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Volatile compounds and sensorial characterisation of red wine aged in cherry, chestnut, false acacia, ash and oak wood barrels

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ABSTRACT

The wood-related volatile profile of wines aged in cherry, acacia, ash, chestnut and oak wood barrels was studied by GC–MS, and could be a useful tool to identify the wood specie used. Thus, 2,4-dihydroxybenzaldehyde in wines aged in acacia barrels, and ethyl-2-benzoate in cherry barrels could be used as chemical markers of these wood species, for authenticity purposes. Also, the quantitative differences obtained in the volatile profiles allow a good classification of all wines regarding wood species of barrels, during all aging time, and they contributed with different intensities to aromatic and gustative characteristics of aged wines. Wines aged in oak were the best valuated during all aging time, but the differences were not always significant. The lowest scores were assigned to wines aged in cherry barrels from 6 months of aging, so this wood could be more suitable in short aging times.

1. Introduction

The structural characteristics and chemical composition of wood can influence the complex physical, chemical and biochemical processes that take place during the oxidative aging of wines in barrels, affecting their composition, modulating their organoleptic quality and complexity, such as aroma, structure, astringency, and persistence, and contributing to their stability. These changes are the consequence of different process such as spontaneous clarification, oxygen diffusion through wood pores, and extraction of many substances from wood, mainly aromatic compounds, ellagitannins and others. Quantitatively, ellagitannins are the most important ones, since they can represent up to 10% in dry weight of oak heartwood. Once in the wine, they are slowly but continuously transformed through condensation, hydrolysis, and oxidation reactions, giving rise to the formation of other compounds, as their ethyl derivatives and the flavano-ellagitannins (Jourdes, Michel, Saucier, Quideau, & Teissedre, 2011). Consequently, their levels in aged wines will be much lower than those of other compounds, and a fractionation step is always required previously to their detection and quantification. The oak heartwood also shows high levels of low molecular weight phenolic compounds, such as ellagic and gallic acids, besides a great variety of aromatic compounds belonging to very different chemical families, like phenolic aldehydes, phenolic ketones and their isomers, volatile phenols, lactones, furanic compounds, pyranones, and furanones among others (Cadahía, Fernández de Simón, & Jalocha, 2003). Their levels can vary greatly depending on oak species and geographical origin, as well as the processing that undergoes in cooperage, seasoning on the open-air and toasting at different intensities (Cadahía et al., 2003; Spillman, Sefton, & Gawell, 2004).

In recent years, heartwood from species as false acacia (*Robinia pseudoacacia*), chestnut (*Castanea sativa*), and cherry (*Prunus avium*), and more rarely, ash (*Fraxinus excelsior* and *F. vulgaris*), mulberry (*Morus alba* and *M. nigra*), beech (*Fagus sylvatica*), alder (*Alnus glutinosa*) and some local woods are being considered as possible sources of wood for the production of wines and their derived products, like spirits, and especially vinegars, in order to give them a special personality. Thus, some papers about the chemical composition of non-oak heartwoods have been shown in scientific literature, although only oak and chestnut are approved by the International Organisation of Vine and Wine (OIV) to wine aging (De Rosso, Cancian, Panighel, Dalla Bedona & Flamini, 2009a; Fernández de Simón, Esteruelas, Muñoz, Cadahía, & Sanz, 2009; Flamini, Dalla Bedona, Cancian, Panighel & De Rosso, 2007; Sanz et al., 2012a). The most studied compounds were polyphenols, pointing out important chemical differences in relation to oak wood that should be taken into account when consider its use in cooperage. In addition, using gas chromatography–mass spectrometry (GC–MS) to analyse 50% hydroalcoholic and model wine extracts, many volatile compounds were identified in seasoned and toasted heartwoods other than oak, 98 were quantified, among

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them lignin and carbohydrate and lipid derivatives. On the whole, compared to oak, toasted acacia and chestnut woods showed a very high richness of these compounds, as lignin as lipid and carbohydrate derivatives, while cherry and ash were much richer than toasted oak wood in lignin derivatives, but much poorer in lipid and carbohydrate derivatives. Therefore we can expect a characteristic sensorial profile (Culleré, Ferreira, Hernández-Orte, Cacho, Fernández de Simón, & Cadahía, 2013).

Little information about the effects of woods other than oak on the characteristics of wines, vinegars, and other beverages, compared to oak, has been presented in literature. Someone have pointed out a different evolution of the phenolic and volatile composition, and organoleptic properties in beverages aged in barrels or in contact with chips made of different woods (Callejón, Torija, Mas, Morales, & Troncoso, 2010; De Rosso, Panighel, Dalla Vedova, Stella & Flamini, 2009b; Kozlovic, Jeromel, Maslov, Pollnitz, & Orlic, 2010; Chinnici, Natali, Sonni, Bellachiona & Riponi, 2011; Sanz et al., 2012b). Some authors highlight that wines or vinegars aged in non-oak barrels had better organoleptic characteristics (Chinnici et al., 2011; Kozlovic et al., 2010; Hillmann, Mattes, Brockhoff, Dunkel, Meyerhof & Hofmann, 2012). However the physical–mechanical properties of wood barrel, like porosity that influence the gas exchange during aging, can in some cases promote a fast polyphenol oxidation. That effect could be minimised using non-oak wood alternative to barrel products like powder, shavings, chips, cubes, or staves, as cheaper substitute techniques.

The main goal of this work is to know the volatile compounds that woods other than oak contributes to the red wines during aging in barrels, if some of them can be used as chemical markers of aging with non-oak woods, and its possible influence on the sensory characteristics of resulting wines. For this aim, medium toasting 225 L barrels were made with cherry, chestnut, acacia, ash and oak wood, and the volatile composition of a red wine aged with them was studied by GC/MS. Moreover, their organoleptic properties were studied by sensory analysis.

2. Materials and methods

2.1. Woods and wines

Cherry (*Prunus avium*), chestnut (*Castanea sativa*), acacia (*Robinia pseudoacacia*), ash (*Fraxinus excelsior*), and oak (French *Quercus petraea*) heartwood were provided as staves for making barrels by Tonelería Intona, SL (Navarra, Spain). The wood was naturally seasoned for 24 months until 15% humidity. Barrels were made following a traditional process, at medium intensity level of toasting, over a wood fire (185 °C for 45 min). The barrel heads were not toasted. All barrels were with a capacity of 225 L and staves of 28 mm thickness.

Red wine D.O. Somontano (Spain) was produced on an industrial scale by Enate wine cellar in 2009, from cv. Syrah (100%) grapes, according to traditional methods. It was put into the barrels in November 2009, and was kept during 12 months at wine cellar where humidity and temperature conditions were controlled at 70–80% and 13–15 °C. The chemical parameters of this wine were: total acidity 3.9 g/L, volatile acidity 0.28 g/L, alcoholic degree 14.3, pH 3.56, free SO_2 12 mg/L, total SO_2 41 mg/L, color intensity 17.54, total anthocyanes 611 mg/L, and total tannins 2.46 g/L. These parameters were evaluated before the wine was transferred into tanks and also during aging, according to OIV methods (OIV 1990). During this storage time in barrels, wine samples from each barrel were taken, after aging 2, 4, 6, 9 and 12 months. All wines were analysed in duplicate.

2.2. Chemicals

Reference compounds were obtained from commercial sources: Fluka Chimie AG (Buchs, Switzerland), Aldrich Chimie (Neu-Ulm, Germany), Roth (Karlsruhe, Germany), Apin (Oxon, UK), Sigma Chemical (St. Louis, MO), Extrasynthèse (Genay, France), and Transmit (Marburg, Germany), with purity higher 98%. Ethanol, dichloromethane, ammonium sulfate, and anhydrous sodium sulfate were purchased from Panreac (Barcelona, Spain).

2.3. Sensory analysis

The sensory assessment of wines was done by a committee of 10 expert judges, at the Instituto de Ciencias de la Vid y del Vino, located in Logroño (La Rioja). The experts had extensive wine tasting experience (oenologist and wine researcher) and were specially trained in the employment of scales and aroma descriptors used. Samples were presented in standard 125 mL glasses in random order. A 10-unit scale, in which 1 was "attribute not perceptible" and 10 was "attribute highly perceptible", was used. The intensity level of each descriptor was then expressed as the mean value of all the judges from two different testing days. All evaluations were conducted at 20 °C under white light in separate booths. The attributes selected by expert judges were descriptors of visual phase (color intensity, quality), olfactive phase related to primary aromas (varietal, vegetal, fruity), olfactive phase related to the wood–wine interaction (woody, vanilla, spicy, almond, caramel, toasty, smoky, balsamic), and gustative phase (mouthfullness, duration and astringency). Data from all judges for all samples were used, and the average values were compared using the so-called "spider web diagrams". The expert judges also assessed the wines in regard to the overall sensory analysis, in a variable scale for each attribute, being 0 the best score for all them, and 9 the worst for visual quality, 18 for olfactive intensity and quality, 27 for gustative intensity and quality, and also 27 for harmony, so the quality of wine varies inversely to the note issued.

2.4. Extraction

Volatile compounds were analysed using the method described in Fernández de Simón, Cadahia, & Jalocha (2003). Three internal standards were used 3,4-dimethylphenol for volatile phenols (20 mg/L in 95% ethanol), *o*-vanillin for phenolic aldehydes and related compounds (1 mg/mL in 95% ethanol), γ-hexalactone for the remaining compounds (2 mg/mL in 95% ethanol). Liquid–liquid extractions were carried out using dichloromethane. In all cases, the samples were analysed in duplicate.

2.5. Gas chromatography–mass spectrometry analyses

Analyses were performed using a Hewlett–Packard 6890N gas chromatograph (Palo Alto, CA) equipped with a mass spectrophotometric detector model HP 5975B. Samples were injected in split mode (30:1, 0.5 min), and volatiles were separated using a fused silica capillary column (Supelcowax-10) (30 m × 0.25 mm i.d. and 0.25 μm film thickness), supplied by Supelco (Madrid, Spain), and under the working conditions described in Fernández de Simón et al. (2003): GC grade helium as carrier gas at flow rate of 1.15 mL/min, 9.00 psi; column temperature program, 45 °C heated, at 3 °C/min, to 230 °C, held for 25 min, and then heated at 10 °C/min to 270 °C (held for 21 min). The injection temperature was 230 °C. Detection was carried out by electron impact mass (EI) in the full scan mode, using an ionisation energy of 70 eV, and interphase detection temperature 290 °C (MS source at 230 °C, and MS quad at 150 °C). The Kovats Index values were calculated in this column, and in another column with different polarity (ZB-5,