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Retention of polyphenols in encapsulated sour cherry juice in dependence of drying temperature and wall material

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ABSTRACT

Present study reports the effect of temperature, type of wall material and its ratio to the juice dry matter on the retention of phenolic acids, anthocyanins and flavonol glycosides in sour cherry Marasca juice encapsulated by spray drying. Individual polyphenols were determined and quantified by HPLC and response surface methodology approach was employed in order to observe the differences between different classes of sour cherry polyphenols regarding the conditions applied for encapsulation process. Maltodextrin 13-17 DE used in ratio 3:1 showed to be the optimal wall material for encapsulation of sour cherry juice at temperature of 200 °C, with the highest retention of phenolic acids and anthocyanins, namely 93.31 and 88.68%, respectively. Adversely, flavonol glycosides were retained to the most (84.01%) in juice encapsulated at 180 °C with gum arabic added in ratio 2:1.

1. Introduction

Sour cherry Marasca (*Prunus cerasus* var. Marasca) is autochthonous Croatian cultivar specific for its small fruit, harmonic taste coming from well-balanced sugar to acid ratio, intense aroma, dark red color and high dry matter content up to 27 g/100 g (Pedisić, Levaj, Dragović-Uzelac, & Kos, 2007). Compared to other sour cherry cultivars, Marasca has high biological activity because of its high content of polyphenols, especially anthocyanins. According to Elez Garofulić et al. (2015) total anthocyanin content in Marasca was 1705 mg/kg of fresh fruit, reaching even to 1730 mg/kg in an earlier study (Elez Garofulić, Dragović-Uzelac, Režek Jambrak, & Jukić, 2013). Kim, Heo, Kim, Yang, and Lee (2005) reported the anthocyanin content of 450 mg/kg f.w. for Balaton cultivar, 650 mg/kg f.w. for Danube and 720 mg/kg f.w. for Šumadinka, while Dragović-Uzelac et al. (2007) determined 545 mg/kg f.w. of total anthocyanins in Cigančica cultivar. All anthocyanins present in Marasca are cyanidin derivates differing in sugar moiety bonded to the aglyconic part of molecule, with cyanidin-3-glucosilrutinoside being present in the highest concentration (Elez Garofulić et al., 2013). Apart from anthocyanins, Marasca cherries contain a significant amount of other colorless polyphenols, especially hydroxycinnamic phenolic acids such as neochlorogenic, chlorogenic, *p*-coumaric and caffeic acid and flavonol glycosides presented by quercetin and kaempherol derivates (Kirakosyan, Seymour, Llanes, Kaufman, & Bolling, 2009; Zorić et al., 2017).

Because of its extraordinary characteristics, sour cherry Marasca is a great material for processing into a variety of food products, especially for functional food products as polyphenols present in its fruit possess strong antioxidant activity and may act preventive in case of different chronic diseases (Zorić, Dragović-Uzelac, Pedisić, Kurtanjek, & Elez Garofulić, 2014). However, polyphenols, especially anthocyanins are very susceptible to degradation during processing due to the prolonged oxygen and elevated temperature exposure (Rodriguez-Saona, Giusti, & Wrolstad, 1999). Therefore, novel techniques and approaches are necessary for production of functional products with maximal preservation of polyphenolic compounds.

Encapsulation techniques are widely studied to protect the polyphenols as the core material, from adverse environmental conditions, such as undesirable effects of light, moisture and oxygen, in order to deliver them to the consumer without destruction (Shahidi & Han, 1993) Thereby, encapsulation contributes to extending the product's shelf life, protecting the active components against degradation during storage and maintaining their functionality; masking unwanted flavor, smell, or taste (Celli, Ghanem, & Brooks, 2015; Luca, Cilek, Hasirci, Sahin, & Sumnu, 2014); and increasing the effectiveness of natural functional compounds that normally have a lower potency at equivalent levels when compared to synthetic ingredients (Yallapu, Gupta, Jaggi, & Chauhan, 2010).

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Common wall materials used for juice encapsulation by spray drying are maltodextrins and gum arabic (Murugesan & Orsat, 2011). The proper selection of wall material is crucial for the stability of encapsulated product and the degree of protection of active substances (Moser et al., 2017) which are both strongly dependent on the structure and physico-chemical properties of wall and core material. Spray drying is the technique most commonly used for the encapsulation of bioactive ingredients in food industry, due to its cost-effectiveness and adequacy for production of stable and functional products (Murugesan & Orsat, 2011; Robert et al., 2010).

In order to make the most of the encapsulation efficiency and achieve the maximal retention of polyphenols, the optimal drying conditions should be used. Therefore, for encapsulation of sour cherry Marasca juice, a detailed and careful selection of both processing conditions and wall material is necessary in order to produce a product of high nutritive and functional value with the highest retention of polyphenols. Although literature reports on the polyphenolic content and stability during the spray drying, there is no data for retention of individual compounds and different classes of polyphenols such as anthocyanins, phenolic acids and flavonol glycosides. These compounds differ in structure, properties and therefore exhibit different behavior during the spray drying encapsulation process.

The aim of this research was to observe the influence of drying temperature and different wall materials (two maltodextrins differing in dextrose equivalent and gum arabic) added to sour cherry Marasca juice in different ratios on the retention of individual and total anthocyanins, phenolic acids and flavonol glycosides during the spray dying encapsulation process. The response surface methodology approach was employed for determination of optimal drying conditions for retention of all observed polyphenolic classes individually, in order to observe the differences in their behavior during the process.

2. Materials and methods

2.1. Material

Sour cherry juice (15 °Brix) used for experiment was obtained from Dona Ltd. company (Gornja Stubica, Croatia). Industrial juice production was described previously by Elez Garofulić, Zorić, Pedisić, and Dragović-Uzelac (2016). Juice was stored in dark glass bottles at 4 °C prior to encapsulation. Three different wall materials were used for production of encapsulated juice, namely, maltodextrin with dextrose equivalent 4–7 (MD 4–7 DE), maltodextrin with dextrose equivalent 13–17 (MD 13–17 DE) and gum arabic (GA). All wall materials were obtained from Sigma Aldrich.

2.2. Chemicals and standards

Methanol, formic acid and acetonitrile used for the extraction and chromatographic analysis were HPLC grade, purchased from Prolabo, UK.

Standards of phenolic acids (caffeic, chlorogenic, *p*-coumaric acid and ferulic acid) were purchased from Sigma (Steinheim, Germany) as well as cyanidin-3-glucoside chloride and quercetin-3-glucoside, while cyanidin-3-sophoroside, cyanidin-3- rutinoside and kaempferol-3-rutinoside were obtained from Extrasynthese, Lyon, France.

2.3. Spray drying process

Encapsulated sour cherry Marasca juice was produced on a laboratory scale spray dryer SD 06 (Labplant, Great Britain). During the process the following parameters were kept at constant level: air flow 3.5 m/s, feed flow 485 mL/h and de-blocking speed at medium level. Spray drying encapsulation was carried out according to the experimental design shown in Table 1.

Three different wall materials (MD 4–7 DE, MD 13–17 DE and GA) were added to the 100 mL of sour cherry Marasca juice (15 °Brix) in three different wall: juice dry matter ratios (1:1, 2:1 and 3:1). The slurry was stirred and pre-heated to 50 °C on a magnetic stirrer (HSC Ceramic Hot Top-Plate Stirrer, Velp, Italy) for 10 min in order to achieve homogeneous dispersion of wall material in juice. While constantly being stirred, the slurries were spray dried at three different inlet temperatures, 150, 175 and 200 °C. The responding outlet temperatures were 78–80 °C, 87–90 °C and 99–102 °C, respectively. All powders were produced in duplicate and stored in dark plastic containers in desiccator at 20 °C for 7 days until analyzed.

2.4. Analytical methods

2.4.1. Extraction of polyphenols from encapsulated sour cherry Marasca juice

Polyphenols were extracted from 1 ± 0.001 g of encapsulated juice with 3 mL of 10 mL/L formic acid in 800 mL/L aqueous methanol solution. The mixture was stirred, extracted for 15 min in ultrasonic bath preheated to 50 °C, filtered through Whatman No. 40 filter paper (Whatman International Ltd., Kent, UK) and made up to 5 mL in a volumetric flask with extraction solvent. All extracts were prepared in duplicate and were kept in an inert nitrogen gas atmosphere, deep freezed at −80 °C (ScanCool SCL210P, LaboGene™, Lynge, Denmark) until used for HPLC analysis.

The same procedure was applied for extraction of polyphenols from fresh juice used for encapsulation.

2.4.2. HPLC analysis

Separation of polyphenols was performed by HPLC analysis, using the HPLC Agilent 1260 quaternary LC Infinity system (Agilent Technologies, Santa Clara, CA, USA) equipped with diode array detector (DAD), an automatic injector and ChemStation software on a Nucleosil 100-5C18, 5 μm (250 × 4.6 mm i.d.) column (Macherey-Nagel). The solvent composition and used gradient conditions were described previously by Elez Garofulić et al. (2015). Identification of phenols was carried out by comparing retention times and spectral data with those of the authentic standards (anthocyanins were identified at 520 nm, flavonol glycoside at 360 nm and phenolic acids at 280 nm).

The quantifications of anthocyanins, flavonol glycosides and phenolic acids were performed by the external standard method using the calibration curves of the standards. For compounds lacking reference standards quantifications were done as follows: cyanidin-3-glucosylrutinoside was determined according to cyanidin-3-glucoside, kaempferol-3-glucoside according to kaempferol-3-rutinoside and neochlorogenic acid according to chlorogenic acid calibration curve. Obtained concentrations were expressed as mg per 100 g of juice dry matter, as mean value ± standard deviation (N = 4 replicates).

2.5. Experimental design and statistical analysis

The experimental design and statistical analysis were done using Statsoft STATISTICA v. 10 Experimental design software (Statsoft Inc., Tulsa, OK, USA). A full factorial design comprising 9 experimental trials for each wall material used was chosen to evaluate the combined effect of two factors, wall: juice ratio and drying temperature, termed as X_1 and X_2, respectively (Table 1), giving in total 27 experimental runs. Experiments were performed in duplicate, in order of lowest wall: juice ratio and lowest temperature. The