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Ellagitannin content, volatile composition and sensory profile of wines from different countries matured in oak barrels subjected to different toasting methods

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ABSTRACT

Ellagitannins and aromatic compounds evolution in Cabernet Sauvignon wines macerated in oak barrels for a year was studied. Identical barrels with different toastings (medium toasting, medium toasting with watering, Noisette) were used in French, Italian and USA cellars. Ellagitannins increased by 84–96% with aging time, as did woody volatiles, by 86–91% in French wines and 23–35% in Italian wines, while fruity aroma compounds declined by 50–57% in the French and Italian wines over a 12-months period. Nevertheless, other behaviors and different kinetics rates for these compounds were observed depending on barrel toasting, wine matrix and their interactions. Perceived overall woody intensity was closely related to *trans*-whiskey lactone, guaiacol and vanillin, whereas astringency and bitterness were significantly linked to ellagitannins ($p < 0.05$).

This is the first study that evaluates the toasting effect on wines from different countries matured in the same oak barrels.

1. Introduction

Ageing in oak barrels is a traditional practice in red winemaking, commonly accepted because of its benefits on flavor, aromatic complexity, color stabilization, astringency modulation, stability and structure configuration of wines (Fernández de Simón et al., 2014). This improvement of the overall quality and organoleptic properties of wines is largely due to the slow and continuous diffusion of oxygen through the wood pores, as well as to the progressive extraction of several oak wood components from barrel to wine during ageing. Aromatic compounds and hydrolysable tannins (ellagitannins) are among these substances.

The volatile compounds may be naturally present in the original oak wood, or may be derived from other wood compounds during the barrel manufacture. Among the aromatic volatiles susceptible to migrate from oak wood to wine during ageing, *cis* and *trans* isomers of β-methyl-γ-octalactone (whiskey lactones), the phenolic aldehyde vanillin, as well as the volatile phenols eugenol and guaiacol, are considered to play a major role in the organoleptic quality of wood-matured wine. Whiskey lactones contribute to olfactory characteristics of wine with coconut, woody and oak-like notes, whereas vanillin is related to vanilla and coffee smells, and volatile phenols to spicy and smoked flavors (Prida & Chatonnet, 2010).

Meanwhile, ellagitannins may occur in levels up to 10% in dry weight of oak heartwood, with castalagin and vescalagin generally being the most abundant ones (Chira & Teissedre, 2013b). Once in the wine, these phenolic compounds are slowly but continuously transformed into other compounds, such as their ethyl derivatives and flavano-ellagitannins (Jourdes, Michel, Saucier, Quideau, & Teissedre, 2011). Ellagitannins have been reported to have a significant impact on astringency sensation and bitterness perception of wines (Chira & Teissedre, 2013b; Glabasnia & Hofmann, 2006).

During the ageing period, the transfer of compounds from oak wood to wine depends mainly on the amount of compounds potentially extractable, on the contact time between wood and wine, as well as on the composition of the wine matured in barrels (García, Soares, Dias, Costa Freitas, & Cabrita, 2012). The pool of potential extractable compounds, both aroma volatiles and ellagitannins, originally present in oak wood is conditioned by the species and geographical origin of the wood, the seasoning of the staves (natural or artificial, length and location) and the toasting of the barrel (method, temperature, length) (Cadahía, Fernández

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de Simon, & Jalocha, 2003; Cadahía, Varea, Muñoz, Fernández de Simón, & García-Vallejo, 2001; Fernández de Simón, Cadahía, del Alamo, & Nevares, 2010; García et al., 2012; Matricardi & Waterhouse, 1999). Among these factors, the toasting process is reported to have the major influence on the chemical composition of the wood (Jordão et al., 2006). Burning the inner surface of the barrels induces severe modifications on wood chemical composition, which in turn will influence the wine composition during ageing. Specifically, this heating treatment results in a thermodegradation of wood constituents. On the one hand, ellagitannin content decrease by hydrolyzation (Cadahía et al., 2001). On the other hand, wood macromolecules such as cellulose, hemicelluloses and lignins, break down and release many new volatile compounds (Cadahía et al., 2003).

Literature usually reports the toasting effect on the composition of the wood (Fernández de Simón, Cadahía, del Alamo, et al., 2010; García et al., 2012; Matricardi & Waterhouse, 1999) and/or of a single wine or model solution (Chira & Teissedre, 2013a, 2013b; Fernández de Simón, Cadahía, del Alamo, et al., 2010; Fernández de Simón, Cadahía, Muiño, Del Álamo, & Nevares, 2010; Soares, García, Costa Freitas, & Cabrita, 2012). Even the impact of the toasting method on the composition of a single wine depending on wood geographical origin has been described by Chira and Teissedre (2015). Nevertheless, research about the influence of toasting on the composition of different wines is rather scarce. Only Návojská, Brandes, Nauer, Eder, and Frančáková (2012) and Rodríguez-Rodríguez and Gómez-Plaza (2011) studied the toasting effect on composition of different wines from the same wine cellar. Specifically, Návojská et al. (2012) investigated the impact of different toasted oak chips on wines from four different varieties, and Rodríguez-Rodríguez and Gómez-Plaza (2011), the effect of different barrel toastings on wines from two consecutives vintages. To the best of the authors' knowledge, no studies about the impact of barrel toasting on wines from different countries have been carried out until now.

Furthermore, there is scarce scientific research focused on the evolution of fruity aromatic compounds during ageing of red wines in oak wood barrels (De Rosso, Panighel, Vedova, Stella, & Flamini, 2009; Garde-Cerdán, Torrea-Goñi, & Ancín-Azpilicueta, 2002, 2004; Jiménez Moreno & Ancín-Azpilicueta, 2006), and the effect of the barrel toasting on it has not been previously investigated.

Thus, the principal aim of this research was to address this lack of information of great interest to optimize wine quality. Might the same toasted barrel lead to different wine evolution depending on the grape origin? And, might a different trend be observed depending on the toasting conditions of the barrel?

To the best of the authors' knowledge, the present study is the first to evaluate and compare the chemical and sensory evolution of wines of the same grape variety from different countries matured in the same oak barrels (Tonnellerie Nadalié, France) during a 12-months period.

2. Materials and methods

2.1. Oak wood origin and drying conditions

All barrels used were made up of French oak from two species (*Quercus robur* and *Quercus petraea*) from the same forest located in the Center region of France. The raw staves (100 cm × 11 cm × 0.12 cm) were naturally seasoned for 24 months in the Tonnellerie Nadalié (Ludon-Médoc, France) wood yard. Once assembled, barrels (225 L) were submitted to different toasting procedures using the traditional way over an oak wood fire. Specifically, three different toasting levels were provided: Noisette, 62 min at 52 ± 3 °C; MT (medium toast) and MTAA (medium toast with watering), 68 min at 57 ± 3 °C. In the case of MTAA toasting, the watering process took place twice (20 L of water in both cases): first, before starting the toasting procedure, and secondly, after 53 min of toasting; then, the barrel drum was placed again above the fire in order to be heated for 15 min more. The barrel heads were not toasted. For the purpose of the study, three barrels of each toasting procedure were provided to each wine cellar.

2.2. Red wine vinification and sample collection

Cabernet Sauvignon grapes (*Vitis vinifera* L.) were manually harvested at maturity in three different countries (France, Italy and USA) during the 2013 vintage. In Château La Tour Carnet (Saint-Laurent-Médoc, France), grapes were crushed the day of harvest and some SO_2 (5 g/100 kg) was added during the transfer of must to stainless steel tanks. *Saccharomyces cerevisiae* was included to perform alcoholic fermentation at 25 °C. Once the alcoholic fermentation was finished (10–12 days), the temperature of the stainless steel tanks was maintained at 21 °C in order to promote spontaneously the malolactic fermentation, which extended for 40–50 days.

In Tolaini Societa' Agricola S.R.L. (Siena, Italy), a lower amount of SO_2 (2 g/100 kg) was added during the transfer of must to stainless steel tanks, but 3.5 g/hL were additionally incorporated to wine after malolactic fermentation. No *Saccharomyces cerevisiae* were added to develop alcoholic fermentation, which lasted for 12 days at 18–29 °C. Then, malolactic fermentation took place during 45 days at a maintained temperature of 19 °C.

In Beaulieu Vineyard (Rutherford, CA, USA), SO_2 (5 g/100 kg) was added over the fermentation processes. Alcoholic fermentation was carried out at 15–30 °C during 10 days, using *Saccharomyces cerevisiae*. Then, wines underwent malolactic fermentation at the same temperature interval for an unknown period of time.

Once the malolactic fermentation was concluded, wines were transferred and kept in oak barrels for ageing during 12 months at a controlled temperature of 15–16 °C. All three toasting methods (MT, MTAA and Noisette) described above were tested with wines from all three countries (France, Italy and USA) in study. A wine sample was taken as control before being placed into the barrels. During the year of storage, wine was sampled from oak barrels of each toasting method and country after 10 days, 6 and 12 months of contact. Then, they were bottled (without filtering) and stored at 16 °C until further analysis.

2.3. Oenological parameters in wines

Conventional oenological parameters of wines, i.e., pH, alcoholic degree (%), titratable acidity (g/L tartaric acid) and total polyphenol index (TPI), were determined in duplicate by Infrared Spectrometry with Fourier Transformation (IRTF) with a WineScan™ Flex (FOSS Analytical, Denmark), which was previously calibrated with wine samples analyzed in accordance with official OIV methods.

2.4. Total phenolics, proanthocyanidins and anthocyanins spectrophotometric analysis

Total polyphenol, proanthocyanidin and anthocyanin contents of wines were spectrophotometrically determined. Total phenolic content was measured according to a modified Folin Ciocalteu method to be applied in 96-well microplates. For the measurement, wines and Folin–Ciocalteu reagent were diluted at a ratio 1:20 and 1:10, respectively, with distilled water. In this order, 20 μL of wine or gallic acid standard solutions (0−200 ppm), 100 μL of the diluted Folin–Ciocalteu reagent and 80 μL of the 7.5% (w/v) Na_2CO_3 solution were placed in each well. The mixture