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Effect of wine micro-oxygenation treatment and storage period on colour-related phenolics, volatile composition and sensory characteristics

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ABSTRACT

In this paper, we have evaluated the effects of micro-oxygenation before malolactic fermentation and after five months of storage on Cencibel red wines. In particular, we have considered the colour characteristics, the phenolic compounds related to red wine colour, the individual volatile composition, and the complete descriptive sensory analysis of the wines. The fact that the concentration of the malvidin-3-glucoside-ethyl-flavan-3-ol adducts and pyranoanthocyanins (B-type vitisins) increased is closely related to the red wine colour stabilization. Red wine aroma quality was slightly improved as a consequence of oxygen addition after five months of storage. New attributes appeared (plum/currant) and others were increased (spicy and liquorice) in micro-oxygenated red wines, whereas herbaceous values were significantly decreased. The results suggest the joint use of both treatments (micro-oxygenation and storage) give rise to an enhancement of the colour stability and the aroma and sensorial quality of red wines.

1. Introduction

The micro-oxygenation treatment produces a stabilization of red wine colour and improves the wine quality by the addition of small, continuous and controlled quantities of oxygen. The micro-oxygenation concept was born by the investigation of Patrick Ducournau and Laplace family in 1993, and is based on the colour stabilization and astringency diminution that normally occurred in oak barrels. The application of oxygen before malolactic fermentation has been described by several authors as the optimal moment (Hernández-Orte et al., 2009; Ortega-Heras, Rivero-Pérez, Pérez-Magariño, González-Huerta, & González-Sanjosé, 2008).

On one hand, the oxygen is involved in several wine reactions, mainly those involving phenolic compounds. As a consequence, new products result from the direct and acetaldehyde-mediated anthocyanin–tannin reactions (Escribano-Bailón, Álvarez-García, Rivas-Gonzalo, Heredia, & Santos-Buelga, 2001). Pyranoanthocyanin formation by condensation of anthocyanins with other molecules having polarizable double bond is also possible (Atanasova, Fulcrand, Cheynier, & Moutounet, 2002; Rentzsch, Schwarz, Winterhalter, & Hermosín-Gutiérrez, 2007). These compounds are more stable than genuine anthocyanins, leading to red wine colour stabilization by condensation reactions.

In spite of the scarce studies about micro-oxygenation effects on the volatile compounds, Ortega-Heras et al. (2008) assert that the results depend to a large extent on the grape cultivar and vintage. The differences between micro-oxygenated and untreated wines in terpenes, esters and acetates concentration disappeared after malolactic fermentation (Hernández-Orte et al., 2009; Ortega-Heras et al., 2008), contrarily to that found by Cerdán, Goni, and Azpilicueta (2004) in esters composition. Moreover, the theoretical expected diminution of green and herbaceous aromas in micro-oxygenated red wines, described by several authors (Bertuccioli, Rosi, Lencioni, Zini, & Siliani, 2001), does not seem to be correlated with the six-carbon alcohols concentration decrease (Ortega-Heras et al., 2008).

On the other hand, red wine ageing in bottle produces different effects, like colour stability improvement, spontaneous clarification and more complex and stabilized phenolic pigments (Cruz et al., 2008). Polymerization and condensation reactions occurred during ageing, mainly between anthocyanins and flavan-3-ols (anthocyanin-flavan-3-ol adducts, by direct reaction or acetaldehyde-mediated reaction), and also other phenolic and non-phenolic compounds (formation of pyranoanthocyanins and hydroxyphenyl-pyranoanthocyanins) (Francia-Aricha, Guerra, Rivas-Gonzalo, & Santos-Buelga, 1997). As a consequence, the red-purple colour

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gradually disappears, giving rise to reddish-brown hue (Atanasova et al., 2002). Also, ageing red wines improves the aroma and taste quality due to the diminution of astringency (Puech, Feuillat, Mosedale, & Puech, 1996). Although the aroma stabilization during storage greatly depends on the grape variety, the floral and fruity aromas produced by monoterpenes and acetates and esters, respectively, decrease after bottling (Pérez-Coello, Martín-Álvarez, & Cabezudo, 1999). The effect on wine colour-related phenolic compounds, volatile composition and sensory characteristics during storage is well-known, but the combined effect of wine micro-oxygenation and storage has not been previously reported.

The aim of this research was to study the effects of micro-oxygenation and later storage on the colour parameters, phenolic and volatile composition and subsequently descriptive sensorial characteristics. The study has been performed in Cencibel red wines, a large extent cultivar in Castilla-La Mancha (Spain), developing a complete study not previously reported.

2. Material and methods

2.1. Winemaking

Red wine made from *Vitis vinifera* grape cv. Cencibel (harvested at the optimal maturity stage and in good sanitary conditions) was elaborated in the experimental winery of Castilla-La Mancha University (in central-southeast Spain), following widespread winemaking methodology. After manual harvest, the mass obtained was sulphited (80 mg/L of total SO_2; 29 mg/L of free SO_2), destemmed and crushed. Fermentations were carried out in duplicate after inoculation with *Saccharomyces cerevisiae* race *cerevisiae* yeasts (CECT No. 10835) with skin maceration. Fermentations were conducted at controlled temperature (24–26 °C). After alcoholic fermentation, and previous to malolactic fermentation, the wine was micro-oxygenated. Inhibition of the development of malolactic fermentation during oxygen treatment was achieved by addition of 20 g/HL of lysozyme. The wine was homogeneously distributed within 4 stainless steel tanks of 2000 L of capacity and 2 m of height, which guarantee the complete oxygen dissolution in wine during the micro-oxygenation treatment. Two tanks were submitted to micro-oxygenation treatment and the other two tanks contained untreated, non-micro-oxygenated control wine.

The micro-oxygenation treatment consisted in an oxygen dose of 10 mL/L/month during 20 days at 20 °C, by means of a micro-diffusion system (Laffort, Spain). The dose supplied were appropriated according to the wine total polyphenol index (TPI = 65-70) and the manufacturer recommendations.

After micro-oxygenation treatment, the red wines were inoculated with 1g/HL of a commercial lactic acid bacteria strain of *Oenococcus Oeni* (Lactobacter SP1; Laffort, Spain). The development of malolactic fermentation was monitored by TLC and malic acid and lactic acid enzymatic measurement. The wine characteristics were monitored at different moments of the process: after micro-oxygenation treatment, after malolactic fermentation completion (only for the colour-related phenolics) and after subsequent five months of storage in stainless steel vats (16 °C of temperature and dark conditions). All the sample replicates were analyzed in duplicate.

Wine conventional analytical data and development of malolactic fermentation were obtained by O.I.V. official methods (1990).

2.2. Analysis of wine polyphenolic compounds and colour parameters

A Hewlett Packard 8452A apparatus was used for the analysis of main phenolic types by spectrophotometry. Total polyphenolic, anthocyanins, hydroxycinnamic acid derivatives and flavonols families (Mazza, Fukumoto, Delaquis, Girard, & Ewert, 1999), flavan-3-ols family (Amerine & Ough, 1980), and tannins (Glories, 1988) have been measured. Also, the chromatic characteristics in the CIELAB space (Pérez Caballero, Ayala, Echávarri, & Negueruela, 2003) L^*, C^*, h^*, a^*, b^* and colour parameters (colour intensity and tonality) (Glories, 1984) were calculated. The method described by Hermosín-Gutiérrez (2003) was used for the determination of the percentage contributions of copigmented and polymerized anthocyanins to the total wine colour at pH 3.6.

HPLC separation, identification and quantification of phenolic compounds were performed on an Agilent 1100 series system (Agilent, Waldbronn, Germany), equipped with a DAD photodiode detector (G1315B) and an LC/MSD Trap VL (G2445C VL) electrospray ionization mass spectrometry (ESI/MS^n) system, according to the method proposed by Castillo-Muñoz, Gómez-Alonso, García-Romero, and Hermosín-Gutiérrez (2007). On one hand, direct injection of the must and wine samples were used for the analysis of the anthocyanins, benzoic acid derivatives and flavan-3-ols compounds, and quantification was made using the DAD-chromatograms obtained at 520 and 280 nm, respectively. Monomeric anthocyanins and anthocyanin-ethyl-flavan-3-ols were quantified using the calibration curve of malvidin-3-glucoside, whereas the calibration curve of pinotin A was used to quantify pyranoanthocyanins and hydroxyphenyl-pyranoanthocyanins. In the case of benzoic acids and flavan-3-ols, individual calibration curves were employed to quantify each individual compound. On the other hand, anthocyanins-free extracts were obtained by SPE on Oasis MCX cartridges for the isolation of red wine flavonols and hydroxycinnamic acid derivatives, according to Castillo-Muñoz et al. (2007). The chromatographic method used was that proposed by Castillo-Muñoz et al. (2009). Quantification was made using the DAD-chromatograms obtained 360 and 320 nm, respectively. Individual calibration curves were obtained for each flavonol, with some exceptions: on the one hand, myricetin and laricitrin 3-glycosides were quantified as myricetin-3-glucoside and, on the other hand, laricitrin, kaempferol 3-glycosides and quercetin-3-galactoside were quantified as myricetin, kaempferol-3-glucoside and quercetin-3-glucoside, respectively. Similarly, individual calibration curves were used to quantify each hydroxycinnamic acid and their respective esters. For identification, the ESI-MS^n was used in positive mode for anthocyanins and flavan-3-ols, whereas both positive and negative modes were used for flavonols and hydroxycinnamic acid derivatives.

2.3. Analysis of wine volatile compounds

After centrifugation, samples were directly injected in a Hewlett Packard 5890 Series II Gas Chromatograph, coupled to a flame ionization detector, according to the method proposed by Sánchez-Palomo, González-Viñas, Díaz-Maroto, Soriano-Pérez, and Pérez-Coello (2007) for the determination of major volatile compounds.

Minor volatile compounds of wines were extracted in duplicate by Solid Phase Extraction (SPE) technique, according to the method proposed by Sánchez-Palomo, Alañón, Díaz-Maroto, González-Viñas, and Pérez-Coello (2009). The extract was injected into an Agilent Technology 6890N Network GC System equipped with an Agilent Technology 5973 *inert* Mass Selective Detector, according to the method proposed by Sánchez-Palomo et al. (2007). NBS75K and Wiley A libraries were used for compounds identification, together with the comparison of the GC retention times and mass spectra of the pure substances. The response factor of each compound was experimentally obtained by injection of commercial standards. For compounds without reference compounds available, the response factors of standards with similar chemical structures were used.