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Oxygen consumption by oak chips in a model wine solution; Influence of the botanical origin, toast level and ellagitannin content



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ABSTRACT

The botanical origin, toast level and ellagitannin content of oak chips in a model wine solution have been studied in terms of their influence on oxygen consumption. French oak chips released significantly higher amounts of ellagitannins than American oak chips at any toast level. The release of ellagitannins by oak chips decreased as the toast level increased in the French oak but this trend was not so clear in American oak. Oxygen consumption rate was clearly related to the level of released ellagitannins. Therefore, oak chips should be chosen for their potential to release ellagitannins release should be considered, not only because they can have a direct impact on the flavor and body of the wine, but also because they can protect against oxidation.

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1. Introduction

Oak chips have been widely used to flavor wine for many years now (Bertrand, Barbe, & Gazeau, 1997; Zamora, 2003a). They enrich the wine with the same substances that are released during oak barrel aging (Zamora, 2003b). It is well known that contact with oak wood, either as barrels or as alternatives (staves, chips, etc.), enriches the wine with substances that improve its aroma such as furfural (toasted almonds), dimethyl pyrazines (cacao), maltol (caramel), whiskey-lactones (cocoa), vanillin (vanilla), eugenol (cloves) and volatile phenols (smoky notes) (Ferreira, 2010; Gallego et al., 2012; Pérez-Juan & Luque de Castro, 2015). Oak also releases other non-volatile compounds that participate in the flavour and texture of wine. Ellagitannins are one of these such non-volatile compound, and they contribute to astringency and mouthfeel (Chira & Teissedre, 2013; De Simón, Sanz, Cadahía, Poveda, & Broto, 2006).

Aging in oak barrels also oxygenates wine because small quantities of oxygen can reach the wine through the pores in the wood, the

interstices between the staves, and the bunghole (Del Alamo-Sanza & Nevares, 2014; Nevares, Gonzalez, Crespo, & Del Alamo-Sanza, 2014; Vivas, Glories, & Raymond, 1997). This supply of oxygen is positive for red wine because it stabilizes color, reduces astringency, and removes excess vegetal notes. The dissolved oxygen leads to the formation of ethanal from ethanol. The ethanal can then react with flavanols to form a very reactive carbocation which, in turn, quickly reacts with either another flavanol molecule or an anthocyanin to produce ethyl-bridged flavanol–flavanol and/or flavanol–anthocyanin oligomers (Escribano-Bailón, Alvarez-Garcia, Rivas-Gonzalo, Heredia, & Santos-Buelga, 2001). It has been reported that ethanal also participates in the formation of new pigments such as vitisin B and other pyranoanthocyanins (Fulcrand, Benabdeljalil, Rigaud, Cheynier, & Moutounet, 1998; Mateus, Silva, Rivas-Gonzalo, Santos-Buelga, & Freitas, 2002). However, in white wines this oxygen supply may be negative because it can lead to wine oxidation. White wines have no anthocyanins and their flavanol concentration is very low (Cáceres-Mella et al., 2013). For this reason white wines are usually aged in oak barrels in contact with the lees, which consume oxygen and protect the wine against oxidation (Comuzzo et al., 2015; Salmon, Fornairon-Bonnefond, Mazauric, & Moutounet, 2000).

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The use of oak alternatives coupled with micro-oxygenation can reproduce the processes taking place in the barrels more economically and quickly (Llaudy et al., 2006; Cejudo-Bastante, Hermosín-Gutiérrez, & Pérez-Coello, 2011). As has been mentioned above, red wines require oxygen to induce a series of reactions between phenolic compounds so that color can be stabilized and astringency decreased (Kontoudakis et al., 2011; Ribéreau-Gayon et al., 1998). When white wines are kept in contact with the lees, reduction odour needs to be prevented (Rodríguez et al., 2005; Zamora, 2002). Particular care should be taken to ensure that the oxygen supply is appropriate since an excess may cause oxidation and affect the quality. In this regard, it has been postulated that the ellagitannins released from oak can consume oxygen and protect the wine from excessive oxidation (Poinsaut, 2000; Vivas, 2001).

Several studies have been made on how oak alternatives can contribute to wine flavor (Chassin, 1999; Pérez-Coello et al., 2000; Wilker & Gallander, 1988), but little is known about how they affect the release of ellagitannins and even less is known about the oxygen they consume (Vivas & Glories, 1996). Therefore, the objective of our work was to study the relationship between the release of phenolic compounds, especially ellagitannins, from oak chips and the oxygen consumption of a model wine solution. In particular, the influence of botanical origin, toast level and ellagitannin release was analyzed.

2. Materials and methods

2.1. Chemicals and equipment

Methanol, formic acid and acetic acid of high performance liquid chromatography (HPLC) grade (>99%) and absolute ethanol, ethyl acetate, L(+)-tartaric acid and sodium hydroxide pellets were purchased from Panreac (Barcelona, Spain); gallic acid, copper (II) sulfate pentahydrate and iron (III) chloride hexahydrate from Sigma-Aldrich (Madrid, Spain); ellagic acid from Fluka (Sigma-Aldrich, Madrid, Spain). Ellagitannins (Vescalagin, Castalagin, Roburin A, Roburin D, Granidin and Roburin E) were purchased from Adera (Pessac, France).

All spectrophotometric measurements were performed with a Helios Alpha UV-vis spectrophotometer (Thermo Fisher Scientific Inc., Waltham, MA, USA).

HPLC separation, identification and quantitation of ellagitannins were performed on an Agilent 1100 Series system (Agilent, Germany), equipped with DAD (G1315B) and an LC/MSD Trap VL (G2445C VL) electrospray ionization mass spectrometry (ESI-MSn) system, and coupled to an Agilent ChemStation (version B.01.03) data-processing station. The mass spectra data were processed with the Agilent LC/MS Trap software (version 5.3).

2.2. Oak chips

For this study we used 2, 5 and 10 g/L of French oak (*Quercus petraea*) and American oak (*Quercus alba*) chips and three toast levels (low, medium and high). The chips were quadrangular with a size of 5–8 mm long; 5–8 mm wide and 3.5–4.5 mm deep. They were provided by the company Tonnellerie Radoux-Pronektar (Jonzac, France). In the case of medium toast French oak, chips with three levels of potential ellagitannin release (PER). The PER was determined by the supplier company itself (Tonnellerie Radoux-Pronektar) using its non-invasive measurement method based on infrared spectrometry, (Radoux OakScan™) (Michel et al., 2011, 2013) high, medium and low PER were also used. Hence, high, medium and low PER staves were employed in this experiment.

2.3. Experimental design

A model wine solution was used to estimate the oxygen consumption. The model wine solution was composed of ethanol: 12% (v/v); tartaric acid: 4 g/L; pH = 3.5; iron: 3 mg/L, in the form of iron (III) chloride hexahydrate and copper: 0.3 mg/L in the form of copper (II) sulfate pentahydrate. Three white wines and three red wines were also used to compare the kinetics of oxygen consumption of the different oak chips with those of these wines. The ethanol content (ET), Titratable acidity expressed as tartaric acid (TA), free sulfur dioxide (FS) and Total phenolic index (TPI) were measures to characterize these wines. The chemical characteristics of these wines was: white wine 1: ET: 10.7% (v/v); TA: 6.7 g/L; FS: 22 mg/L and TPI: 4.7; white wine 2: ET: 11.2% (v/v); TA: 6.3 g/L; FS: 18 mg/L; TPI: 4.3; white wine 3: ET: 12.7% (v/v); TA: 5.3 g/L; FS: 28 mg/L; TPI: 6.2; red wine 1: ET: 13.2%; TA: 5.9 g/L; FS: 18 mg/L; TPI: 56.1; red wine 2: ET: 12.6%; TA: 5.6 g/L; FS: 23 mg/L; TPI: 48.6; red wine 3: ET: 14.3%; TA: 4.8 g/L; FS: 32 mg/L; TPI: 71.3. None of these wines had been in contact with oak or had been supplemented with ascorbic acid or oenological tannin.

The oak chips were placed in clear glass bottles into which a pill had previously been inserted (PreSens Precision Sensing GmbH, Ordering Code: SP-Pst3-NAU-D5-CAF; Batch number: 1203-01_Pst3-0828-01, Regensburg, Germany) for the non-invasive measurement of dissolved oxygen by luminescence (Nomasense™ O₂ Trace Oxygen Analyzer by Nomacorc S.A., Thimister Clermont, Belgium). Three doses of oak chips (2, 5 and 10 g/L) were assayed. The bottles were completely filled with the model wine solution previously saturated with oxygen. In parallel, other bottles were filled with the different wines without supplementation with oak chips.

The wine model solution and the various wines were saturated in oxygen by bubbling with air for 10 min. Once the bottles had been closed with a crown cap and bidule, to minimize the volume of headspace, oxygen (Diéval, Vidal, & Aagaard, 2011) was measured periodically. Oxygen was measured every day during the first 5 days and after every 2–3 days until the end of the experiment. Fig. 1 shows a schematic of the experimental design. All assays were performed in triplicate taking control bottles with the oxygen-saturated model wine solution without added oak chips as control reference. After 140 days of maceration, the bottles with 10 g/L of oak chips were opened for ellagitannins analysis. The various samples were strained and centrifuged (12,000×g 10 min).

2.4. Color analysis

The color intensity (CI) was estimated as described by Glories (1984). The CIELab coordinates, lightness (L*), chroma (C*) and

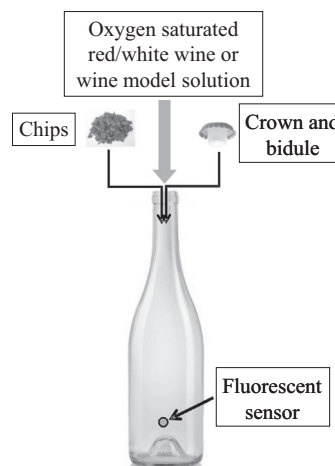


Fig. 1. Experimental design for oxygen consumption measurement.

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