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Analytical Methods

Determination of low molecular weight volatiles in *Ficus carica* using HS-SPME and GC/FID

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ABSTRACT

Ficus carica L. is one of the earliest cultivated fruit trees, having an important consumption in Mediterranean countries. In this work, the volatile compound profiles of two characteristic Portuguese white varieties (“Pingo de Mel” and “Branca Tradicional”) was determined by HS-SPME and GC/FID. Leaves, pulps and peels, submitted to freezing and lyophilisation treatments, were analysed.

The two varieties presented a similar profile composed of eight volatile compounds: acetaldehyde, ethyl acetate, methanol, ethanol, hexanal, limonene, (*E*)-2-hexenal and octanal. The total volatile content was different among the vegetal materials, following the order leaves > peels > pulps. Methanol and ethanol are the major compounds in all samples.

The developed procedure revealed to be rapid, sensitive, reproducible and accurate. The detection limit values were low, and the method precise. The recovery values for acetaldehyde, ethyl acetate, methanol and ethanol were generally high, suggesting that it will be most suitable for compounds with low molecular weight. Due to its rapidity and low cost, this technique can be useful in the quality control of fig fruit and leaves.

1. Introduction

Fig (*Ficus carica* L.) is a tree belonging to the Moraceae family, one of the first plants cultivated by humans as a seasonal food. It is an important constituent of the Mediterranean diet (Solomon et al., 2006). Previous studies with the fruits indicated that they are a source of minerals, vitamins and dietary fibre; they are fat and cholesterol-free and contain a high number of amino acids (Solomon et al., 2006; Vinson, Zubik, Bose, Samman, & Proch, 2005). Figs. also present sugars and organic acids that influence their quality, and exhibit one of the highest concentrations of polyphenols among the commonly consumed fruits and beverages, which contribute positively to human health (Veberic, Colaric, & Stampar, 2008; Vinson et al., 2005; Oliveira et al., 2009).

The formation of volatile compounds in fruits is a dynamic process, and generally the typical flavour of most of them is not present at harvest but develops after the ripening process. Volatile compounds present in fresh and processed fruits significantly affect their flavour and aroma quality, which is formed by a complex group of chemical substances (Riu-Aumatell, Castellari, López-Tamames, Galassi, & Buxaderas, 2004). The variability in aroma compounds has been reported to depend on climatological conditions, cultivar, maturity and technological factors, like harvest, post-harvest treatments, processing and storage conditions (Douillard & Guichard, 1990; Rizzolo, Polesello, & Polesello, 1992; Botondi, De Santis, Bellicontro, Vizovitis, & Mencarelli, 2003; Lin, Rouseff, Barros, & Naim, 2002).

Gas chromatography (GC) methodologies have been described for the analysis of the volatile fraction of several matrices, using different techniques (Portari, Marchini, & Jordão, 2008; Ferrari, Arado, Nardo, & Giannuzzi, 2003). The determination of volatile compounds use headspace (HS) sampling coupled to GC as the preferred technique (Portari et al., 2008). The direct sample injection in packed columns (Ferrari et al., 2003) became obsolete and was gradually substituted by headspace techniques in capillary columns (Wasfi, Al-Awadhi, Al-Hatali, Al-Rayami, & Al-Katheeri, 2004) and, more recently, by the selective headspace injection using solid-phase microextraction (SPME) fibres (Riu-Aumatell et al., 2004; Dong, Mei, & Chen, 2006).

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