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Different compounds are extracted with different time courses from fruits during microwave hydrodiffusion: Examples and possible causes

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ABSTRACT

We set out to determine how nutrients diffuse during extraction, using fractional collection. The highest concentrations of sugars (195.5, 64.8 and 60.8 g/L, respectively for grape, 'Najbolia' plum and apricot) were found for the earliest stages of extraction, with a decrease in concentration (to 41.4 g/L, 48.2 g/L and 1.7 g/L, respectively) at the end of extraction process. Total polyphenols showed the same trends for plum and apricot (from 4.1 g/L to 2.9 g/L for 'Najbolia' plum, from 2.2 to 0.2 g/L for apricot) but highest concentrations of total polyphenols (for grape and cherry) were obtained at fraction 5 or 6 (out of 7). Carotenoids from cherry tomato also had highest concentrations (at circa 25 mg/L) almost at the end of extraction. For volatile molecules from sweet cherry, hexanal, 2-hexenal and linalool had their highest concentrations at fractions 3–4 (out of 7).

Diffusion of nutrients depended on fruit destructuring, molecule solubility and localization of the compounds. Fruit size seemed unimportant.

1. Introduction

Extraction, notably from fruits for production of juices or specific ingredients, is a highly studies subject. Several key aspects of fruit extraction have been examined, including the nature of the fruit and the location of the components to be extracted with respect to tissue structures. Numerous pre-treatments of the tissue prior to extraction have been proposed to increase yields or rates of extraction: effects of cell-wall degrading enzymes (Ribeiro, Enrique, Oliveira, Macedo, & Fleuri, 2010), ultrasound (Vilkhu, Mawson, Simons, & Bates, 2008), microwave (Gerard & Roberts, 2004), pulsed electric fields (Turk, Vorobiev, & Baron, 2012), etc. However, these studies at best compare initial fruit and global juice composition. The extraction is treated as a single step. To our knowledge, no studies have described modifications of juice composition and/or different time courses during the fruit extraction. There is a "black box" about the diffusion of different molecules during extraction. While studying microwave hydrodiffusion (MWH) as a means of juice production from fruits (Cendres, Chemat, Maingonnat, & Renard, 2011), we noticed that juice colour and viscosity varied markedly during the treatment. This finding prompted us to investigate the variation in juice composition.

Microwave hydrodiffusion (MWH) can be used for the extraction of juice from fruits or for the production of various extracts (Orio et al., 2012). Microwave heating and hydrodiffusion allow rapid extraction of juice from fresh or frozen fruits. Using frozen fruit gives better yields (on average + 30%) due to dual destructuring (through both freeze–thaw and MW heating) (Cendres et al., 2011, 2012). From these experiments, the process of extraction from frozen fruit can be described independently of the plant matrix. Ice occupies more space than liquid water and water transfers between intra- and extracellular compartments occur during ice crystal formation. Consequently, freezing and thawing cause loss of structure and turgor, and disruption of membranes (Petzold & Aguilera, 2009). Some water in the fruit is in liquid form (bound water or localized thawing during transport to MW oven). This water absorbs microwaves and forms hot spots. A temperature rise then spreads out from these hot spots, causing the ice to melt. Release of the first drop during microwave hydrodiffusion is the result of the melting of ice crystals. More and more water molecules become susceptible to microwave irradiation. The average temperature increases to approximately 100 °C, with a change in the mechanism of extraction. In this second step, the extraction of juice proceeds by the generation of steam as in fresh fruits. This step corresponds to further destructuring of the plant matrix. It is during this stage that the maximum flow rate of juice is reached.

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An added advantage of this process is that the freshly extracted juice has very low contaminant levels, probably owing to (i) limited contacts with the outer surface of the fruit and (ii) elevated temperatures. This extraction method preserves the colour and natural taste of fruits.

Thus, to investigate the different step of extraction, and try to understand how the extraction of the different molecules proceeds, we decided to use MWH. This method had two advantages for our investigations: (i) the extraction can be described independently of the matrix, and (ii) we could collect samples for analysis at different times along the extraction. The extraction of juice during MWH is visibly inhomogeneous and probably involves different fruit constituents during the extraction, depending on the specific characteristics of the molecules such as localization and solubility. Qualitative and quantitative data are required to study and understand the difficulties met in the extraction of the different molecules or families of molecules. To study this and assess impact and factors of variability, we carried out fractional collection of juices in a MWH experiment at different power density settings. We linked the time courses of different components to their physico-chemical properties and their distribution in fruit.

We studied major compounds of fruits with sugars and acids, using different fruits (plum, grape, sweet cherry, apricot and tomato) in order to gather data on different size and nature of fruits. These compounds are highly water-soluble and distributed throughout the fruit. Concerning microconstituents, polyphenols and specifically anthocyans were the class of molecules for which we noticed the existence of differences along the extraction (Cendres et al., 2011). Therefore polyphenols, with lower, variable solubilities, were studied in different fruits that presented different ratios of concentrations notably of highly visible anthocyans in skin and flesh (grape and plum cv. 'President' et 'Najbolia' with red skin and yellow flesh, sweet cherry (Burlat) with red skin and red flesh). For carotenoids, which are hydrophobic, tomato was chosen due to the high concentrations of lycopene. Finally, volatile compounds were analysed for cherry as its aromas are known and have been noticed to be very different from flesh or stone.

2. Materials and methods

2.1. Chemicals and standards

Acetonitrile, methanol, (chromatographic quality) and hexane were from Fisher Scientific (Illkirch, France), toluene-α-thiol was from Merck™ (Darmstadt, Germany). Formic acid of purity 98–100% was from Sigma–Aldrich (Stenheim, Germany). Dichloromethane (for HPLC) was from Carlo Erba (France).

Enzymatic kits to measure D-glucose/fructose ref.: E 0139106, D-glucose ref.: E 0716251, L-malic acid ref.: E 0139 068 and citric acid ref.: E 0139076 were from R-Biopharm (Darmstadt, Germany), and tartaric acid ref. 207.18.233 was from VWR (Fontenay-sous-Bois, France).

All phenolic standards (*p*-coumaric acid, rutin, quercitrin, isoquercitrin, hyperoside, cyanidin-3-glucoside, cyanidin-3-rutinoside and peonidin) were from Extrasynthèse (Lyon, France), except for chlorogenic acid, (+)-catechin and (−)-epicatechin, which were from Sigma–Aldrich (Stenheim, Germany). Lycopene was from Extrasynthèse (Lyon, France), β-carotene and apocarotenal were from Sigma–Aldrich (Stenheim, Germany). Benzaldehyde, 2-hexenal and linalol were from Merck™ (Darmstadt, Germany).

2.2. Plant material

Fruits free of visible defects were chosen randomly, washed, dried using paper, weighed and frozen.

Plums (*Prunus domestica* L., cv. 'President' and 'Najbolia'), grapes (*Vitis vinifera* L., cv. Muscat), and apricots (*Prunus armeniaca* L., A3844) were obtained as described in Cendres et al. (2011). Cherry tomato samples (*Lycopersicon esculentum* L., cv. sweet 100 F1) were obtained from a local wholesaler from Avignon in August 2009. Sweet cherries (*Prunus avium* L.var. Burlat) were obtained from a private garden and harvested at maturity.

All fruits were portioned in batches of 500 g (except for apricot 250 g), frozen and stored at −18 °C.

2.3. Microwave juice extraction and procedure

Material and procedure is described in detail in Cendres et al. (2011). Microwave extraction of fruit juice (Fig. 1) was performed in a Milestone "DryDist" microwave laboratory oven (Milestone, Bergamo, Lombardy, Italy). In a typical procedure performed at atmospheric pressure, 500 g of frozen fruits (250 g for apricots) were MW heated at power density 1 W g^{-1}. A mixture of hot "crude juice" (*in situ* water) and steam exited the microwave cavity (through an opening underneath, due to gravity and pressure created by steam). The juice ran out and the steam was condensed in a condenser. Fractions of the fruit juice were collected (40 mL per fraction, 20 mL for apricot) in 50 mL plastic tubes (Falcon, Becton Dickinson, Franklin Lakes, New Jersey, USA). The extraction was continued until no more fruit juice was obtained or overheating was detected. At the end of the process, all the fractions were individually frozen, stored at −18 °C and analyzed for sugars, acids, polyphenols, carotenoids and/or volatile compounds depending on the fruit species.

For cherries, juice (called steamer juice) was also produced by using steam-cooker (Seb, France).

2.4. Colorimetric and enzymatic methods

The concentration of total phenolics was measured by the colorimetric method of Folin–Ciocalteu (Singleton & Rossi, 1965). The absorbance versus prepared blank was read at 730 nm in a Varian UV–Visible spectrometer (Varian, Palo Alto, USA). Total phenolics contents of juice were calculated against a calibration curve with chlorogenic acid.

Total anthocyans were determined by the pH differential method of Wrolstad (1982), with the formula:

$$\mathrm{Conc(g/L)} = \mathrm{Mw}\ \varepsilon\ (A_{510\ nm} - A_{700\ nm})_{pH1} - (A_{510\ nm} - A_{700\ nm})_{pH7}$$

The absorbances were read at 510 and 700 nm in a Varian UV–Visible spectrophotometer (Varian, Palo Alto, USA). The concentrations were expressed in cyanidin-3-rutinoside (plums cv. Najbolia and President) or cyanidin-*O*-glucoside (apricot, grape, cherry) equivalent with ε = 28,800 L/mol/cm and molecular weight 595 Da for plums, and ε = 26,900 L/mol/cm and molecular weight 449 Da, respectively for apricot, grape and cherry.

Malic acid, tartaric acid, sucrose, glucose and fructose were analyzed with colorimetric enzymatic tests using kits by an automatic analyzer (Hitachi, Maidenhead, United Kingdom). These results are expressed as sum of sugars, i.e. the sum of fructose, glucose and sucrose, and sum of acids, i.e. the sum of malic acid, citric acid and tartaric acid (the last exclusively for grape).

2.5. Determination of specific polyphenols by HPLC

Polyphenol analysis of plum and cherry was carried out on freeze-dried fruit and juice material. Thioacidolysis was performed as described by Guyot, Marnet, Sanoner, and Drilleau (2001). The HPLC apparatus was a Shimadzu LC-20AD equipped with an SPD-M20A DAD detector (Shimadzu, Kyoto, Japan). The column was a