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Technological characteristics and selected bioactive compounds of *Opuntia dillenii* cactus fruit juice following the impact of pulsed electric field pre-treatment

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ABSTRACT

Selected technological characteristics and bioactive compounds of juice pressed directly from the mash of whole *Opuntia dillenii* cactus fruits have been investigated. The impact of pulsed electric fields (PEF) for a non-thermal disintegration on the important juice characteristics has been evaluated in comparison to microwave heating and use of pectinases. Results showed that the cactus juice exhibited desirable technological characteristics. Besides, it also contained a high amount of phenolic compounds being the major contributors to the overall antioxidant activity of juice. HPLC-DAD/ESI-MS^n measurements in the fruits' peel and pulp showed that isorhamnetin 3-*O*-rutinoside was determined as the single flavonol found only in the fruit's peel. Treating fruit mash with a moderate electric field strength increased juice yield and improved juice characteristics. Promisingly, the highest release of isorhamnetin 3-*O*-rutinoside from fruit's peel into juice was maximally achieved by PEF.

1. Introduction

Consumer's demand for high-quality healthy foods rich in natural bioactive compounds such as vitamins, phenolic compounds (i.e. flavonoids), pigments, dietary fibers, etc is drastically increasing. Therefore, improving the content of bioactive compounds or keeping them at least stable represents a present challenge in food processing. In this context, the emerging and innovative non-thermal technologies such as *pulsed electric fields* (PEF), *high hydrostatic pressure* (HHP), *cold atmospheric pressure plasma* have been considered as promising applications for preserving foods or enriching foods with more available bioactive compounds. Non-thermal cell disintegration using PEF is a promising preservation technology for liquid or semi-liquid, viscous food products (such as juices, milk, yogurt, etc.). The impact of pulses of high voltage on the permeability of biological membranes leads to the inactivation of microorganisms, consequently improving shelf life and quality of the preserved food (e.g., Torregrosa, Esteve, Frígola, & Cortés, 2006). On the other hand, cell disintegration in plant tissue by PEF treatment might enhance also the release of bioactive substances such as polyphenols from the cell cytoplasm (Puértolas, Saldaña, Condón, Álvarez, & Raso, 2010). However, influence of PEF pre-treatment on *Opuntia* cactus fruits has not yet been comprehensively reported, so far.

Opuntia, the most important genus from *Cactaceae* with regard to food products, includes a huge number of species and varieties. Up to now, the most common and heavily consumed cactus fruits are from *Opuntia ficus-indica*. Though considerable nutritional value, their high pH value and total soluble solids (TSS) values represent a challenge for juice processing. Recent studies showed that even some other *Opuntia* spp. fruits might deserve more attention for exploiting them as food products. One of them is *Opuntia dillenii* (in the past often misleadingly described as *Opuntia macrorhiza*) (Moussa-Ayoub, El-Samahy, Rohn, & Kroh, 2011). Under Egyptian cultivation conditions, the harvest season of *O. ficus-indica* fruits starts late June and to early September, while harvest season of *O. dillenii* fruits starts mid-November and to mid-January. In comparison to cactus *O. ficus-indica* fruits, cactus *O. dillenii* fruits exhibit desirable technological characteristics such as low pH,

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moderate TSS, plain fruit taste, and a plentiful deep red-purple color distributed in the entire fruit. Similar to xoconostles, *O. dillenii* cactus fruits are comparatively acidic and small varying in weight (often 10–20 g) and size between 20 mm to 50 mm and 15 mm to 30 mm in length and width, respectively. The fruit consists of a deep red-purple thick peel with a thin fruit skin and a red-purple pulp full with small, hard seeds. The plain acidic taste and high color impact makes the *O. dillenii* fruit's juice a highly desirable juice for mixing with other highly flavored fruit juices (i.e. strawberry juice). To the best of our knowledge, the *O. dillenii* fruit's juice was not investigated as a potential alternative to the common products of *O. ficus-indica* fruit's juice. Although pharmacological features, nutritional value and bioactivity of *O. dillenii* have been described (Chang, Hsieh, & Yen, 2008; Díaz Medina, Rodríguez Rodríguez, & Díaz Romero, 2007; Gao et al., 2015), information about bioactive compounds such as flavonols is still rare. With regard to the fruits, Moussa-Ayoub, El-Samahy, Rohn, et al. (2011) investigated the flavonols, betacyanins, phenolic contents, and antioxidant activity of *O. dillenii* fruits in comparison to red *O. ficus-indica* fruits. They reported that *O. dillenii* fruit is comparatively richer in betacyanins and phenolic compounds and correspondingly higher in antioxidant activity. However, total flavonol content of *O. dillenii* fruit was lower than content of *O. ficus-indica* fruit (Moussa-Ayoub, El-Samahy, Kroh, & Rohn, 2011; Moussa-Ayoub, El-Samahy, Rohn, et al., 2011; Moussa-Ayoub et al., 2014). Isorhamnetin-3-*O*-rutinoside, as the dominant flavonol, was found in the *O. dillenii* fruit's peel, while the fruit's pulp has no flavonol at all. Taking the low costs for cultivation into account, *O. dillenii* exhibits a high nutritional valuable profile and bioactivity for being a promising food product. In the context of fruit products, juices or nectars seem to be the most common products all over the world. However, in conventional cactus juice production (using maceration and/or thermal (pre)-treatments), the high viscosity of mashed fruits might cause a low yield of the juice and furthermore, a corresponding loss of bioactive compounds in the fruit waste (in particular those bioactives occurring in the fruit's peel). Further thermal treatments of the raw material during juice processing might lead to adverse sensory properties (e.g., brown color, off-flavors). To the best of our knowledge, studies on an enrichment of cactus fruit juice by increasing their flavonol content resulting from an improved recovery from the fruit or better retention during processing have not been reported yet. Therefore, the impact of one of the most innovative and emerging non-thermal food technologies – PEF – was tested for its applicability as a pre-treatment process for increasing *O. dillenii* juice yield, its flavonol content, as well as for improving or maintaining juice characteristics. The present study evaluated the impact of a cell disintegration using PEF in comparison to microwave heating (representing an intense thermal treatment) and enzymatic pre-treatments (representing a typical treatment, especially for high viscous products) on some selected technofunctional characteristics (pH, TSS, color, viscosity, and yield) of *Opuntia dillenii* juices. This has not been comprehensively done before, neither for Opuntia products nor for products of such a high viscosity. Besides, the rheological properties of the juices produced were evaluated. Moreover, the yields of selected bioactive compounds (flavonols and betacyanins), total phenolic content, and antioxidant activity of fruit juices and the residues remaining were compared.

2. Materials and methods

2.1. Plant sample preparation and pre-treatments

Cactus fruit samples from *O. dillenii* were collected in December from the experimental field owned by Suez Canal University, Ismailia, Egypt. The fruits were washed and glochides and the two distal parts (top and bottom parts) were removed. After that, whole fruits were mashed in a mixer by interval mixing for 10 s. The whole mash was divided into four batches. The first part was not treated, but kept as control sample ('fresh juice'). The second part was subjected at ambient temperature to the non-thermal disintegration using PEF in a batch treatment chamber with two parallel stainless steel electrodes (distance 3 cm), applying 56 exponential decay pulses at maximum electric field strength of 3 kV/cm resulting in a total specific energy input of 5 kJ/kg fruit mash. The third part was subjected to thermal treatment using microwave heating (at 1800 W) up to 90 °C with a holding time of 3 min and subsequent cooling. The fourth part was macerated enzymatically using a commercial preparation of pectolytic enzymes, (Fructozym color, Erbslöh, Geisenheim, Germany). As described by the manufacturer, this liquid preparation is highly concentrated pectolytic enzyme preparation for making particularly color-intensive juices. The maceration of fruit mash was carried out using a preparation dosage of 150 µL/kg of fruit mash for a holding time of 90 min at 50 °C as recommended by the manufacturer. All mash batches were pressed using a manual laboratory juice press (Tinkturenpresse HP-2H, Fischer Maschinenfabrik, Neuss, Germany) through double layers of textile cloths as follows: 2 min pressing at 2.5 bar, 2 min at 5 bar, and further 6 min at 7.5 bar. The technological and rheological properties and vitamin C content were determined directly in the fresh juices. The rest of analyses were carried out in freeze-dried samples. Juice samples were frozen immediately after processing. In the fruit mash residues resulted from the pressing, seeds were removed and only yield of selected bioactive compounds (betalains and flavonols), total phenolic content, and antioxidant activity were evaluated in the freeze-dried material.

2.2. Chemicals and reagents

The flavonol standards were purchased from Extrasynthese (Genay, France). HPLC solvents and further chemicals were purchased from Carl Roth GmbH (Karlsruhe, Germany).

2.3. Determination of cell disintegration index (CDI) of fruit mashes

The impact of pre-treatments on the degree of cell disintegration (CDI) of fruit material has been determined according to Angersbach, Heinz, and Knorr (1999) compared to intact fruits and based on following equation: $\mathrm{CDI} = [(\sigma^{\mathrm{i}}_{\mathrm{h}}/\sigma^{s}_{\mathrm{h\ real}})\sigma^{s}_{1\ \mathrm{real}} - \sigma^{\mathrm{i}}_{1}]/[\sigma^{\mathrm{i}}_{\mathrm{h}} - \sigma^{\mathrm{i}}_{1}]$. In the present study, the CDI ranges from 0 (intact tissue) to 1 (complete cell rupture). Determination of CDI in the mash sample was carried out by measuring the conductivity of initial intact (σ^{1}_{h} and $\sigma^{\mathrm{i}}_{\mathrm{h}}$) and treated ($\sigma^{\mathrm{s}}_{1\mathrm{real}}$ and $\sigma^{\mathrm{s}}_{\mathrm{h\ real}}$) sample at low and high frequencies (range of 5.5 kHz–5.6 MHz) using impedance measurement equipment (Biotronix GmbH, Hennigsdorf, Germany). The measuring cell consisted of two cylindrical stainless steel electrodes (diameter 1 cm) separated to a distance of 1 cm by a polyethylene test tube. For measuring, a cylinder of cactus fruit tissue was placed into this adjusted gap (1 cm) of the polyethylene test tube between the two electrodes.

2.4. Juice yield, technological properties and vitamin C content

Yields of juices produced from fruit mashes were calculated according to the following equation: Juice yield (%) = [juice weight/fruit mash weight] × 100. The pH and total soluble solids TSS was determined directly from the pressed juices. Color parameters were measured according to Moussa-Ayoub, El-Samahy, Rohn, et al. (2011) using a Minolta Chroma Meter CR- 300 (Tokyo,