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Relationships between the varietal volatile composition of the musts and white wine aroma quality. A four year feasibility study

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ABSTRACT

In this work, the harvest variability of the varietal composition of Fernão-Pires grape variety was evaluated using a dichloromethane liquid–liquid continuous extraction followed by GC-qMS of the free (F) and potential varietal components (PVC) from the musts across four harvests (1998, 1999, 2000, and 2002). This study showed that Fernão-Pires musts exhibited varietal volatile composition variability along the years. Based on the data obtained, using GC-qMS in tandem with PCA, relationships were established between the varietal volatile composition of the musts and the white wine aroma quality classification conferred by the official wine taster chamber. The results of the volatile analyses were consistent with the wine quality classification, indicating that the wine aroma quality is clearly associated with the musts free varietal volatile composition. On the other hand, the PVC fraction allowed the distinction of the musts according to the composition of potential aroma precursors. The proposed approach can provide information to winemakers concerning the winemaking methodologies that can be implemented to improve the varietal wine aroma quality.

1. Introduction

The aroma is one of the most important factors in determining wine character and quality. Several studies recognised a relationship between the wine character and the grape and musts volatile compounds, namely terpenoids (Gunata, Bayonove, Baumes, & Cordonnier, 1985; Wilson, Strauss, & Williams, 1986), and aromatic alcohols (Rocha et al., 2000). The study of the volatile components originating from the nonvolatile precursors has also been the object of several investigations (Gunata, Bayonove, Baumes, & Cordonnier, 1985; Ugliano, Bartowsky, McCarthy, Moio, & Henschke, 2006; Ugliano & Moio, 2008; Voirin, Baumes, Gunata, Bitteur, & Bayonove, 1992; Voirin, Baumes, Sapis, & Bayonove, 1992). These precursors have been reported as glycosides having the aroma compounds as their aglycons. The grape aroma potential might be potentially released by different manners. It may be naturally revealed during fruit maturation by endogenous enzymes identified as β-glucosidases. Since these enzymes show activities that are variable across the different years and do not always release their whole aroma potential, hydrolytic experiments have been performed with exogenous β-glucosidases (Gunata, Bayonove, Tapeiro, & Cordonnier, 1990; Rocha, Coutinho, Delgadillo, Dias Cardoso, & Coimbra, 2005). Furthermore, some monoterpenoids may be formed during winemaking by yeast-driven hydrolysis of glycosides, which allows to infer a synergy between grape volatile composition and yeast metabolism. This synergy can play an important role in the final wine volatile concentration (Ugliano & Moio, 2008; Ugliano et al., 2006). Some monoterpenoids and norisoprenoids can also be formed during winemaking mainly associated with acid-catalysed reactions (Ugliano & Moio, 2008; Ugliano et al., 2006).

Bound aroma, potentially developed during winemaking, is unknown for the majority of grape cultivar appellations. Some French, German, and Spanish varieties (Carro-Mariño, López-Tamames, & García-Jarés, 1995; Falqué, Fernández, & Dubourdieu, 2002; Gómez, Martínez, & Laencina, 1995; Guth, 1997; Lopéz-Tamames et al., 1997; Rosillo, Salinas, Garijo, & Alonso, 1999; Sefton, Francis, & Williams, 1993; Skouroumounis & Winterhalter, 1994; Versini, Orriols, & Serra-Adalla, 1995; Vilanova & Martínez, 2007; Vilanova & Sieiro, 2006; Vilanova, Zamuz, Vilariño, & Sieiro, 2007) have been studied to establish databases of flavour compounds. The knowledge of the varietal volatile composition offers a means of evaluating the potential aroma of a variety and to improve the wine aroma quality.

Bairrada is one of the ancient winemaking regions in Portugal, although Bairrada Appellation was only officially created in 1979. Fernão-Pires is the main white grape variety in Bairrada, which

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represents 80% of the white vineyard. This variety is spread throughout Portuguese Appellations, where it is also known by the names "Maria Gomes" and "Gaeiro". Fernão-Pires contains terpenoids (Coelho, Rocha, Barros, Delgadillo, & Coimbra, 2007; Rocha, Coelho, Zrostlíková, Delgadillo, & Coimbra, 2007a), aliphatic and aromatic alcohols, ketones, aliphatic acids, and C_{13} norisoprenoids (Coelho et al., 2007; Rocha, Coutinho, Barros, Delgadillo, & Coimbra, 2007b; Rocha et al., 2000). However, not all volatile components of this variety occur in free form but can be released after the use of an appropriate winemaking technology (Rocha et al., 2000, 2005), contributing individually to the overall wine aroma (Cordonnier & Bayonove, 1978). This fact was particularly relevant for this variety, as previously demonstrated (Rocha et al., 2005). Because of the considerable importance of volatile monoterpenoids, aromatic alcohols, and C_{13} norisoprenoids to flavour and varietal character of *Vitis vinifera* varieties (Bayonove, Baumes, Crouzet, & Günata, 1998), a particular attention was devoted in this study to compounds from these classes.

In wines produced from grapes of the same variety and from the same *terroir*, the factors that have a paramount influence on the maturation process and, consequently, on the composition of grapes and wines quality are related to the climatic conditions, such as precipitation, temperature, wind, and sun exposure (Esteves & Orgaz, 2001; Jones & Davis, 2000). In particular, the sun exposure effect has been recognised to have a very important role in the variations of the grapes and wine volatile composition (Belancic et al., 1997; Bergqvist, Dokoozlian, & Ebisuda, 2001; Bureau, Baumes, & Razungles, 2000). Since these external factors may vary across the harvests, they may have a major influence on the wine quality. Thus, the possibility to assess the quality of the wine aroma from the must will allow the selection of the appropriate winemaking procedure for each harvest to achieve the best wine quality.

Although several studies have been focused on the Fernão-Pires volatile characterisation (Coelho et al., 2007; Rocha et al., 2000, 2005, 2007a, 2007b), no information is yet available concerning its harvest variability. Thus, in this work, the variability of the varietal composition of Fernão-Pires grapes across four harvests (1998, 1999, 2000, and 2002) was evaluated using a liquid–liquid continuous extraction followed by analysis by gas chromatography coupled with quadrupole mass spectrometry (GC-qMS) of the free (F) and potential varietal components (PVC) present in their musts. Based on the data obtained, a PCA was applied to seek relationships between the varietal volatile composition of the musts and the white wine aroma quality classification.

2. Materials and methods

2.1. Materials

V. vinifera var. healthy-state Fernão-Pires grapes from 1998, 1999, 2000, and 2002 harvests were collected in the Bairrada Appellation, and the musts were prepared in Estação Vitivinícola da Bairrada, Anadia. The musts were treated with SO_2 (60 mg l^{-1}) and were clarified by centrifugation. The samples were stored at −20 °C until use. Each must was analysed (liquid extraction plus GC-qMS analysis) in the following days of its preparation.

The white wines were prepared in Estação Vitivinícola da Bairrada, using the same winemaking procedure. Each wine was prepared in the year of harvest. After one month of production, the wines were decanted, transferred to 0.75 l bottles, sulphited, and the bottles stoppered and stored at 10 °C until use. The wines were sensory evaluated one year after bottling by the Bairrada Appellation official taster chamber of CVRB (Comissão Vitivinícola da Região da Bairrada), involving at least 5 of the 8 official tasters. As the panel has experience in the evaluation of Fernão-Pires white wines, no specific training step was implemented. It was used the official room with individual spaces for each taster, white light, and normalised sensory glasses. Three bottles of wine obtained from each type of must (1998, 1999, 2000, and 2002) were evaluated and rated according to a scale ranging between 0 (very weak) and 5 (excellent), corresponding to a global classification based on aroma, colour, taste, and transparency global properties.

2.2. Methodology for obtaining the musts volatile composition

This methodology comprises two approaches: i) the direct liquid–liquid extraction of the musts which allows to obtain the free volatile compounds (F); and ii) the heat treatment of the musts followed by an enzymatic treatment and liquid–liquid extraction, which allows to obtain the potential volatile compounds (PVC).

2.2.1. Heat treatment and enzyme hydrolysis

To inactivate the endogenous enzymes and to eliminate the free volatile components, the musts were heated under stirring for 15 min at 100 °C, at pH 3.2 (Cordonnier & Bayonove, 1974; Rocha et al., 2000). A commercial enzymatic preparation usually used in white winemaking, Lallzyme de Lalvin (14.1 mg), obtained from Lallemand/Proenol (Vila Nova de Gaia, Portugal), was added to 250 ml of must, and the mixture was incubated at 35 °C for 24 h. This enzymatic preparation is reported by the producer to have activity of β-glucosidase, pectinase, arabinosidase, and rhamnosidase. After incubation, the released aglycons were extracted as described below.

2.2.2. Extraction method

The extraction procedure was a modification of the method described by Etievant (Etievant, 1987; Rocha et al., 2000). The musts obtained without and with heat + enzymatic treatment, which contained, respectively, the free (F) and the potential volatile components (PVC), were submitted to a process of liquid–liquid continuous extraction with dichloromethane. Six independent extractions were done for each one of the eight must samples (98_F, 99_F, 00_F, 02_F 98_{PVC}, 99_{PVC}, 00_{PVC}, and 02_{PVC}), in a total of 48 extractions. The musts (250 ml), supplemented with internal standard (130 mg of 3-octanol) and 75 ml of dichloromethane, were placed in the extractor. Extractions were carried out for 24 h at *ca.* 50 °C. The dichloromethane extracts were cooled to −20 °C to separate the frozen water from the organic phase by decantation and then dried over anhydrous sodium sulphate. The excess of low-boiling point solvent was removed by distillation at low pressure using a trap with liquid nitrogen. The concentrates (*ca.* 1 ml) were stored in glass screw-top vials at −20 °C.

2.3. Chromatographic conditions (GC-qMS)

The musts extracts were analysed by GC-qMS on a Hewlett–Packard 5890 series II gas chromatograph, equipped with a 30 m × 0.32 mm (*i.d.*) DB-FFAP fused silica capillary column, 0.25 μm film thickness (J&W Scientific, Folsom, CA, USA), connected to a Hewlett-Packard quadrupole mass selective detector, according to the method described by Rocha et al. (Rocha, Delgadillo, & Ferrer-Correia, 1996; Rocha et al., 2000). Splitless injections were used (5 min). The oven temperature was programmed from 35 to 220 °C at 2 °C min^{-1}, the injector temperature was 255 °C, and the transfer line was heated at 250 °C. The helium carrier gas had a flow of 1.7 ml min^{-1}. The mass spectrometer was operated in the electron impact mode at 70 eV, scanning the range m/z 30–300 in a 1 s cycle, in a full scan acquisition mode. Identification of volatile compounds was achieved by comparison of the GC retention times and mass