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Study of the volatile composition and sensory characteristics of new Sherry vinegar-derived products by maceration with fruits

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ABSTRACT

Several experiments of maceration of a Sherry wine vinegar with different fruits (orange, lemon, strawberry, grapefruit, and lime) have been carried out. After a preliminary study (only peel, no heating and 7 days as maximum time of maceration), parameters such as volatile compounds and sensory characteristics were determined in Sherry wine vinegars macerated with two amounts of peel (100 and 200 g/L) and for two maceration times (3 and 7 days). For volatile compounds, the statistical studies revealed that the factor "fruit" was the most significant with those vinegars obtained from lime, lemon and orange characterized by a high content in terpenic compounds. For those vinegars macerated with orange, high contents in several esters, alcohols, aldehydes and acetates were also observed. The vinegars macerated with strawberry showed very similar volatile profiles to that corresponding to the vinegar without maceration (control vinegar). In the case of the sensory study, the judges (from 12 to 15) preferred different amounts of fruit and maceration time for each fruit. For those vinegars macerated with orange and lime, which were the most preferred by the judges, the best qualifications corresponded to those macerated with the highest amount of fruit.

1. Introduction

Nowadays, the beverage market is turning toward diversification of traditional beverage categories, with different and improved sensory properties in relation to the conventional products. Moreover, the development of new products with functional properties is becoming an incentive to raise consumers' consciousness about their consumption. Some of these products include traditional drinks combined with fruit that improve their health-promoting characteristics, preventing different diseases such as cancer or obesity (González-Molina, Domínguez-Perles, Moreno, & García-Viguera, 2010; Tripoli, La Guardia, Giammanco, Di Majo, & Giammanco, 2007). On the basis of the development of new products with different organoleptic characteristics, volatile compounds play an important role. On the one hand, aroma is diversified by maceration with fruits, due to the addition of new volatile compounds derived from them and, on the other hand, it is improved by the enrichment of other volatile compounds already present in the traditional product. Both facts influence positively on the final quality of new products.

Volatile compounds are present to a large extent in vegetal products, such as fruits (Almenar, Hernández-Muñoz, & Gavara, 2009; Lan-Phi, Shimamura, Ukeda, & Sawamura, 2009; Moufida & Marzouk, 2003). Particularly, citrus fruits are a great source of volatile compounds, belonging to several families: alcohols, aldehydes, esters, acids, sesquiterpenes, monoterpene alcohols and monoterpene hydrocarbons. These are the main families of volatile compounds from *citrus* fruits (lemon, lime and orange) (Ferhat, Meklati, & Chemat, 2007), in which limonene is the most predominant monoterpene hydrocarbon (Lan-Phi et al., 2009; Moufida & Marzouk, 2003). Other important compounds present in citrus fruits are β-myrcene, γ-terpinene and α-pinene, identified by several authors in Spanish and Turkish oranges (Jordán, Goodner, & Laencina, 2003; Nisperos-Carriedo & Shaw, 1990; Selli, Cabaroglu, & Canbas, 2004), Japanese and Tunisian lemons (Lan-Phi et al., 2009; Moufida & Marzouk, 2003) and different varieties of limes (Ramesh Yadav, Chauhan, Rekha, Rao, & Ramteke, 2004). With regard to monoterpenes alcohols, α-terpineol and terpinen-4-ol, together with linalool, are the main compounds in *citrus* fruits (Moufida & Marzouk, 2003; Selli et al., 2004), as well as the sesquiterpene nootkatone in different varieties of grapefruit (Caccioni, Guizzardi, Biondi, Renda, & Ruberto, 1998).

The main sensory characteristic which maceration with different *citrus* fruits provides on the new products is the citrus-like

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aroma, for what monoterpene alcohols and hydrocarbons are responsible. By this way, α-terpineol, linalool and nerol indicated citrusy notes in at least four out of the six samples of yuzu (*Citrus junos*) peel oils (Lan-Phi et al., 2009), as well as β-myrcene and limonene in orange and lemon (Lan-Phi, Minh Tu, Nishiyama, & Sawamura, 2006; Selli et al., 2004). Moreover, nerol, geraniol and their respective acetates, α- and β-citronellol and carvone developed lime-like notes by means of GC–Olfactometry.

Regarding vinegar, new and improved products derived from it are now starting to be developed and studied. Asian countries have a consolidated industry in this type of products, whereas few researches have been carried out in the rest of the world. Physicochemical properties of different concentrated fruit vinegars have been studied by Chang, Lee, and Ou (2005), and subtropical fruit vinegars have also been analyzed by other authors (Ma et al., 2007; Su & Chien, 2007). By this way, Wu, Wang, Wang, and Jia (2007) developed a new-type of aromatic vinegar beverage using rice as main ferment material, mixing with several substances such as ginger and liquorice root. However, no scientific studies about new-type wine vinegars have been found in the literature; only wine vinegars macerated with different fruits have been recently studied by our research group, on the basis of their polyphenolic composition and antioxidant activity (Cejudo-Bastante et al., 2010). Further researches of these new-type wine vinegars are needed to improve and diversify the enological market.

The objective of this research is to study the volatile composition of Sherry wine vinegars macerated with several fruits and their sensory acceptability. In this way, new vinegar-derived products with optimal sensory characteristics have been developed.

2. Material and methods

2.1. Vinegar samples

Individual macerations with different fruits of a Sherry wine vinegar supplied by a local winery, were carried out. This vinegar was not aged and presented 7 acetic degrees and 1 alcoholic degree (an acetic or alcoholic degree express the amount in grams of acetic acid or ethanol respectively present in 100 mL of vinegar). All the fruits employed in these studies presented an optimal maturity and health stage: orange (*Citrus sinensis* and *Citrus aurantium*, from Valencia, Spain), lemon (*Citrus limon*, from Murcia, Spain), lime (*Citrus latifolia*, from Mexico), red grapefruit (*Citrus paradisi*, from Valencia, Spain) and strawberry (*Fragaria ananassa*, from Segovia, Spain). The Sherry wine vinegar without maceration was used as control vinegar. It was maintained under the same conditions as the macerated vinegars.

2.2. Preliminary study of maceration conditions

With the aim of establishing the best conditions of the maceration process, three experiences employed orange as representative fruit were carried out. Three different parameters (part of the fruit, maceration time and heating) were studied. For this objective, three dark glass containers were filled with 2 L of Sherry wine vinegar. Pieces of orange peel of 3 × 3 cm were added (200 g/L) (both peel and pulp to one container, about 61% of peel and 39% of pulp, and just only peel to other two containers) in order to determine the most appropriate part of the fruit. The maceration times considered were 7 and 14 days. A high temperature (40 °C) was only applied to the vinegar macerated with peel. The experiences with heating and pulp plus peel were discarded because authors thought that the joint use of both variables, high temperature and pulp, could produce different and numerous reactions which could deteriorate vinegar's sensorial profile. All the containers were continuously stirred at 300 rpm at room temperature, and every 12 h they were completely mixed to facilitate a closer contact of the upper material with the vinegar. Each experience was developed in duplicate.

2.3. Maceration with different fruits

A second scheme of maceration conditions with different fruits and without temperature was carried out. The fruits selected were orange, lemon, lime, grapefruit and strawberry. 3 and 7 days as maceration times and 100 g/L and 200 g/L as amount of peel were studied. In the case of strawberry, 100 g/L and 200 g/L of the entire fruit were employed. All the experiences were also carried out in duplicate.

2.4. Analysis of volatile compounds

2.4.1. Chemicals and reagents

All the aroma standards employed in this work were supplied by Merck (Darmstadt, Germany) and Sigma (Steinheim, Germany). 4-methyl-2-pentanol was used as internal standard. NaCl were purchased from Scharlau (Barcelona, Spain).

2.4.2. Sample preparation

10 mm × 0.5 mm (length × film thickness) PDMS commercial stir bars, supplied by Gerstel (Mülheim a/d Ruhr, Germany) were used for the extractions. For each SBSE (Stir Bar Sorptive Extraction) analysis, a volume of 25 mL of sample was pipetted and placed into a 100-mL Erlenmeyer flask with 5.85 g of NaCl and 50 μl of a solution of 4-methyl-2-pentanol (22.22 mol/m^3 in Milli-Q water containing 1332.22 mol/m^3 of acetic acid), according to the method proposed by Durán Guerrero, Natera Marín, Castro Mejías, and García Barroso (2006).

The Erlenmeyer flask was placed on a 15 position magnetic stirrer (Mülheim a/d Ruhr, Germany). Later, the stir bar was stirred at 1250 rpm at 25 °C for 120 min. After removal from the vinegar sample, and in order to remove NaCl, the stir bar was placed for a few seconds in distilled water and gently dried with a lint-free tissue. Then, it was transferred into a glass thermal desorption tube and then thermal desorption was carried out.

2.4.3. Instrumentation

A commercial TDS-2 thermal desorption unit (Gerstel) connected to a programmed-temperature vaporisation (PTV) injector CIS-4 (Gerstel) by a heated transfer line were used for the thermal desorption of the coated stir bars. The PTV was installed in an Agilent 6890 GC-5973 MS system (Agilent Technologies, Palo Alto, CA, USA). An empty baffled liner was used in the PTV. The thermodesorption unit was equipped with an MPS 2L autosampler (Gerstel) capable of handling the program for 98 coated stir bars. The desorption temperature was programmed from 40 °C to 300 °C (held for 10 min) at 60 °C min^{-1} under a helium flow (75 mL/min) and the desorbed analytes were cryofocused in the PTV system with liquid nitrogen at −140 °C. Finally, the PTV system was programmed from −140 °C to 300 °C (held for 5 min) at 10 °C s^{-1} for analysis by GC–MS. An Agilent 6890 GC-5973N MS system (Agilent, Little Falls, DE, USA), equipped with a DB-Wax capillary column (J&W Scientific, Folsom, CA, USA), 60 m × 0.25 mm I.D., with a 0.25 μm coating were used in order to perform the capillary GC–MS analysis in the electron impact mode. The carrier gas was helium at a flow rate of 1.0 mL/min.

Peak identification was carried out using the Wiley 7N Edition Library (Wiley Registry of Mass Spectral Data, 7th Edition, 2000) by analogy of mass spectra (with a minimum of 90% of correspondence) and conformed by retention times of standards when they