



Concentration of natural aroma compounds from fruit juice hydrolates by pervaporation in laboratory and semi-technical scale. Part 1. Base study



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ABSTRACT

The first article of a two-article series presents pervaporation (PV) of volatile aroma compounds from fruit juice hydrolates (plum, apple, blackcurrant and cherry). The purpose of this research was to evaluate the effectiveness of fruit juice hydrolates separation on a laboratory scale (inert gas flow pervaporation system) and semi-technical (vacuum pervaporation system) by means of pervaporation. To create precise matrices for hydrolates before and after pervaporation for each of the separated systems, solid phase microextraction (SPME) technique and the gas chromatography-mass spectrometry (GC-MS) was applied. Sensory analysis confirmed improvement of aroma note of concentrated permeates as compared to feed hydrolates solutions. The results indicated that pervaporation may be applied in condensing aromatic water or fruit juice hydrolate, which may significantly enhance product quality and lengthen shelf life.

1. Introduction

Growing demand for technological innovation and high competitiveness make manufacturers of food flavors, juices and natural aromas constantly improve the quality of their produce and lower the prices (Álvarez et al., 2000; Mitkowski et al., 2009; Mitkowski, 2011; Saffarionpour and Ottens, 2017; She & Hwang, 2006). Recently more attention is paid to the origin of e.g. aroma substances used in foodstuffs, cosmetics and pharmaceuticals. With increasing consumer demand for health and wellness products as well as penetration of organic foodstuffs, natural flavors have attracted attention of food manufacturers. Flavors are segmented as natural, natural identical and artificial flavorings (Branen, Davidson, Salminen, & Thorngate, 2002; Erten and Cadwallader, 2017). A natural flavorings may be even 50 more expensive than its synthetic equivalent (Lipnizki, Olsson, & Trägårdh, 2002). From 2015 to 2020 a continuous growth rate of 5% is expected to food flavor market (Marketsandmarkets.com, 2015). The value of this market is projected to reach 15.1 billion USD by 2020 (Marketsandmarkets.com, 2015).

Fruits flavors are mixtures of hundreds of various organic compounds that are present in fruits at very low concentrations. Not all of them exude fragrance. Esters that are predominantly responsible for fragrance of a particular plant raw material may make up even 95% of compounds (López et al., 2007). Aromatic compounds include also alcohols, ketones, aldehydes and terpenes. Each group exudes a different

fragrance tone of the fruit. Esters are usually responsible for sweet and fruitlike odours, aldehydes for fresh and grass-like, alcohols –for the bitter odour (Branen et al., 2002; Saffarionpour and Ottens, 2017). The concentrations of particular substances differ greatly and their influence on juice fragrance is not commensurate with their amount. Though the whole array of scents contributes to the overall fragrance of juice or fruit, yet specific compounds are behind a particular character. These aromas are obtainable during the production of fruit juices. Flavor-active compounds occur in concentrations not larger than ppm levels, hence even a very small loss during concentration may negatively affect the taste and above all the fragrance of the final product (Olsson & Trägårdh, 1999; Saffarionpour and Ottens, 2017).

The fruit sector is key element of Europe Union agriculture. In 2016, the total fruit production in EU was 35.4 million tonnes. This sector offers a large number of different fruits, where apples (12.6 million tonnes), oranges (6.4 million tonnes) and peaches (2.7 million tonnes) are the most important in terms of the volume of harvested products (Eurostat, 2016). Poland ranks among the most important fruit growers-12.2% (Italy-23.8%, Spain-29.1%), and producers of juices in Europe, especially apples, cherries, plums and blackcurrants (Eurostat, 2016). A block scheme of the fruit juice manufacturing process is presented in Suppl. Fig. 1. The first production stage includes washing, grinding and pressing of the fruit. Volatile organic aroma compounds are recovered and concentrated usually by means of distillation or steam distillation and finally added to clarified juice (De Vasconcelos

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Facundo, De Souza Neto, Maia, Narain, & Dos Santos Garruti, 2010). Recovered aroma solution is known as fruit juice hydrolate or aromatic water. Hydrolates are highly diluted aqueous solutions, where the concentration of volatile aroma compounds, which are biologically active, does not exceed 1 wt% (Álvarez et al., 2000; Prusinowska, Śmigielski, Stobiecka, & Kunicka-Styczynska, 2016).

At present volatile aroma compounds are concentrated by distillation, steam distillation, partial condensation or extraction. However, thermal processes are energy and cost consuming. Additionally, at high temperatures there occur irreversible, undesirable losses or transformations of heat-sensitive aroma compounds and these, even if small, may negatively affect organoleptic properties of the ready product (Aghakarimiha, Raisi, & Pazuki, 2018; Pereira, Ribeiro, Nobrega, & Borges, 2006).

Pervaporation can become an attractive alternative during separation of thermolabile flavors from natural sources. Because of its ability to separate components under mild conditions (moderate temperature, no additional reagents needed) pervaporation may be regarded as a highly viable process, which might be a very good alternative to classical methods of aroma compounds separation (Baker, 2004; Kołtuniewicz and Drioli, 2008; Olsson and Trägårdh, 1999; Strathmann, 2011; Van der Bruggen and Luis, 2015). It may be applied for recovering aroma compounds from fruit juices (Börjesson, Karlsson, & Trägårdh, 1996; Figoli et al., 2006; Isci, Sahin, & Sumnu, 2006; She and Hwang, 2006), for concentrating aromatic water/hydrolate (Dawiec-Liśniewska et al., 2017), for the elaboration of full flavored low alcohol wines or beers (Paz et al., 2017; Salgado et al., 2017) as well as for separation of aroma compounds from e.g. coffee (Weschenfelder, Lantin, Viegas, Castilhos, & Scheer, 2015). Despite the numerous works on pervaporation of volatile aroma compounds from model aqueous solution (Aroujalian and Raisi, 2007; Dawiec, Witek-Krowiak, Podstawczyk, & Pokomeda, 2015; Garcia et al., 2008; Pereira et al., 2005; Rafia, Aroujalian, & Raisi, 2011; Raisi, Aroujalian, & Kaghazchi, 2008; Shepherd, Habert, & Borges, 2002), there are many unexplained aspects of the process, especially regarding separation of multicomponent and real systems.

Practical application of pervaporation is limited, mainly due to the fact that most research efforts on pervaporation have been focused on the separation of model aroma-water systems in a narrow range of volatile compounds compositions. Pervaporation of real systems has received comparatively less attention (Dawiec-Liśniewska et al., 2017; Paz et al., 2017). This work aims to fill the gap in pervaporation of industrial solutions by offering complete research on a laboratory and semi-technical scale.

Presented study gives important information about the concentration profile of apple, blackcurrant, cherry and plum hydrolates during pervaporation, and identifies the most important parameters influencing the flavor recovery efficiency. The SPME GC-MS technique was used for the creation of precise matrices for apple, blackcurrant and cherry hydrolates and hydrolate permeates, which make this work unique since the available studies concentrate mainly on the separation of model multicomponent aroma solutions. To the best of our knowledge, this is the first work that shows the application of the Box-Behnken design (BBD) for evaluation of the cooling temperature effect on pervaporation performance. Additionally, a comprehensive study on the semi-technical pervaporation of juice hydrolates presented in the first part of the series together with the detailed economic analysis shown in the second manuscript (Podstawczyk, Mitkowski, Dawiec-Liśniewska, & Witek-Krowiak, 2017) may constitute the base for the industrial scale pervaporation planning process and implementation.

This study focuses on the effectiveness of fruit juice hydrolates separation on a laboratory scale (inert gas flow PV system) and semi-technical (vacuum PV system) by means of pervaporation. The fruit juice hydrolates under study were manufactured in Poland. At the first stage the optimal process parameters (feed temperature, feed flow rate and cooling temperature) of plum hydrolate PV were determined

(STAGE I). Next apple, blackcurrant and cherry hydrolates were pervaporated on a laboratory scale. In order to assess the qualitative and quantitative contents of natural hydrolates before and after separation, gas chromatography with mass spectrometry and solid phase micro-extraction (SPME GC-MS) was performed (STAGE II). At the third stage apple, blackcurrant and cherry hydrolates were concentrated on a semi-technical scale. The effectiveness of pervaporation of natural aroma compounds was examined relative to process temperature (50 and 60 °C) (STAGE III). At the last stage sensory analysis was done to determine the organoleptic quality of the obtained permeates (STAGE IV).

2. Materials and methods

Pervaporation experimental set-up on a laboratory and semi-technical scale and its optimization with RSM method is presented in [Supplementary Material \(Suppl. Chapter 1\)](#).

2.1. Analytical methods

2.1.1. Gas chromatography

Gas chromatography was used to do quantitative analysis of aroma compounds in the feed and permeate solutions of plum hydrolates (STAGE I-RSM study) (GC Varian 3800 with CP-8400 Autosampler, SpectraLab Scientific Inc., Canada). GC was equipped with a FactorFour™ Capillary Column (30 m × 0,53 mm ID × 1,5 μm film thickness) and a flame ionization detector (FID). Helium was used as a carrier gas at a flow rate of 1 ml/min. The GC oven temperature was kept at 60 °C for 3 min and raised up to 120 °C for 3 °C/min then up to 300 °C for 15 °C/min and kept constant. Injector and detector temperatures were 200 and 300 °C, respectively. Split 1:20. Before GC analysis, 0.5 ml of analysed sample was mixed with essential oil (2-Udecanone) as an internal standard and then, the mixture was extracted from aqueous solutions with methylene chloride two times, in the ratio 1:1. Afterwards, the water was removed from the sample with a drying agent: MgSO₄ and one microliter of the prepared solution was injected to GC-FID. Mass fraction of aromas in the solution was determined on the basis of an internal standard content.

2.1.2. Solid phase micro extraction and gas chromatography with a mass spectrometer analysis (SPME GC-MS)

The chemical composition of volatiles presented in juice hydrolates: apple, blackcurrant, cherry, pervaporated on laboratory scale (STAGE II) as well as semi-technical scale in case of apple hydrolate (STAGE III), was identified using solid phase micro extraction and gas chromatography with a mass spectrometer SPME GC-MS method. The method was also described in [Dawiec-Liśniewska, Podstawczyk, and Witek-Krowiak \(2017\)](#). The solid phase micro extraction procedure was performed as follows: 1 ml of the liquid under investigation was mixed with internal standard (2-Udecanone) and the sample was introduced into a 20 ml glass vial. The SPME fiber assembly (StableFlex, Supelco, Sigma-Aldrich) coated with a mixture of Divinylbenzene/Carboxen/Polydimethylsiloxane (DVB/CAR/PDMS) was exposed for 20 min in the head-space at 40 ± 2 °C; afterwards, the fiber was withdrawn into the needle and transferred to the injector of the GC-MS, where the analytes were thermally desorbed from the fiber within 5 min.

A gas chromatograph (GC) with a mass spectrometer (MS), a Saturn 2000 MS Varian Chrompack with ZB-1 (Phenomenex) column (30 m × 0,25 μm film × 0,25 mm ID) was used. The MS was equipped with an ion-trap analyzer set at 1508 for all analyses with an electron multiplier voltage of 1350 V. Scanning (1 scan/s) was performed in the range of 39–400 *m/z*, using electron impact ionization at 70 eV. The analyses were carried out with helium as a carrier gas at a flow rate of 1.0 ml/min in a split ratio of 1:20 and the following program: 60 °C at the beginning and hold 3 min; 3 °C/min to 120 °C; 15 °C/min to 300 °C. The injector and detector were held at 200 and 300 °C respectively. Most of the compounds were identified by means of 3-analytical

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