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Extraction of oak volatiles and ellagitannins compounds and sensory profile of wine aged with French winewoods subjected to different toasting methods: Behaviour during storage

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ABSTRACT

In Merlot wines the evolution of volatile and non-volatile (ellagitannins) compounds extracted from winewoods while being macerated for 12 months was studied. Seven types of winewoods subjected to different toasting methods were used. Different rates of extraction, depending mainly on wood compounds origin (toasting or naturally present in wood) and on the watering process during toasting, were observed, which were reflected in sensory differences. Globally, volatile phenols together with aldehydes, phenols and lactones showed an increase with increasing maceration time. Ellagitannins were extracted faster during the first 3 months; after 6 months an important decrease was observed. Wines with winewoods subjected to watering during toasting were lower in ellagitannins concentrations and demonstrated the greatest decrease of these compounds during maceration. Astringency and bitterness intensified with increasing ellagitannins. Lactones induced positive sweetness sensations, whereas furanic and guaiacol compounds influenced bitterness and astringency. Spicy and vanilla descriptors were related to eugenol, vanillin and other odorous chemicals.

1. Introduction

Oak barrels have long been used in fine wine making, initially for easy product handling during production, storage and transport. Oak wood positive effects on wine development became appreciated, namely the ceding of pleasant aromas, and the regulation of red wine colour. During ageing in oak barrels, the composition of wines change because of the addition of phenolic compounds and other molecules extracted from the wood. Such compounds include lignins, hydrolysable and condensed tannins, gallic acid, ellagic acid, aromatic carboxylic acids, and various aldehydes.

Ellagitannins (hydrolysable tannins) are among these substances. In oak heartwood they may represent 10% of the dry weight and are responsible for the high durability of this wood (Scalbert, Monties, & Favre, 1988). These compounds occur in important levels in European oak barrels (De Simon, Sanz, Cadahía, Poveda, & Broto, 2006; Masson, Moutounet, & Puech, 1995; Prida & Puech, 2006); they can be hydrolysed and are soluble in model wine solutions (Jordão, Ricardo-da-Silva, & Laureano, 2005), in wines and spirits (Moutounet, Rabier, Puech, Verette, & Barillere, 1989). They possess antioxidant activity (Alañón, Castro-Vázquez, Díaz-Maroto, Gordon, & Pérez-Coello, 2011) and they have an impact on astringency and bitterness (Glabasnia & Hofmann, 2006; Sáenz-Navajas, Fernández-Zurbano, & Ferreira, 2012a, 2012b).

Oak also contains a high level of volatile compounds that have a great impact on wood-matured wine aroma. The main volatile compounds susceptible to migration from oak wood to wine are the *cis* and *trans* isomers of whiskey lactone, furfural and its derived compounds, phenolic aldehydes such as vanillin and syringaldehyde, and volatile phenols such as eugenol, guaiacol, and ethyl- and vinylphenols. In a sensory wine study (Spillman, Pocock, Gawel, & Sefton, 1996), vanillin concentration in white wines was positively correlated with 'smoky' and 'cinnamon' descriptors ($p < 0.05$ and 0.01, respectively) but only loosely associated with 'vanilla' ($p < 0.10$). In red wines, vanillin was associated with 'vanilla' descriptor ($p < 0.05$) but was most strongly associated with 'coffee' descriptor ($p < 0.001$), as well as with 'dark chocolate' and 'smoky' ($p < 0.01$). The descriptor 'vanilla' in red wines was most strongly correlated with the concentration of *cis*-β-methyl-γ-octalactone (whiskey lactone; $p < 0.001$).

The sensory role of aromatic aldehydes, even if they form a major proportion of oak wood volatile compounds (Boidron, Chatonnet, & et Pons, 1988), is still largely a matter for conjecture. Opinion on the sensory impact is largely based on threshold data of individual compounds in non-oaked wines and does not take

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into account the possibility of sensory interactions with other volatiles derived from oak or from microbial activity during the maturation phase. Thus, threshold data (Boidron et al., 1988) suggest that vanillin can have a strong influence on wine aroma, while furfural and 5-methylfurfural have, on their own, no more than a minor impact. However, furfural has been reported to have an important modifying effect on the perception of the aroma of oak lactones in wine (Reazin, 1981). The volatile compounds extraction of oak barrels depends mainly on the quantity of compounds that are potentially extractable, on the contact time between wine and oak wood and on the wine composition. However, compounds extracted by wine from barrels undergo transformations, mainly microbiological ones, which modify the concentration of these substances in wine over time (Spillman, Iland, & Sefton, 1998).

In the course of barrel production, the oak used for barrels must pass through several processing stages important to wine flavour. After being split, the wood is submitted to a drying process, to ensure the mechanical resistance of the barrels. In order to give form to the barrels, oak wood is heated. In cooperage, three types of toasting are used: light, medium and heavy. This stage is considered as having the most important influence on the chemical composition of oak wood. The thermal treatment causes thermodegradation of some components of oak wood, which produces numerous volatile compounds. Furanic compounds are formed through thermal degradation of carbohydrates; volatile phenols come from the thermal degradation of lignin and oak lactones are products of the dehydration of the acids present in wood. Medium toasting corresponds to the maximum synthesis of these volatile compounds (Koussissi et al., 2009).

At present, alternatives to the oak barrel are being looked at to carry out the wine-ageing process. This practise, the addition of alternatives products to the wine, recently was approved and legislated by the European Community (CE 2165/2005 and CE 1507/2006), but in some countries, such as Australia, USA, South Africa, and Chile, this practise has been used for several years. Different shapes of oak wood pieces are used: chips, cubes, powder, shavings, dominoes, and blocks. Factors such as piece size, amount of added wood, and contact time between wood and wine affect the sensory and chemical characteristics of wines (Del Alamo Sanza, Escudero, & De Castro Torío, 2004; Del Alamo Sanza & Nevares Domínguez, 2006; Frangipane, Santis, & Ceccarelli, 2007), especially their wood-related volatile composition (Arapitsas, Antonopoulos, Stefanou, & Dourtoglou, 2004). Up to now, it does not seem very logical to establish an ageing period in barrels or with wood pieces through legislation. Hence, it would be important to know more about the oak wood compounds extraction process in the wine. It is likely that a study of wine volatile and non-volatile composition, along with a tasting assessment, would be a more efficient method to establish the optimum time of contact between wine and oak wood.

Therefore, the objectives of this study were defining the chemical (ellagitannins and volatile composition) and sensory characteristics of wine treated with winewoods representing different toasting methods with the aim to monitor the extraction kinetics of the above compounds during 12 months. The toasting level impact on both volatile, non-volatile compounds and sensory perception was studied in parallel. The relationship between the chemical composition and the sensory assessment of oak wood was also investigated.

2. Materials and methods

2.1. Wood origin and drying conditions

The wood samples were constituted from *Quercus robur* oak species from the same forest located in the Centre region of France. The raw winewoods ($100 \times 11 \times 0.12$ cm) were stored for 24 months in the Tonnellerie Nadalié (Ludon-Medoc, France) seasoning park. Then they were submitted to different toasting procedures, according to the desired final product, using oak fire.

2.2. Red wine ageing in stainless steel tanks with winewoods

Merlot grapes were manually harvested at maturity in the Bordeaux region of France at the end of September 2010. The same day, the grapes were crushed, and some SO_2 was added (5 g/100 kg) during the transfer of the must to 80-hL stainless steel tanks. *Saccharomyces cerevisiae* yeast was added to perform alcoholic fermentation at 25 °C. After the alcoholic fermentation, the temperature of the stainless steel tanks was maintained at 21 °C, in order to initiate spontaneously malolactic fermentation, which lasted for 40–50 days. At the end of fermentation the wine possessed a total phenolic index of 60, a pH of 3.61, 12.8% (v/v) alcohol level and 4.85 g/L tartaric acid.

After malolactic fermentation, the red wine was transferred and kept in 2-hL stainless steel tanks for ageing. Seven different types of winewoods (LT (Light Toast), LT+ (Light Plus Toast), MT (Medium Toast), MT+ (Medium Plus Toast), HT (Heavy Toast), Noisette, Special) were added in separate stainless steel tanks for 12 months (2 ww/h L and 0.24 m^2/ww). Table 1 shows the temperature and the toasting time of every winewood used. For MT, Noisette and Special the same toasting temperature is used. However in the case of Noisette, there is a prolongation of the toasting time, whereas in the case of Special, 30 min before the end of the toasting process a watering process takes place.

For the purpose of our study, two tanks were used for every trial and a tank containing only wine was used as control. During the year of ageing in tanks with winewoods, each red wine was sampled at 1, 2, 3, 6, 9 and 12 months, then the quantification of elagitannins and of aromatic compounds was performed by HPLC–UV and GC–MS analysis, respectively. Sensory analysis was performed in parallel.

2.3. Extraction of volatile compounds

Wine solutions were extracted with dichloromethane. Two-hundred microlitres of a solution of 1-dodecanol as internal standard were added to 50 mL of samples. Three extractions were then carried out using 4, 2, and 2 mL of dichloromethane. The organic fractions were combined and dried on anhydrous sodium sulfate and then concentrated to 500 μL under a nitrogen stream. In all cases, the samples were analysed in duplicate.

2.4. Gas-chromatography analysis

A simple and reliable GC method for quantitative determination of the volatile compounds arising from oak wood was used according to an adaptation of a previous method (Barbe & Bertrand, 1996). An Agilent HP 5890 GC (Hewlett–Packard, Wilmington, DE,) was coupled with a mass spectrometer (HP 5972, electronic

Table 1
Winewood characteristics.

Winewood	Toasting temperature (°C)	Toasting time (h)
LT (Light Toast)	165	2.0
LT+ (Light Plus Toast)	170	2.5
MT (Medium Toast)	180	3.0
MT+ (Medium Plus Toast)	190	3.5
HT (Heavy Toast)	200	4.5
Noisette	180	5.0
Special (MT with watering)	180	3.0