packaging permeation. The sample subjected to vacuum impregnation with only distilled water (0%) showed lower O_2 and higher CO_2 values compared to the sample immersed in water at atmospheric pressure, indicating a higher respiration rate. The samples impregnated with solutions containing rosemary extract showed results similar to sample 0%.

In **Table 3**, the brightness (L *) and red index (a *) values measured in minimally processed potato samples during storage are reported. Immediately after the treatment, the vacuum impregnated samples showed significantly lower L* and a* values compared to the sample immersed in water at atmospheric pressure. During storage, a progressive decrease in the value of L* and an increase of a* were observed for the control sample, probably due to enzymatic browning. In the 0% and 4% impregnated samples, no significant differences were found with regard to the brightness (L*) between the beginning and the end of storage, but only in relation to a*. However, samples impregnated with solutions containing 8% and 12% of essential oil, were characterized by a higher browning level until the 10^{th} day of storage, but only in terms of a*.

Immediately after treatment, the samples subjected to VI showed lower average hardness values (data not reported) compared to the sample impregnated at atmospheric pressure (control), although the differences were not statistically significant because of the high natural variability of the data (typical of texture measurements of cell turgid vegetables). In any case, a reduction in hardness may be due to an alteration of the potato structure due to the application of the vacuum. On

289 the contrary, during storage, the hardness was almost unchanged and there were no
290 differences among the various samples (data not reported).
291 The microbial loads related to, respectively, mesophilic bacteria, molds and yeasts and
292 coliforms have been detected in minimally processed potato samples during storage
293 (**Table 3**). The initial values of total mesophilic loads were between 1.5 and 3.5 log
294 CFU/g with differences between samples probably due to natural variability, since
295 they did not show any relation with the treatments or the concentration of the
296 impregnating solution. During storage the values increased, but only slightly, up to
297 values between 3 and 4 log CFU/g after 14 days, without significant differences
298 between the samples.
299 Yeast and molds count was rather low during all the storage, rising above the limit of
300 detection in only a few samples at the end of the storage, but not with a significant
301 trend. Similarly, the charges of coliforms were very similar in all the samples during
302 storage, remaining almost unchanged and almost below the limit of detection.
303 In **Table 4** the evolution of the amount of volatile compounds in fresh potato sticks
304 during storage is reported. The main detected compound was eucalyptol, followed by
305 camphor, 3-methyl-apopinene, α-pinene, 1,3,8-p-menthatriene, α-camphene and
306 linalool. The quantity of the main volatile compounds like eucalyptol, camphor,
307 generally decreased along with storage time. While the quantity of some other
308 compounds like 1,3,8-p-menthatriene, α-pinene increased during the first week and
309 then decreased. At the end of storage, camphor, 1,3,8-p-menthatriene and α-linalool
310 were no longer detectable. At the last day of storage, the presence of ethanol was
311 detected, probably due to the endogenous cell metabolism and/or growth of
312 microorganisms.
313
314 **Physico-chemical and sensorial parameters of fried french-fries**
315 The brightness (L*) and red index (a*) values detected on fried potatoes were
316 consistent with those of minimally processed samples in terms of changes during
317 storage (**Table 5**).

Immediately after frying, the L* and a* values were higher in samples impregnated with the 4, 8 and 12% rosemary oil solutions compared to the control and 0% samples. However, while values did not change during storage for 0% impregnated samples the 8 and 12% samples have undergone a decrease of both L* and increase of a*. Actually, the a* value after frying showed a progressive increase with the increase of the storage time in all the groups. Nevertheless, while at the beginning the differences among the samples were minimal, after 14 days of storage, VI samples showed significantly higher values than the control sample, impregnated at atmospheric pressure. Moreover, the 12% sample was characterized by the highest value compared to the others. In terms of volatile compounds, the frying procedure seems to flat the differences detected on minimally processed samples, even if the persistency of rosemary aromatic compounds was confirmed (**Table 5**).

In terms of sensorial properties (**Figure 1**), at day 0 the parameter linked to the appearance (color uniformity) did not show significant difference among the samples. The control group has maintained similar values up to the 14th day of storage, while a decrease in the score was observed for the other samples along with storage time. Furthermore, the samples impregnated with higher concentrations of essential oil showed significantly lower values at the end of storage, which was consistent with the findings of the instrumental assessment of the color. For parameters related to smell, typical smell of potatoes and rosemary were evaluated. In general, the typical potato odor was perceived in a similar manner in all samples up to the end of storage, except for the sample 12% at time 0, in which probably the high concentration of essential oil has limited its perception. Rosemary oil was perceived proportionally to the amount, although significant differences ($p<0.05$) were not always found among samples. In addition, only in the 4% sample a decrease in the value was observed during storage, while the 8 and 12% samples have shown similar values up to the 14th day. Parameters related to flavor, typical potato and rosemary flavor, showed similar results to those obtained by the odor evaluation. The perception of the typical potato flavor tended to diminish with the increase of the concentration of rosemary of the

impregnating solution, although not always significantly. Furthermore, the perception of flavor of rosemary increased with the concentration of the solution, but in the 4% sample decreased during the storage, until the 14th day of storage, in which it was not different from control and 0% samples.

Discussion

Vacuum impregnation has been previously used for nutritional enrichment of vegetable products (Alzamora et al. 2005) and only once for the aromatic enrichment of apple slices (Comandini et al. 2010), proving to be an effective method for introducing compounds of interest into a vegetable porous tissue and hence for product innovation. However, it is important to evaluate the effect of the treatment on the qualitative properties of the obtained products and its stability during storage.

In the present study, VI has been used to introduce aromatic components in potato sticks intended for frying and some qualitative and stability aspects of the obtained products have been evaluated.

The porosity of the potato has shown to be relatively low compared to other vegetables, as already observed by other authors (Hironaka et al. 2011; Hironaka et al. 2014). This parameter is a key factor to consider for vegetable tissue to be vacuum impregnated, because it gives an indication of the entity of the intercellular spaces that are normally filled with air. The impregnation levels obtained are significantly higher compared to the porosity of the potato, indicating that apart from the intercellular spaces, some diffusion phenomena occurred and cell membrane selectivity has caused the entry of water into the cells (Yadav and Singh 2014). The application of vacuum increased further the weight gain promoting the inflow of the solution inside the pores in the tissue (Tylewicz et al. 2013), hence fostering the mass exchanges due to osmotic phenomena, which has been showed in previous studies (Fito et al 2001; Shi et al. 1995). Besides, Tylewicz et al. (2013) demonstrated that vacuum impregnation of apple tissue resulted in the formation of membrane vesicles inside the cells; this phenomenon may also occur in potato tissues, but should be further clarified.

In minimally processed products, the evaluation of the package headspace is important because is related to the product shelf-life as the development of anaerobic metabolism can lead to the formation of off-flavours and odours. In the present study, the application of vacuum lead to a faster decrease of O_2 and increase of CO_2 in the internal packages' atmosphere.

As suggested by Sanzana et al. (2011) that studied the effect of VI on various vegetables, this CO_2 increase could be due to the mechanical tissue stress as a result of vacuum application. Some authors found that after VI the onset of an anaerobic metabolism was observed, however results are generally inconsistent (Castellò et al. 2006; Igual et al. 2008; Sanzana et al. 2011). Considering that the packaging film used in this experiment was characterized by a medium barrier to gas permeability, it is not possible to obtain precise information about the type of respiratory metabolism occurring.

In previous studies, the effect of the impregnating solution was observed to decrease the respiration rate of samples (Tappi et al. 2017; Sanzana et al. 2011), indicating a possible effect of the bioactive compounds to compensate the stress caused by the application of vacuum to the tissue. In the present research, the essential oil effect on metabolic changes in the tissue was not so evident, in disagreement with previous studies that have pointed out that rosemary essential oil could cause biophysical perturbation of membranes (Pèrez-Fons et al. 2009). This difference may be explained by the concentration of the essential oil.

An important aspect in minimally processed potatoes is the enzymatic browning caused by the action of polyphenoloxidases (PPO). The initial difference observed in the colour of vacuum impregnated samples compared to the fresh one is probably due to the variation of the refractive index of the tissue as a result of the filling of the intercellular spaces with the impregnating solution, as already observed in previous works (Neri et al. 2016). However, a reduction of enzymatic browning, on the base of the colour data, following vacuum impregnation was found in the present research. A similar result has already been observed and was attributed to the reduction of the

presence of oxygen in the tissue (Tylewicz et al. 2013). However, the higher essential oil concentration lead to an increase browning phenomenon during storage, probably because of a low stability of the essential oil components, that may have undergone oxidation phenomena. This change in the appearance of the product was detected also after frying, both by instrumental color analysis and sensorial test.

Tappi et al. (2017) found that impregnating apples with a green tea extract lead to a significant change in samples color during storage. This variation was reduced by the presence of ascorbic acid in the impregnating solution that probably acted as antioxidant preserving the green tea components. The use of an antioxidant in the impregnating solution may, also in this case, reduce the color variation during storage.

Also, microbial development was not affected not by the application of vacuum nor by the presence of essential oil. Tappi et al. (2016) observed a faster microbial spoilage in minimally processed melon subjected to VI during storage. This effect was attributed to the irreversible alteration to the visco-elastic properties of the fruit tissues caused by the application of a vacuum pressure that may enhance nutrients availability for microbial growth. However, as shown by other qualitative parameters such as water content and texture, in the present study the structure of the potato tissue was not negatively affected by the vacuum treatment.

Concerning the aromatic enrichment, that was the main aim of the treatment, the number of volatile compounds detected in this study was lower compared to previous literature data on rosemary essential oil (Tawfeeq et al. 2016; Tomi et al. 2016), probably due to the dilution of the oil (4-12%) and to its low penetration into the potato tissues. In addition, other chemical and/or bio-chemical compounds in the solution and in the potato tissue may have blocked the release of volatile compounds. However, the aromatic components were well perceived by the panel, proportionally to their concentration and, at the highest concentration consistently during storage for both raw and fried product.

Conclusions

The vacuum impregnation process resulted an effective method for the aromatic enrichment of potatoes intended for frying. The aroma was successfully incorporated in the tissue although the aromatic profile of the potato samples indicated a loss of the aromatic compounds during storage. Sensory analysis has virtually confirmed the data obtained by instrumental one indicating a reduction of rosemary aroma with the storage time. The microbial load of the samples was relatively low until the end of storage, indicating that the treatment did not adversely affect the microbiological shelf-life of the samples. However, there are few issues that need further investigation in order to obtain a product with high quality characteristics and stability. The color of the potatoes during storage appeared adversely affected proportionally to the content of essential oil, probably due to oxidation phenomena. The addition of an antioxidant compound in the impregnating solution could be tested. Furthermore, the vacuum treatment seems to promote an alteration of metabolism measured by the respiration rate that should be better clarified. Anyhow, vacuum impregnation presents high potentiality to modulate the sensorial profile of porous vegetable tissue, being a cold formulation process that does not cause the thermal degradation of specific aromatic compounds of the impregnating solution.

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562 **Figure captions**

563 Figure 1. Results of sensory descriptive analysis (DA) performed on fried samples.

564

Table 1 Sensory terms, definitions and reference of each descriptor.

Sensory modalities	Descriptors	Definitions	References[a] and their values on scale
Appearance	Colour	Typicality and homogeneity of French fries colour	Weak (0); strong (8)
Aroma and flavour	Potato odour	Intensity of fried potato odour	Weak (0); strong (8)
	Rosemary odour	Intensity of rosemary odour	Weak (0); strong (8)
	Potato flavour	Intensity of fried potato flavour	Weak (0); strong (8)
	Rosemary flavour	Intensity of rosemary odour	Weak (0); strong (8)
Texture	Crispness crust	Intensity of crust crispness	Weak (0); strong (8)

[a] References established by the panel during the training section

Table 2 Physico-chemical properties of potatoes and rosemary solutions

Parameters	Potatoes	Rosemary oil solutions			
		0%	4%	8%	12%
pH	6.30±0.01[a]	-	2.99±0.01[b]	2.98±0.01[b]	2.96±0.01[b]
a_w	0.997±0.003[b]	1.000±0.000[a]	0.997±0.001[b]	0.998±0.002[b]	0.997±0.001[b]
Viscosity (cP)	-	1.0±0.0[a]	0.97±0.6[a]	0.97±0.06[a]	0.97±0.06[a]
Porosity (%)	1.87 ± 0.45	-	-	-	-

Values followed by different letters are significantly different ($P < 0.05$).

Table 3 Physico-chemical and microbial loads of packed minimally processed potato sticks during storage at 4°C.

Storage time (d)	0	3	7	10	14
Sample					
			Headspace gases		
			O_2 (%)		
Control	20.90$^{A, a}$	15.77$^{B, abc}$	9.47$^{C, ab}$	7.03$^{D, a}$	1.13$^{E, a}$
0%	20.90$^{A, a}$	15.55$^{B, b}$	8.00$^{C, b}$	1.35$^{D, b}$	0.55$^{D, a}$
4%	20.90$^{A, a}$	14.67$^{B, c}$	8.55$^{C, ab}$	3.80$^{D, c}$	0.45$^{E, a}$
8%	20.90$^{A, a}$	15.70$^{B, abc}$	8.25$^{C, b}$	2.37$^{D, bc}$	1.57$^{D, a}$
12%	20.90$^{A, a}$	14.97$^{B, abc}$	9.87$^{C, a}$	3.85$^{D, c}$	1.37$^{E, a}$
			CO_2 (%)		
Control	0$^{A, a}$	4.10$^{B, a}$	7.30$^{C, a}$	8.43$^{C, b}$	10.43$^{D, a}$
0%	0$^{A, a}$	4.00$^{B, a}$	7.77$^{C, a}$	10.85$^{D, a}$	10.85$^{D, a}$
4%	0$^{A, a}$	4.47$^{B, a}$	7.90$^{C, a}$	10.55$^{D, a}$	11.50$^{D, b}$
8%	0$^{A, a}$	4.05$^{B, a}$	7.60$^{C, a}$	10.93$^{D, a}$	11.47$^{D, ab}$
12%	0$^{A, a}$	4.50$^{B, a}$	7.40$^{C, a}$	10.00$^{D, a}$	10.53$^{D, a}$
			Colour		
			L*		
Control	70.15$^{A, a}$	66.20$^{AB, a}$	66.93 $^{AB, a}$	64.67 $^{B, a}$	63.44 $^{B, a}$
0%	62.90$^{A, b}$	67.77$^{A, a}$	66.87 $^{A, a}$	65.81 $^{A, a}$	64.90 $^{A, a}$
4%	63.69$^{A, b}$	66.50$^{A, a}$	64.92 $^{A, a}$	63.20 $^{A, a}$	62.21 $^{A, a}$
8%	65.03$^{A, b}$	66.81$^{A, a}$	65.21 $^{AB, a}$	62.71 $^{AB, a}$	61.45 $^{B, a}$
12%	64.51$^{A, b}$	66.65$^{A, a}$	65.48 $^{AB, a}$	61.71 $^{AB, a}$	59.81 $^{B, a}$
			a*		
Control	-0.46 $^{A, a}$	0.21 $^{AB, a}$	0.27 $^{AB, a}$	0.59 $^{B, c}$	2.09 $^{C, a}$
0%	-1.08 $^{A, b}$	0.16 $^{B, a}$	0.14 $^{B, a}$	1.09 $^{BC, bc}$	1.57 $^{C, a}$
4%	-1.04 $^{A, b}$	0.45 $^{B, a}$	0.40 $^{B, a}$	2.18 $^{C, ab}$	2.66 $^{C, a}$
8%	-0.99 $^{A, b}$	0.27 $^{B, a}$	0.63 $^{B, a}$	2.58 $^{C, ab}$	2.78 $^{C, a}$
12%	-0.75 $^{A, ab}$	0.18 $^{B, a}$	0.37 $^{B, a}$	3.12 $^{C, a}$	2.94 $^{C, a}$
			Microbial load (log CFU g^{-1})		
			Mesophilic bacteria		
Control	3.13 $^{A, a}$	-	3.46 $^{A, a}$	-	3.36 $^{A, a}$
0%	3.35 $^{A, a}$	-	3.33 $^{A, a}$	-	3.53 $^{A, a}$
4%	3.54 $^{A, a}$	-	3.63 $^{A, a}$	-	3.67 $^{A, a}$
8%	1.75 $^{A, b}$	-	3.18 $^{B, b}$	-	3.83 $^{C, a}$
12%	2.12 $^{A, b}$	-	3.52 $^{B, a}$	-	3.76 $^{B, a}$
			Psycotrophic bacteria		
Control	0 $^{A, a}$	-	0 $^{A, a}$	-	2.48 $^{B, a}$
0%	0 $^{A, a}$	-	0 $^{A, a}$	-	2.84 $^{B, a}$
4%	0 $^{A, a}$	-	0 $^{A, a}$	-	2.90 $^{B, a}$
8%	0 $^{A, a}$	-	0 $^{A, a}$	-	0.56 $^{B, b}$
12%	0 $^{A, a}$	-	0 $^{A, a}$	-	0.91 $^{B, b}$
			Yeasts and moulds		

Control	1.60 $^{A, a}$	-	3.02 $^{B, a}$	-	3.80 $^{B, a}$
0%	1.45 $^{A, a}$	-	3.23 $^{B, a}$	-	3.78 $^{C, a}$
4%	1.60 $^{A, a}$	-	2.98 $^{B, a}$	-	3.57 $^{C, a}$
8%	0 $^{A, b}$	-	1.52 $^{B, b}$	-	3.58 $^{C, a}$
12%	0 $^{A, b}$	-	1.95 $^{B, b}$	-	3.15 $^{C, b}$

Capital letters indicate significant differences ($p<0.05$) among the same sample at different sampling time, lowercase letters indicate significant differences ($p<0.05$) among samples at the same sampling time.

Table 4 Volatile compounds (expressed as area/weight 10^{-6}) detected in minimally processed potato samples at 0, 7 and 14 days of storage at 4 °C.

	Control			0%			4%			8%			12%		
Storage time (d)	0	7	14	0	7	14	0	7	14	0	7	14	0	7	14
Volatile compound															
Ethanol	ND	ND	0.48 $^{A, a}$	ND	ND	0.53 $^{A, a}$	ND	ND	0.32 $^{A, a}$	ND	ND	0.53 $^{A, a}$	ND	ND	0.33 $^{A, a}$
3-methyl-apopinene	ND	ND	ND	ND	ND	ND	4.52 $^{A, b}$	8.99 $^{B, a}$	4.99 $^{A, b}$	8.85 $^{A, a}$	11.23 $^{A, a}$	4.34 $^{B, b}$	9.56 $^{A, a}$	10.44 $^{A, a}$	15.84 $^{B, a}$
Camphene	ND	ND	ND	ND	ND	ND	1.97 $^{A, a}$	2.40 $^{A, a}$	0.47 $^{B, a}$	3.84 $^{A, b}$	3.89 $^{A, b}$	0.42 $^{B, a}$	4.30 $^{A, b}$	3.76 $^{A, b}$	3.52 $^{A, b}$
α-pinene	ND	ND	ND	ND	ND	ND	2.45 $^{A, a}$	5.26 $^{B, a}$	3.34 $^{A, a}$	6.07 $^{A, b}$	7.11 $^{A, b}$	2.69 $^{B, a}$	6.76 $^{A, b}$	6.59 $^{A, b}$	8.82 $^{B, b}$
Eucalyptol	ND	ND	ND	ND	ND	ND	54.83 $^{A, a}$	19.71 $^{B, a}$	11.1 $^{C, a}$	94.71 $^{A, b}$	36.01 $^{B, b}$	12.75 $^{C, a}$	112.91 $^{A, b}$	32.24 $^{B, b}$	14.66 $^{C, a}$
1,3,8-p-menthatriene	ND	ND	ND	ND	ND	ND	5.32 $^{A, a}$	6.01 $^{A, a}$	ND	9.46 $^{A, b}$	8.18 $^{A, b}$	ND	6.72 $^{A, a}$	8.41 $^{A, b}$	ND
3-octanol	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.55 $^{A, a}$	ND	ND
Camphor	ND	ND	ND	ND	ND	ND	15.26 $^{A, a}$	3.11 $^{B, a}$	ND	19.09 $^{A, b}$	8.82 $^{B, b}$	ND	20.64 $^{A, b}$	7.13 $^{B, b}$	ND
α-Linalool	ND	ND	ND	ND	ND	ND	3.32 $^{A, a}$	ND	ND	4.61 $^{A, b}$	ND	ND	4.45 $^{A, b}$	1.47 $^{B, a}$	ND
p-Menth-1-en-4-ol	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.20 $^{A, a}$	ND	ND	0.77 $^{A, b}$	ND	ND
Borneol, (1S,2R,4S)-(-)-	ND	ND	ND	ND	ND	ND	2.44 $^{A, a}$	2.31 $^{A, a}$	ND	3.95 $^{A, b}$	6.36 $^{B, c}$	ND	3.96 $^{A, b}$	4.99 $^{B, b}$	7.08 $^{C, a}$
p-Menth-1-en-8-ol	ND	ND	ND	ND	ND	ND	1.85 $^{A, a}$	ND	ND	2.71 $^{A, b}$	ND	ND	2.50 $^{A, b}$	ND	ND

ND, not detected

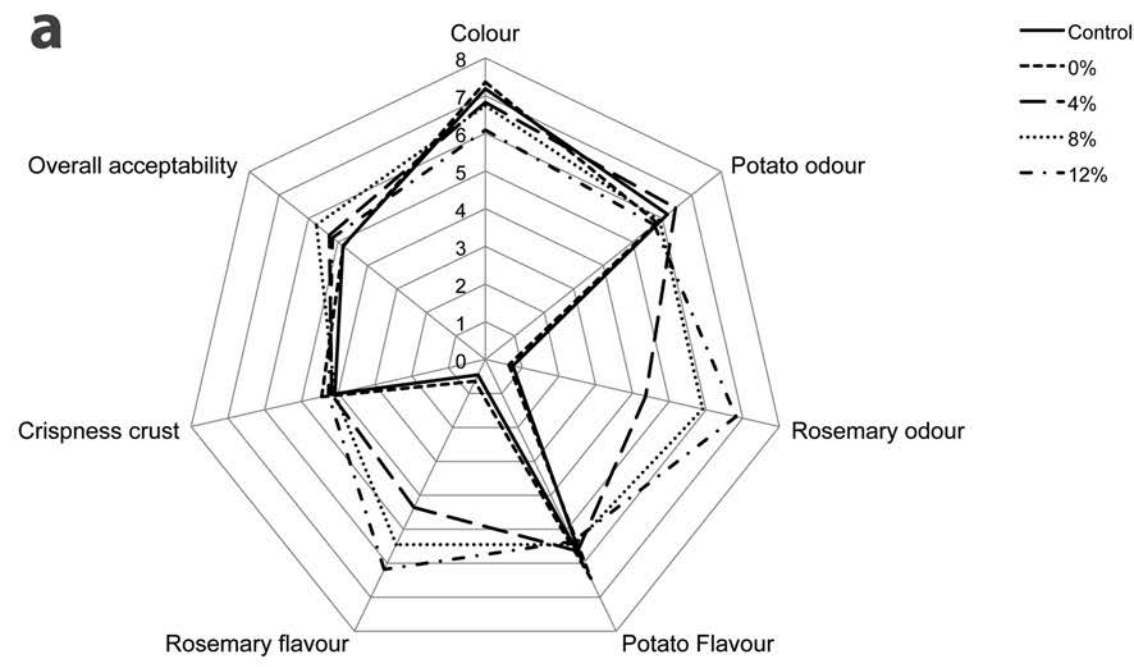
Capital letters indicate significant differences ($p<0.05$) among the same sample at different sampling time, lowercase letters indicate significant differences ($p<0.05$) among samples at the same sampling time.

Table 5 Volatile compounds (expressed as area/weight 10^{-6}) and color parameters detected in fried potato samples at 0, 7 and 14 days of storage at 4 °C.

	Control			0%			4%			8%			12%		
Storage time (d)	0	7	14	0	7	14	0	7	14	0	7	14	0	7	14
Volatile compound															
2-methyl-propanal	0.84 $^{A, a}$	0.77 $^{A, a}$	0.56 $^{A, a}$	0.78 $^{A, a}$	0.66 $^{A, a}$	0.60 $^{A, a}$	0.59 $^{A, a}$	0.71 $^{A, a}$	0.87 $^{A, a}$	0.67 $^{A, a}$	0.82 $^{A, a}$	0.57 $^{A, a}$	0.85 $^{A, a}$	0.69 $^{A, a}$	0.77 $^{A, a}$
Eucalyptol	ND	ND	ND	ND	ND	ND	93.22 $^{A, a}$	65.40 $^{B, a}$	37.21 $^{C, a}$	113.41 $^{A, b}$	92.35 $^{B, b}$	50.75 $^{C, ab}$	163.21 $^{A, c}$	122.40 $^{B, c}$	53.45 $^{C, b}$
Pyrazine	4.38 $^{A, a}$	3.59 $^{A, a}$	5.28 $^{A, a}$	4.43 $^{A, a}$	5.25 $^{A, a}$	5.07 $^{A, a}$	5.13 $^{A, a}$	3.87 $^{A, a}$	4.46 $^{A, a}$	4.59 $^{A, a}$	5.58 $^{A, a}$	5.29 $^{A, a}$	5.48 A, a	3.52 $^{A, a}$	3.99 $^{A, a}$
Camphor	ND	ND	ND	ND	ND	ND	28.59 $^{A, a}$	16.38 $^{AB, a}$	11.20 $^{B, a}$	44.26 $^{A, ab}$	19.29 $^{AB, b}$	11.08 $^{B, b}$	63.65 $^{A, b}$	36.02 $^{B, c}$	15.71 $^{C, c}$
Colour															
L*	62.49 $^{B, b}$	66.77 $^{A, a}$	64.28 $^{AB, a}$	63.97 $^{A, a}$	64.83 $^{A, ab}$	62.52 $^{A, a}$	67.42 $^{A, a}$	67.56 $^{A, a}$	63.13 $^{B, a}$	65.24 $^{A, a}$	61.43 $^{B, b}$	62.41 $^{AB, a}$	66.86 $^{A, a}$	62.20 $^{B, b}$	58.93$^{B, b}$
a*	-0.97 $^{A, a}$	-0.04 $^{A, c}$	0.86 $^{B, d}$	-1.69 $^{A, b}$	-0.01 $^{B, c}$	1.68 $^{C, c}$	-1.71 $^{A, b}$	-0.28 $^{B, bc}$	1.90 $^{C, c}$	-2.09 $^{A, b}$	0.86 $^{B, b}$	2.38 $^{C, b}$	-1.90 $^{A, b}$	0.33 $^{B, a}$	4.16 $^{C, a}$

ND, not detected

Capital letters indicate significant differences ($p<0.05$) among the same sample at different sampling time, lowercase letters indicate significant differences ($p<0.05$) among samples at the same sampling time.


a
Colour
Potato odour
Rosemary odour
Potato Flavour
Rosemary flavour
Crispness crust
Overall acceptability
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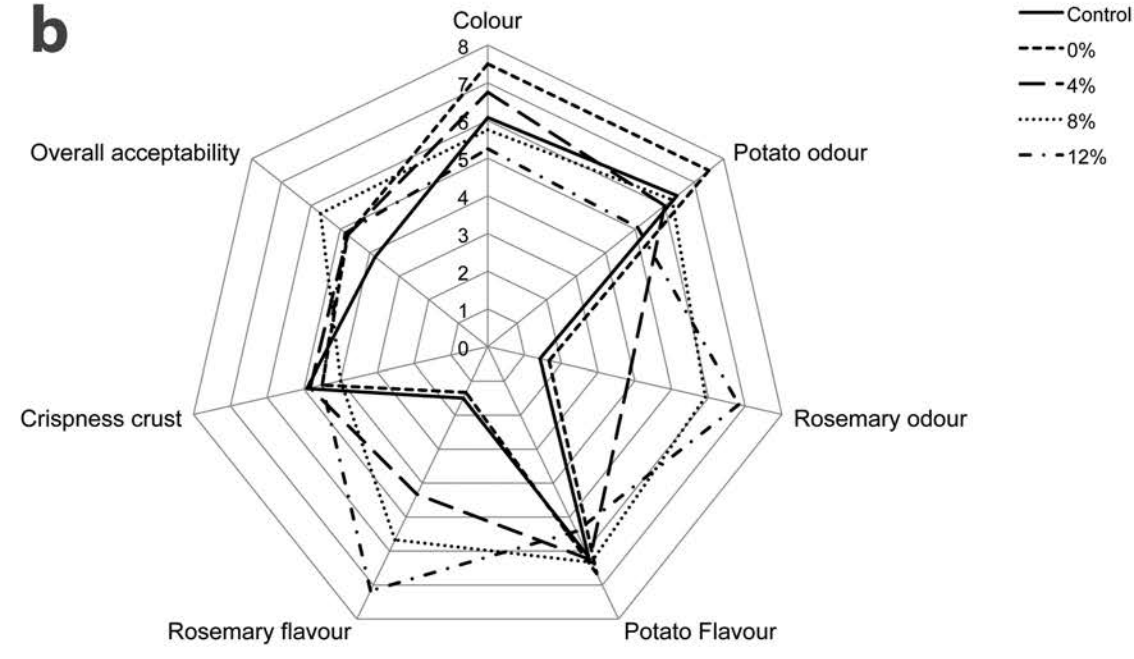
b
Colour
Potato odour
Rosemary odour
Potato Flavour
Rosemary flavour
Crispness crust
Overall acceptability
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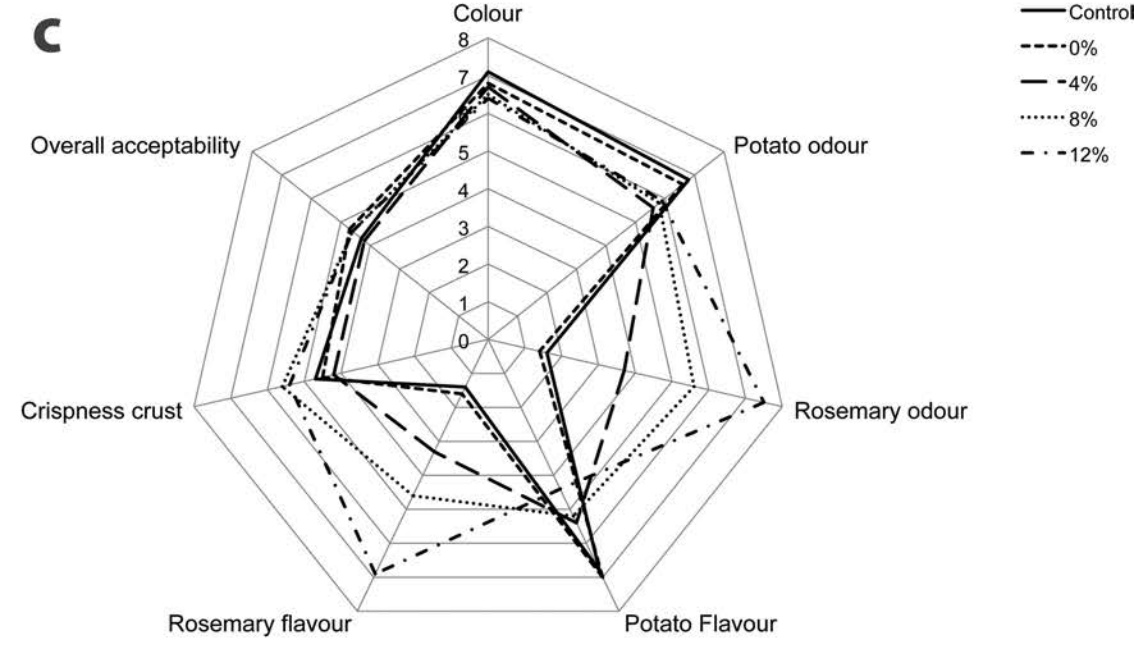
c
Colour
Potato odour
Rosemary odour
Potato Flavour
Rosemary flavour
Crispness crust
Overall acceptability
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