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Comparison of stir bar sorptive extraction in the liquid and vapour phases, solvent-assisted flavour evaporation and headspace solid-phase microextraction for the (non)-targeted analysis of volatiles in fruit juice



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n-hexyl butanoate (PubChem CID: 17525)

decanal (PubChem CID: 8175)

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ABSTRACT

The volatile fraction plays an important role on the organoleptic properties and overall acceptability of fruit juices. This work reports for first time a non-targeted approach for the analysis of the volatile fraction of fruit juice by stir bar sorptive extraction (SBSE¹) in liquid and vapour phase (HSSE²) at three extraction times (30, 60, and 120 min), two temperatures (room temperature and 40 °C), and two sample volumes (5 and 10 mL). The resultant volatile profiles were compared with solvent-assisted flavour evaporation (SAFE³) and headspace solid-phase microextraction (HS-SPME⁴). SBSE and HSSE enabled the detection and identification of more compounds than HS-SPME and less than SAFE. Compared with classic extraction methods, SBSE did not use organic solvents, was easier to perform, required less than two hours and exhibited the highest reproducibility. SBSE allowed the semi-quantification of esters, ketones, terpenes, alcohols known as key aroma compounds in juice. Moreover it was the only method resulting in the identification of *n*-dodecanoic, *n*-tetradecanoic, *n*-pentadecanoic and *n*-hexadecanoic acids and the best one to recover other compounds of particular interest in fruit juices such as nootkatone.

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1. Introduction

Fruit juices are considered beneficial for health and well-being due to their high content in polyphenols and vitamins. They are also appreciated by consumers for their organoleptic properties. However, due to their high free sugar content they are also linked to some health diseases such as obesity, diabetes and caries (World

Health Organization, 2015). The flavour of fruit juice relies on a subtle balance among sweetness, sourness, bitterness and aroma. The volatile fraction responsible for aroma plays an important part in overall acceptability, and thus it is important to identify and quantify volatile compounds that may impact aroma perception (Rouseff, Ruiz Perez-Cacho, & Jabalpurwala, 2009).

The selection of a suitable extraction methodology is a key step in assessing an accurate volatile composition through gas chromatography mass spectrometry (GC-MS⁵) and an odour evaluation through gas chromatography olfactometry (GC-O⁶) (Caven-

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¹ SBSE: Stir Bar Sorptive Extraction.

² HSSE: HeadSpace Stir Bar Sorptive Extraction.

³ SAFE: Solvent-Assisted Flavour Evaporation.

⁴ HS-SPME: HeadSpace Solid-Phase Microextraction.

⁵ GC-MS: Gas Chromatography Mass Spectrometry.

⁶ GC-O: Gas Chromatography Olfactometry.

Quantrill & Buglass, 2006; Marsili, 2001; Thomsen, Gourrat, Thomas-Danguin, & Guichard, 2014; Yan, Wenlai, & Qian, 2007; Yan et al., 2007). Despite the wide variety of extraction (and concentration) methodologies available, there is no universal extraction method to produce a representative or rich extract. Moreover, an efficient and powerful analytical technique cannot compensate for errors of sensitivity, specificity, and selectivity during the extraction step. Analytical techniques are easier optimized in a target approach where an optimization can be done for a specific quantitative expression, for example the recovery of a specific volatile compound. However, for the non-targeted approach, the goal is mainly to detect as many volatile compounds as possible present in a particular extract, but the optimization of all compound simultaneously is a challenge (Grauwet, Vervoort, Colle, Van Loey, & Hendrickx, 2014).

The extraction methodologies usually applied in analysing volatile compounds in foodstuffs include steam distillation, extraction with classic solvents, supercritical fluids and pressurized fluids, simultaneous distillation-extraction, headspace techniques, and solid-phase extraction techniques, among others (Marsili, 2001). The specific usefulness of stir bar sorptive extraction (SBSE) in non-targeted analyses of volatile compounds in juices has been only reported in grape juice (Camino-Sanchez, Rodriguez-Gomez, Zafra-Gomez, Santos-Fandila, & Vilchez, 2014; Caven-Quantrill & Buglass, 2006) and never in comparison to contemporary extraction techniques, such as headspace solid-phase microextraction (HS-SPME) (Pawliszyn, 1995) and solvent-assisted flavour evaporation (SAFE) (Engel, Bahr, & Schieberle, 1999).

SBSE consists of a magnetic sealed inside a glass tube and is coated with a thick layer of sorptive material. It has two sampling modes, liquid (SBSE) and vapour phase (HSSE) and allows rapid sample preparation before concentration of volatile compounds on the stir bar during stirring in a liquid sample (i.e., fruit juices). Subsequently, it is placed in an empty thermal desorption glass tube followed by thermal desorption, in which analytes are thermally released and transferred to the gas chromatograph - mass spectrometer (GC-MS) system (Baltussen, David, Sandra, Janssen, & Cramers, 1999). In some specific applications, researchers have reported higher recoveries than with SPME because of the higher amount of sorptive material applied (Bicchi, Iori, Rubiolo, & Sandra, 2002), more reliable quantitative results because of the higher reproducibility of the methodology (Ruan, Aalhus, Juarez, & Sabik, 2015), and the potential to identify more compounds than by, for example, micro-scale simultaneous distillation-extraction due to the higher sensitivity (enrichment) of the technique (Caven-Quantrill & Buglass, 2006). SAFE allows the extraction of volatile compounds and has been widely used to isolate odorant compounds in complex matrices (Engel et al., 1999). However, SAFE requires a larger amount of sample compared to SBSE, and the extraction procedure is longer and completely manual, with a consequent loss of reproducibility. SPME permits solvent-free sample preparation and does not require any complex equipment. A lower adsorbent material can be used, with consequently lower recoveries and sensitivity compared to SBSE (Bicchi et al., 2002; Demyttenaere, Martinez, Verhe, Sandra, & De Kimpe, 2003). The maximum polydimethylsiloxane (PDMS⁷) volume coated onto the fiber is 0.6 μL (100- μm fiber) in SPME but 125 μL in SBSE (Bicchi et al., 2002). SPME is a completely automated procedure and is frequently used. However, SPME is limited by the small number of stationary phases commercially available as fiber coatings, the low volume coated on the fiber (Lopez, Huerga, Batlle, & Nerin, 2006) and also due to the phenomene of coating saturation and

competition between components of the stationary phase that may cause losses of analytical determination (Contini & Esti, 2006).

Volatile compounds present even at low concentrations (ppm or ppb) in fruit juice may have an important impact on overall aroma. The extraction methodology— isolation, concentration, and separation—is a complex process, particularly for low-concentration, highly labile, or reactive compounds, and must be carefully selected. A priori, the greater the number of compounds extracted, the higher the probability of extracting the compounds of interest. However, for volatile active compounds with a low odour threshold that are present in very low amounts, high selectivity and sensitivity (enrichment or focusing) are required. Semi-quantification is frequently used in non-targeted analyses to quickly ascertain the relative concentrations of volatile compounds between samples. Unfortunately, many studies do not include extraction method selection when reporting the analysis of a volatile profile or aroma evaluation; thus, several active compounds might be missed in the extract and, consequently, in the evaluation of key odorant molecules.

The literature contains only a few examples of the use of SBSE or HSSE for non-targeted volatile compounds analysis in real food extracts or beverage samples. These techniques have been used to analyse coffee (Bicchi et al., 2002), grilled lean beef (Ruan et al., 2015), grape juice (Caven-Quantrill & Buglass, 2006; Caven-Quantrill & Buglass, 2008), whisky (Demyttenaere et al., 2003), apple pomace (Rodriguez Madrera, & Suarez Valles, 2011), vinegar (Guerrero, Marin, Mejias, & Barroso, 2006; Marrufo-Curtido et al., 2012), brandy (Delgado, Duran, Castro, Natera, & Barroso, 2010), wine (Alves, Nascimento, & Nogueira, 2005; Weldegergis, Tredoux, & Crouch, 2007), and pesto Genovese (Salvadeo, Boggia, Evangelisti, & Zunin, 2007) without any comparison with other extraction techniques, particularly HS-SPME and SAFE. The use of SBSE in food applications has recently been reviewed (Kawaguchi, Nakazawa, & Takatsu, 2013). However, SBSE has been more frequently reported for trace analysis of targeted compounds in food samples, such as pesticides and additives, due to the effectiveness of SBSE for the extraction of non-polar compounds from liquid samples (Camino-Sanchez et al., 2014) and has been used for targeted analyses of the main constituents of food (Fang & Qian, 2006; Zalacain, Marin, Alonso, & Salinas, 2007; David & Sandra, 2007; Mun Wai et al. 2013; Ochiai, Sasamoto, & Kishimoto, 2015; Hjelmeland, Wylie, & Ebeler, 2016).

The aims of this work were *i*) to evaluate the influence of several parameters of SBSE for 10 targeted compounds important for their impact on aroma perception in fruit juices, such as sampling mode (liquid or vapour phase), extraction time, sample volume, and temperature; *ii*) to assess the effect of sampling mode (SBSE and HSSE) on the non-targeted reliable analysis of a wide range of compounds with different volatilities and polarities in fruit juice; and *iii*) to compare the non-targeted analysis results with those obtained by SPME and SAFE methods in terms of number of compounds identified and recovery obtained. The injection modes for gas chromatography differ among the extraction methods, with a thermal desorption and cooled injection system (TDU-CIS⁸) used for SBSE and HSSE and program temperature vaporizer (PTV⁹) for HS-SPME and SAFE, thus preventing direct comparison. However, this study provides valuable information for selecting an extraction method that is rapid and easy to perform and provides a non-targeted volatile profile in samples with high sugar content, such as fruit juice.

⁷ PDMS: Polydimethylsiloxane.

⁸ TDU-CIS: Thermal Desorption Unit - Cooled Injection System.

⁹ PTV: Program Temperature Vaporizer.

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