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ORIGINAL PAPER

Chemical and sensory evaluation of wine matured in oak barrel: effect of oak species involved and toasting process

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Abstract The effect of toasting method and forest origin on volatile compounds and ellagitannin concentration extracted into Merlot wine during 1-year barrel maturation is described. Sensory analysis concerning vanilla, spicy, overall woody, astringency, bitterness and sweetness is conducted in parallel. The study is realized in two different experimental designs having only a common parameter, the wine. For the first one, common toasting methods (light, medium) and specific ones (medium toast with watering, medium plus toast, medium plus toast with watering, medium toast with toasted head and Noisette) are chosen. For the second one, the oak from which barrels are made is sourced from Center, Limousin, Allier and Colbert forests in France, from Pennsylvania forest in America, and from Slavonia forest in East Europe. Wine with different characteristics is obtained from the same wine in relation to forest origin and toasting process. Watering process during toasting enhances furanic compounds vanillin and oak lactones extraction, whereas toasting barrel head pieces may lead to eugenol and ellagitannin degradation. Wine in light toast barrel is perceived as less sweet, bitter and more astringent. Ellagitannin concentration is closely correlated to astringency, reflecting the relationship between them, $R = 0.599$, $p < 0.001$. Forest origin induces important changes; wine in American barrels is differentiated from those in Slavonia and French barrels. Ellagitannin concentration in barrels from Slavonia is halfway between French and American, and ellagitannin concentration is related linearly to astringency intensity ($R = 0.811$, $p < 0.001$). Wine aged in Slavonia showed characteristics similar and intermediate to those of the same wine aged in French and American oak woods, suggesting that they are suitable for barrel production of quality wine.

Keywords Merlot · Oak wood · Toasting · Forest origin · Ellagitannin · Volatile compound · Astringency

Introduction

Wooden barrels have been used for well over 2000 years for wine storage and transport. The quality benefits of barrel aging were established early in their use, and wine maturation in wood barrels modifies its smell and taste and reduces its astringency; hence, wine organoleptic properties are improved. Red wine undergoes important modifications, as spontaneous clarification, slow and continuous oxygen diffusion through oak barrel wood pores, and the extraction of many oak wood substances (e.g., aromatic compounds and ellagitannins). Hydrolysable tannins (ellagitannins) are among these substances, and in oak heartwood, they may represent 10 % of the dry weight and are responsible for the high wood durability [1]. The oral sensation imparted by ellagitannins was described as astringent at relatively low threshold concentrations spanning from 0.2 to 6.3 μmol/L, whereas bitterness was strongly dependent on ellagitannin structure and a bitter taste was perceived at threshold concentrations between 410 and 1650 μmol/L [2]. Stark et al. [3] observed that all ellagitannin derivatives induce a smooth and velvety astringent oral sensation at rather low threshold concentrations ranging from 0.9 to 2.8 μmol/L. Recently, Chira et al. [4] observed

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that ellagitanin levels in a model wine solution explained 45 % of the total astringency variance.

The main volatile compounds susceptible to migration from oak wood to wine are the *cis* and *trans* isomers of β-methyl-γ-octalactone, furfural and its derived compounds, phenolic aldehydes such as vanillin, syringaldehyde and volatile phenols such as eugenol, guaiacol, and ethyl- and vinylphenols [5]. Phenolic aldehydes such as vanillin contribute to olfactory characteristics of wine with notes of vanilla, coffee, dark chocolate and smoke, with a synergistic effect of whiskey lactone [6]. If aromatic aldehydes form a major proportion of oak wood volatile compounds [7], their sensory role is still largely a matter for conjecture. Opinion on the sensory impact is largely based on threshold data of individual compounds in nonoaked wine and does not take into account the possibility of sensory interactions with other volatiles derived from oak or from microbial activity during the maturation phase. Thus, threshold data [7] 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 oak lactones aroma [8]. For example, Reazin [8] observed that 1 ppm of oak lactone solution in 80°P (Proof) spirits has an odor described as oak wood with a trace of coconut. Furfural was found to affect the organoleptic properties of oak lactone, levels of 10 ppm furfural and 1 ppm oak lactone imparted a pleasant wood, caramel or vanilla-like odor. As the levels of furfural are increased, the wood aroma is decreased, while the caramel or vanilla-like odor of the furfural is accentuated.

The extraction of these compounds depends mainly on the pool of potential extractible compounds originally present in the barrel wood. Oak wood chemical composition is influenced by two groups of main factors, on the one hand, the oak species, the geographical origin, and the silvicultural treatment of the tree [9, 10], and, on the other, the wood processing in cooperage, that is, the method used to obtain the staves, the seasoning method (natural or artificial, length, and location) [11, 12], and the oak toasting intensity during barrel's manufacturing [13]. Therefore, these factors are important variables to be considered by the cooper and the winemaker when selecting wood for cooperage.

Traditionally, the oak species most often used in cooperage are *Quercus alba* and some related species, otherwise known as American white oak, and two European species, *Quercus robur* L. (pedunculate oak) and *Quercus petraea* Liebl. (sessile oak). In general, American oak wood is richer than European in low molecular weight phenolic compounds, β-methyl-γ-octalactone, and some volatile compounds, but poorer in ellagitannins. Among European oak woods, *Q. robur* is richer in ellagitannins than *Q. petraea* [14]. Recent studies have demonstrated that both phenolic composition and volatile composition of *Quercus pyrenaica* and *Quercus faginea* species are comparable to chemical data reported for other oak wood species (*Q. robur* and *Q. petraea*) commonly used for barrel making [15–18]. *Quercus pyrenaica*, being an autochthonous oak wood species grown in the Iberian Peninsula, is acquiring great importance in the manufacturing of barrels. Indeed, red wine aged in barrels made of *Q. pyrenaica* wood was highly regarded, and preference was shown for it over the red wine aged in barrels of other oak species [19]. Wine with chips and staves from *Q. pyrenaica* showed higher aromatic intensity, complexity, woody, balsamic and cocoa than American or French ones [19].

At the same time, some factors such as the age of the wood, time and seasoning technique, and the heat treatment of oak wood influence wine chemical composition [20–23]. During heating treatment, a severe wood chemical composition modification is induced. Depending on the degree of toasting, pyrolysis and hydrothermolysis will degrade wood constituents to some extent, not only ellagitannins, which are easily hydrolyzed [15, 24], but also lignins and hemicelluloses will be altered. The degradation of these compounds will contribute to raise the volatile compound level [25]. Thus, toasting will strongly influence the oak wood chemical composition, which in turn will influence wine cooperaging.

Within this work, we had the opportunity to evaluate the influence of geographic origin and toasting level on ellagitannin extractible amounts and on principal oak volatile compounds.

The first target was to examine the influence of common toasting methods (light toast, medium toast) and of specific ones (medium toast with watering, medium plus toast with watering, medium toast with toasted head and Noisette) of the same forest origin on wine qualitative characteristics. Possible implication of volatile and nonvolatile composition on the final quality perception of wine was also examined.

The second target was to monitor the variation of some qualitative characteristics of Merlot wine aged in medium-toasted barrels made of oak wood of four French forests: Centre, Limousin, Allier and Colbert of American forest in Pennsylvania and of East European forest (Slavonia). East European oak wood is traditionally used in local cooperage. This oak wood is of great interest for world barrel making due to its forest resources, whereas its chemical composition has not been investigated. Previous studies for Russian oak (global indices and selected volatile compound data) [26] and for Moldavian oak [14] revealed the prospects of East European oak use for wine and brandy maturation.

To the best of our knowledge, this is the first complete report concerning the oak wood from wood species of Slavonia forest focusing also on the toasting impact on a selection of volatile and nonvolatile compounds and on sensory perception.

Table 1 Toasting time and temperature for each toasting level designation

Barrels	Toasting time (min)	Toasting temperature (°C)
LT (light toast)	56	47 ± 3
Noisette	62	52 ± 3
MT (medium toast)	68	57 ± 3
MT AA (medium toast with watering)	68	57 ± 3
MT TH (medium toast with toasted head)	68	57 ± 3
MT+ (medium plus toast)	68	62 ± 3
MT+AA (medium plus toast with watering)	68	62 ± 3

Materials and methods

Wood origin and drying conditions

The wood samples were made up from two oak species (*Q. robur and Q. petraea*) grown in four French forests: Center (*Q. robur* and *Q. petraea*), Limousin (*Q. robur*), Allier (*Q. petraea*) and Colbert (*Q. petraea*). Colbert is a selection of extra tight grain from Allier forest and Center. These wood samples were compared with others of the same species (*Q. robur* and *Q. petraea*) but of different origin (Slavonia, forests in East Europe) and with samples of different species and origin (American *Q. alba* derived from Pennsylvania forest). The raw staves (100 cm × 11 cm × 0.12 cm) were stored for natural seasoning during 24 months in Nadalié Cooperage (Ludon-Medoc, France) wood yard. Then, they were submitted to different toasting procedures according the desired final product using the traditional way over a wood fire, according the process used at Nadalié cooperage (Table 1). Heartwood staves of French and Slavonia oak of *Q. robur* and *Q. petraea*, before and after toasting, and of American oak of *Q. alba*, after toasting, were also provided by Nadalié Cooperage.

Red wine aging in barrels

Merlot grapes were manually harvested at maturity in Bordeaux region in 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 stainless steel tank. *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 the malolactic fermentation, which lasted for 40–50 days. Then, in January 2011, Merlot wine of 12.92 (% vol), 3.01 g/L H_2SO_4 and 3.74 pH was transferred into oak barrels for aging during 1 year before bottling, and temperature was maintained at 16 °C. For the purpose of our study, three barrels manufactured were used for every trial; wine was pooled for three barrels before bottling. One part of the study was carried out by storing the young wine in 225 L barrels having the same oak wood origin (*Q. robur* and *Q. petraea* from Allier forest) but different toasting methods, namely light toast (LT), medium toast (MT), medium plus toast (MT+), medium toast with watering (MT AA), medium plus toast with watering (MT+AA), medium toast with toasted head (MT TH) and Noisette. The other part of the study was performed by storing the young wine in 225-L medium-toast oak barrels, from six different forests, Centre (Ct), Limousin (Lim), Allier (Al), Colbert (Co), Slavonia forest (Sla) and American (Ao).

During the year of aging, each red wine was sampled at 12 months; then, the quantification of elagitannins and of volatile compounds was made by HPLC–UV and GC–MS analysis, respectively. At the same time, a sensory profile was carried out.

Analysis of wine volatile compounds by gas chromatography

About 200 µl of a solution of dodecan-1-ol as internal standard was added to 50 mL of wine sample. 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. Once volatile compounds extracted, the chromatographic analysis was carried out with an Agilent HP 5890 GC (Hewlett–Packard, Wilmington, DE, USA) coupled with a mass spectrometer (HP 5972, electronic impact 70 eV, eMV = 2 kV). One-microliter sample of organic extract was injected in split-less mode. The working conditions were as follows: column BP21 (SGE) (50 m × 0.32 mm, 0.25 µm); carrier gas helium (pressure: 70 kPa); temperatures: injector, 250 °C; detector, 280 °C; oven, 60 °C for 1 min programed at a rate of 3 °C/min to 240 °C, the final step lasting 40 min; the split-less time was 30 s with a split flow of 30 ml/min [27]. Working in the SIM mode, the following ions were used: syringaldehyde, m/z 182; vanillin, m/z 151; eugenol, m/z 164; guaiacol, m/z 124; β-methyl-γ-octalactone, m/z 99; 5-methyl-2-furfuraldehyde, m/z 110; furfuryl alcohol, m/z 98 and m/z = 83 for the internal standard (dodecan-1-ol). The concentrations of each substance were measured

Table 2 Mobile phase gradient of the HPLC method

Time (min)	Flow rate (µL/min)	% of mobile phase A*	% of mobile phase B*
Initial	300	99	1
1.00	300	85	15
2.00	300	78	22
6.00	300	77	23
10.00	300	76.5	23.5
15.00	300	74.5	25.5
17.00	300	60	40
23.00	300	52	48
26.00	300	1	99
27.00	300	1	99
29.00	300	99	1
32.00	300	99	1

* A, H_2O/HCOOH (99.6/0.4); B, MEOHl/HCOOH (99.6/0.4)

by comparison with calibrations made with pure reference compounds analyzed under the same conditions. The corresponding calibration was made for each compound in concentrations ranges 0–8000 µg/L, and linear regression coefficients between 0.98 and 0.999 were obtained.

Total ellagitannin level determination

The red wine (50 mL) was evaporated under reduced pressure, and the resulting residue was dissolved in methanol (20 mL); then, 4 mL of this mixture was loaded in the hydrolysis tubes for the determination of the total ellagitannin level [28]. The total ellagitannin concentration was determined by the quantification of ellagic acid released during acidic hydrolysis (2 h at 100 °C, 2 N HCl in MeOH). Each sample was analyzed in triplicate, and each reaction mixture was subjected to HPLC–UV using an Hypersil GOLD (50 × 2.1 mm, 1.9 µm) column. The mobile phases used were solvent A [H_2O/HCOOH (99.6/0.4)] and solvent B [MeOH/HCOOH (99.6/0.4)]. The solvent gradient is described in Table 2, and the flow rate was set at 300 µL/min. Peak integration of ellagic acid is carried out at 370 nm, whereas that of the internal standard, naphtanol, is performed at 280 nm. These peaks could be confirmed in mass with 301 m/z for the ellagic acid (negative mode of ionization) and 145 m/z for naphtanol (positive mode of ionization).

Sensory analysis

Judges

Twenty judges, 12 women and 8 men, from the Oenology department at the University of Bordeaux took part in the experiment. They were all selected on the basis of interest and availability. They were trained in the employment of scales and descriptors according to ISO 8586-22 [29]. The descriptors selected were related to wood–wine interaction; olfactive descriptors (vanilla, spicy, overall woody) and gustatory descriptors (sweetness, astringency and bitterness). Before the official evaluation, judges attended 16 training sessions over a period of 2 months. This training period included a first general phase a second and a third specific training phase [30]. The general phase was dedicated to the recognition of sensations and aromas perceived. Aqueous solutions of vanillin (20 µg/L), eugenol (60 µg/L), furfurylthiol (0.8 ng/L) and oak wood chip (5 g/L medium toast) were proposed for vanilla, spicy and overall woody character, respectively. Aqueous solutions of quinine sulfate (0.25 g/L), aluminum sulfate (3 g/L) and sucrose (4 g/L) were proposed to set bitterness, astringency and sweetness. Samples were presented, and participants were instructed to identify the solutions as sweet, bitter, astringent, spicy, overall woody or vanilla. All participants correctly identified all solutions.

During the second phase (eight sessions), the judges have been trained to evaluate the descriptors: sweet, bitter, astringent, spicy, woody or vanilla in various concentrations. For their improvement, scaling training (ranking of solution according to concentration of descriptor) was used. Fourth sessions were used for the olfactory attributes and four for the gustatory attributes. After this second phase, the discriminative ability of participants was assessed.

The third phase (seven sessions) was allocated to familiarize the judges with the intensity scale used (0–7). The first two sessions have been dedicated to the overall assessment of the descriptors of interest. Oak wood chips of different concentrations (0–8 g/L) and of different toast (light, medium, high) were added in model wine solutions in order to determine the repeatability of judges from one session to another. For the last five sessions, oak wood chips of different concentrations (0–8 g/L) and of different toast (light, medium, high) were added in red wine. After these last sessions, the judges became familiar with intensity rating of spicy, woody, vanilla, sweetness, bitterness and astringency using a 0–7 point scale.

In the formal sessions, the panelists were provided with 30 mL of wine in coded standard clear wine glasses, covered with a watch glass to minimize the escape of volatile components, and coded with random three-digit numbers. Assessment took place in a standard sensory-analysis chamber, equipped with separate booths, and with a uniform source of lighting, absence of noise and distracting stimuli, and ambient temperature between 19 and 22 °C.

Wine was sniffed and tasted. In every session, the expert judges had to start with evaluation of the orthonasal odor (first without moving the glass, then moving it gently), and then, after a short break, they evaluated the gustatory attributes (sweet, bitterness and astringency). For the astringency and bitterness intensity, each note on this scale corresponds to a specific tannin characterization (0 = amorphous, 1 = hollow, 2 = soft, 3 = mellow, 4 = slight astringency, 5 = tannic, 6 = hard, 7 = rough for astringency; and 0 = nul, 1 = very weak, 2 = weak, 3 = mean, 4 = barely strong, 5 = strong, 6 = very strong, 7 = depreciative for bitterness) [31].

Data analysis

Statistical data analysis was performed using the analysis of variance (ANOVA) of Statistica V.7 software (Statsoft Inc., Tulsa, OK). All the chemical analysis is based on three repetitions, whereas the sensory analysis is based on two repetitions. Tukey's HSD and Duncan's tests were used as comparison tests when samples were significantly different after ANOVA ($p < 0.05$) for chemical and sensory analysis, respectively. Principal Component Analysis (PCA) was used to examine any possible grouping of samples according to forest origin and toasting process. Principal component analysis (PCA) was performed on the correlation matrix using the attributes that differed significantly by ANOVA. Pearson's correlation analysis was used to investigate relationships between chemical composition and sensory perception.

Results and discussion

Oak wood extractible composition

Among oak wood extractives, ellagitannins and volatile substances were chosen to be quantified in each oak sample using HPLC and GC–MS technique. The following wood volatile compounds were studied: furanic aldehydes, furfural and 5-methylfurfural, the two isomers of methyl-γ-octalactone, *cis* and *trans* (commonly known as oak lactones or whiskey lactones); the volatile phenols guaiacol, eugenol; and vanillin.

Toasting process effect

Furfural derives from pentoses, which are the main constituents of hemicelluloses. In general, during the toasting process, some oak wood components such as hemicelluloses, which are the most thermosensitive polymers in wood, are preferentially degraded, thus leading to the formation of furfural [29]. This degradation process contributes significantly to make furfural the main furanic derivative in toasted oak wood. Figure 1 illustrates that furanic derivative concentration was dependent on toasting process. Wine aged in MT AA and MT+AA barrels presented the highest concentrations, meaning that watering process during toasting enhances furanic compound extraction. MT+ sample presented the less important levels, suggesting that increasing the toasting temperature up to 62 °C either may degradate these compounds or in this type of toasting furanic aldehydes conversion into their corresponding alcohols could surpass its extraction in the wine [5].

Relating to phenolic alcohols, their extraction is dependent on toasting method. MT along with MT AA sample presented the highest eugenol levels, whereas LT sample had lowest levels. The phenolic alcohols, eugenol, guaiacol, 4-methyl guaiacol, come from lignin thermodegradation. Its formation arises from the second phase of heating after the phenolic aldehydes, and consequently, these compounds are found in higher quantities when the toasting process is realized at 57 °C. Suggesting that toasting up to 62 °C (case of MT+ and MT+AA) results in eugenol degradation. Wine matured in MT barrels present more eugenol compared to wine matured in MT TH barrels, meaning that toasting the barrel head pieces (MT TH) may lead to eugenol degradation.

Cis-oak lactone and *trans*-oak lactone are products of the dehydration of 2-methyl-3-(3,4-dihydroxy-5-methoxybenzo)-octanoic acid, and they are among the most important volatile substances coming from wood. The *cis* isomer is regarded from the sensory aspect as among the most important oak wood volatile components that are extracted into wine during barrel aging, as its perception threshold is 46 µg/L while the *trans* isomer has its threshold at 460 µg/L [32]. For the studied samples, the *cis* isomer was the predominant compound in the wine with concentrations above its perception threshold. Toasting process affects significantly lactone levels, and LT barrels released more oak lactones to wine than the toasted barrels, probably due to the thermodegradation of these heat-sensitive compounds or their loss by volatilization when the oak wood is subjected to high temperatures or even charring [4]. Both MT AA and MT+AA barrels liberate more lactones than MT and MT+, meaning that watering process during toasting may enhance lactones extraction. Previous studies using oak wood alternatives [4, 30] have also highlighted the importance of watering process during toasting in lactones accumulation, suggesting that the formation of β-methyl-γ-octalactone precursors is enhanced by the watering process during toasting. Previous studies [33, 34] showed that oak lactones are produced under strong acid hydrolysis and pyrolysis conditions. Pérez-Prieto et al. [35] found that oak lactone concentrations increased during 270 days of barrel maturation and then continued to increase during the first 180 days of bottle storage, that is, after removal from oak contact. The generation of additional quantities of oak lactone during cooperage, bottle storage, led to the realization that oak lactone occurs in oak wood in both free and

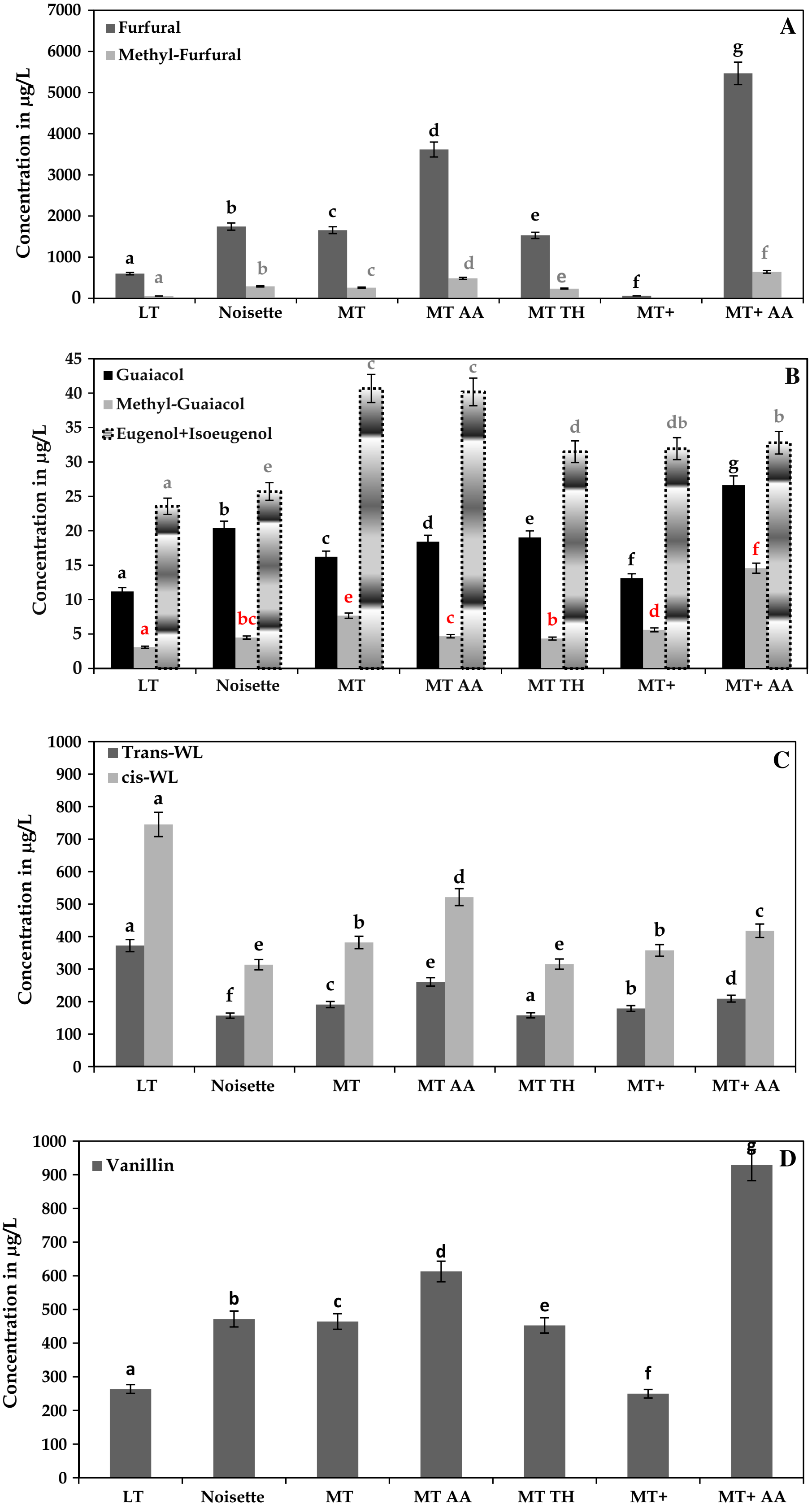

Fig. 1 Quantitative evaluation of furanic compounds (**A**), volatile phenols (**B**), lactones (**C**) and vanillin (**D**) of wines aged in barrels representing different toasting processes: light toast (LT), medium toast (MT), medium toast with watering (MT AA), medium toast with toasted head (MT TH), medium plus toast (MT+) and medium plus toast with watering (MT+AA). The *bars* represent the mean values of three repetitions; *error bars* show the standard deviation; ANOVA toasting effect for wine samples; *a*, *b*, *c*, *d*, etc., of *each column* show the significant differences between toasting processes (Tukey's test, $p \leq 0.05$)

precursor forms. To date, a number of glycoconjugate precursors of oak lactones have been identified as constituents of oak wood. Masson et al. [34] mentioned the presence of another lactone precursor, the 6′-*O*-gallate derivative of (3*S*,4*S*)-4-*β*-*D*-glucopyranosyloxy-3-methyloctanoic acid (**2**, galloylglucoside), from French oak wood. In a subsequent study, Hayasaka et al. [33] identified four isomers of **2**, as well as (3S,4S)-*cis*- and (3S,4R)-*trans*-3-methyl-4-*O*-*β*-*D*-glucopyranosyloctanoic acid (**3**, glucoside) and (tentatively) 3-methyl-4-O-(6′-O-*α*-L-rhamnosyl)-β-*D*-glucopyranosyloctanoic acid (**4**, rutinoside), in extracts of French and American oak woods. Both 2 and 3 have been shown to produce oak lactone under strong acid hydrolysis and pyrolysis conditions. The influence of watering during toasting on oak lactones accumulation should be further explored.

Among phenolic aldehydes, vanillin considered to have the most important influence on wine aroma. Vanillin is primarily formed in wood during the toasting process, and its concentration in wood is considered to reflect the intensity of the toasting process. In general, as it can be seen in Fig. 1, Noisette barrel elicit more vanillin than MT and LT barrel with the latter extracting less than the former, suggesting that a toasting temperature between LT and MT improves vanillin extraction. Medium toast with watering (MT AA and MT+AA) extracts more vanillin than MT, indicating that watering process influences vanillin extraction.

Forest origin effect

Taking into consideration that the variations among individuals from the same species and from the same forest origin are important, species comparison remains statistically significant because each species has its own chemical profile in terms of aroma compounds and tannins [17, 36]; a comparison among forest origins can be made. Thus, if we compare wine aged in Ao oak barrels with those aged in East European and French oak barrels, it can be seen that, in general, the concentration of furfural was highest in the latter, with the exception of the Ct sample (Fig. 2). Particularly, *Q. petraea* of Co forests had higher extractible furanic compounds than white oak *Q. alba.* Slavonia oak wood had an intermediate position between the two French oak Al and Co. *Q. robur* and *Q. petraea* of Ct forest presented at trace level these compounds. Such behavior may be due to different effects on these compounds by the wood: immediate in Co barrels and gradual in Ct barrels.

Phenolic alcohols concentration is affected also by forest origin, American oak wood extracted less guaiacol but elicited significantly more eugenol comparing to French and Slavonia oak. In a comparison of French samples, Ct (*Q. robur* and *Q. petraea*), Al (*Q. petraea*) and Co (*Q. petraea*) contained higher levels of eugenol than Lim (*Q. robur*). This observation is suggesting that *Q. robur* oak extract less eugenol than *Q. petraea.* This result was in agreement with other studies that compared extractives from *Q. robur* and *Q. petraea* stavewood [11].

In the matter of lactones, American white oak wood is richer in *cis*-oak lactone than either *Q. robur* or *Q. petraea*. Maturation in Slavonia oak resulted in the lowest concentrations of *cis*-oak lactone. Ct and Al oak produced intermediate amounts of *cis*-oak lactone, with the former providing more than the latter. The finding that the Ao barrels imparted significantly higher levels of *cis*-oak lactone than French barrels is in accordance with other reports. Guichard et al. [37]. found that brandies matured in American white oak (*Q. alba*) barrels contained a higher concentration of oak lactone than did those matured in barrels made with *Q. robur.* Similarly, other authors [38] have reported American white oak wood extracts to be richer in *cis*-oak lactone than either *Q. robur* or *Q. petraea* extracts. However, wine aged in Lim barrels had significantly lower concentrations of *cis*-oak lactone than those matured in Ct, Al and Co barrels. The richness of the aromatic extractives such as *cis*-oak lactone and eugenol in *Quersus petraea* oak (e.g., Al and Co) might be one of the reasons for these oaks being traditionally favoured over Limousin oak for wine maturation.

With regard to vanillin, East European (Slavonia) wood held the intermediary place between American and French oaks according to their vanillin levels. Notably, Al sample presented the highest concentrations, followed by Sla, Lim, Co, Ao and Ct.

Total ellagitannin concentration

Toasting process effect

The ellagitannin level in each sample was determined by HPLC–UV. In a first approach, the total ellagitannin level was estimated by the determination of the amount of ellagic acid released after acidic hydrolysis [39]. During this reaction, each ellagitannin monomer or dimer released one molecule of ellagic acid. The hydroalcoholic and slightly acidic (i.e., pH ~ 3–4) wine solution enables the solid–liquid extraction of these ellagitannins. The total ellagitannin level, expressed as milligram per L of released ellagic acid of wine, revealed a large diversity of concentrations ranging from 10 to 35 mg of released ellagic acid/L of wine. Figure 3 indicates that ellagitannin concentration is strongly dependant on toasting method. Light toasted presented highest ellagitannin concentration comparing to medium toasted. Noisette barrels being toasted 6 min less than medium toasted and at a lower temperature, elicited more elagitannins. Such differences were expected since ellagitannins undergo thermolytic degradation during

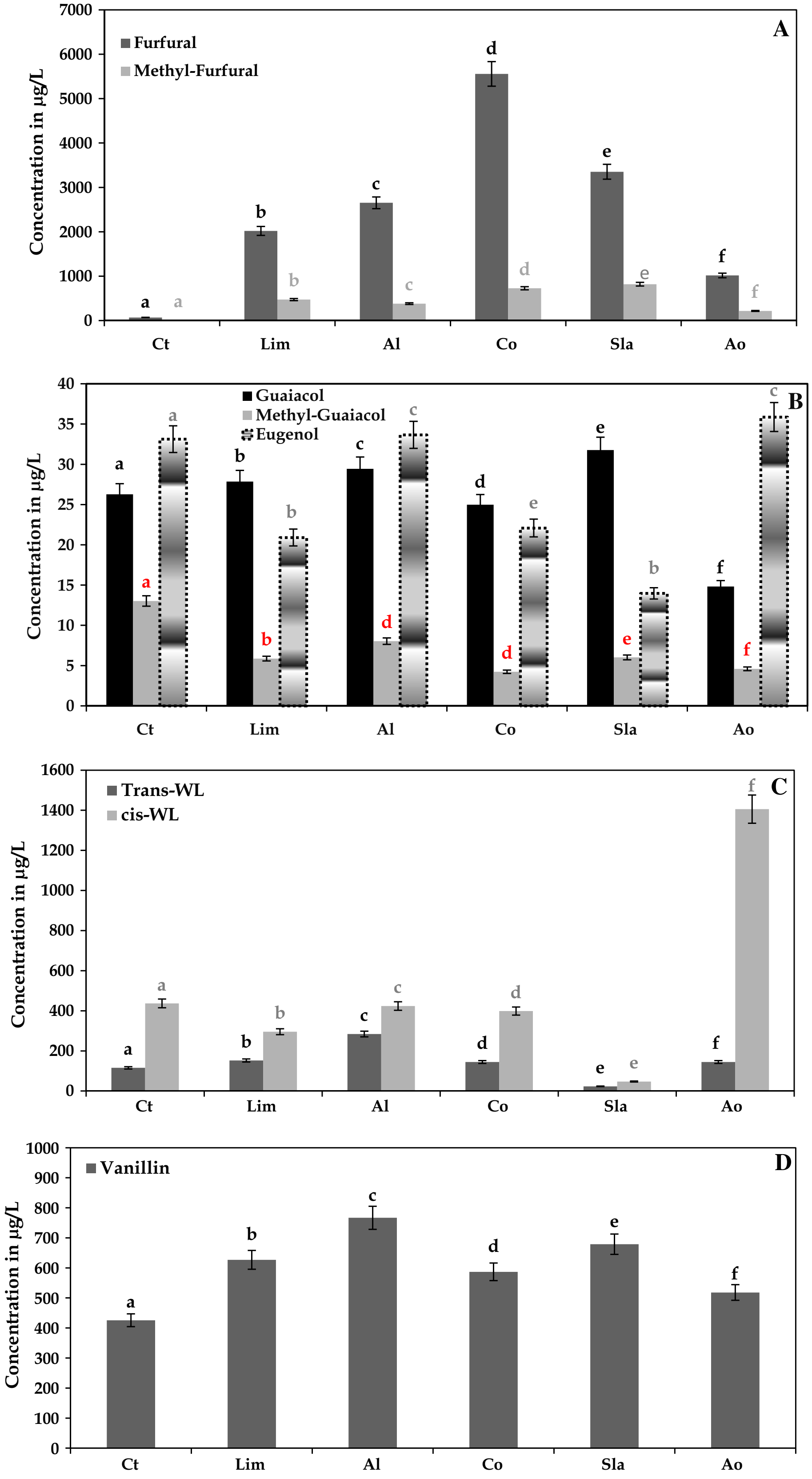

Fig. 2 Quantitative evaluation of furanic compounds (**A**), volatile phenols (**B**), lactones (**C**) and vanillin (**D**) of wines aged in barrels representing different forest origins: Centre (Ct), Limousin (Lim), Allier (Al), Colbert (Co), Slavonia forest (Sla) and American (Ao). The *bars* represent the mean values of three repetitions; *error bars* show the standard deviation; ANOVA forest origin effect for wine samples; *a*, *b*, *c*, *d*, etc., of each column show the significant differences between forest origins (Tukey's test, $p \leq 0.05$)

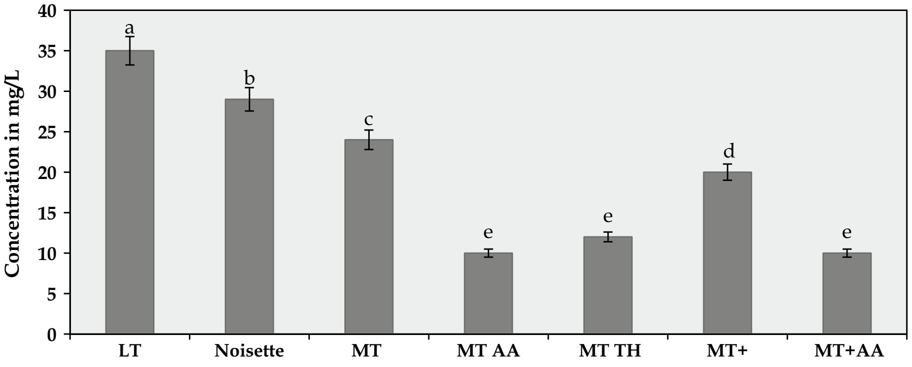

Fig. 3 Ellagitannin levels of wines aged in barrels representing different toasting processes: light toast (LT), medium toast (MT), medium toast with watering (MT AA), medium toast with toasted head (MT TH), medium plus toast (MT+) and medium plus toast with watering (MT+AA). The *bars* represent the mean values of three repetitions; *error bars* show the standard deviation; ANOVA toasting effect for wine samples; *a*, *b*, *c*, etc., of each column show the significant differences between toasting processes (Tukey's test, $p \leq 0.05$)

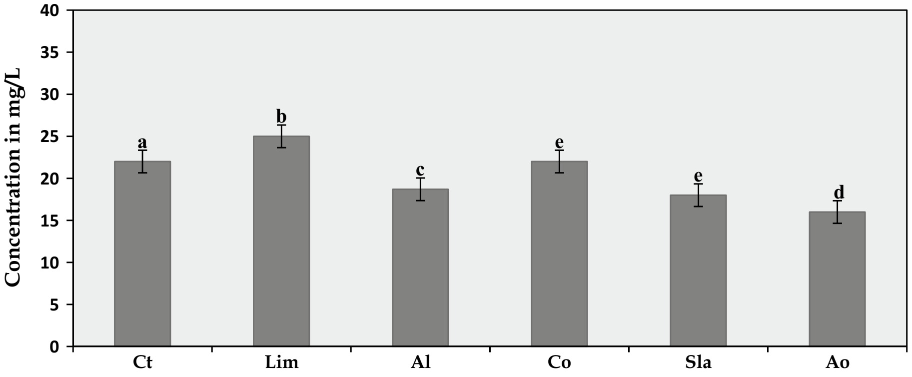

Fig. 4 Ellagitannin levels of wines aged in barrels representing different forest origins: Centre (Ct), Limousin (Lim), Allier (Al), Colbert (Co), Slavonia forest (Sla) and American (Ao). The *bars* represent the mean values of three repetitions; *error bars* show the standard deviation; ANOVA forest origin effect for wine samples; *a*, *b*, *c*, *d*, etc., of each column show the significant differences between forest origins (Tukey's test, $p \leq 0.05$)

toasting process [30, 36]. More precisely, gallic acid is so extremely sensitive to heat treatment that its content systematically quickly decreases in the wood with the duration of the toasting. Moreover, wine aged in barrels that were submitted a watering process during toasting (MT AA and MT+AA) as well as wine matured in barrels with toasting head pieces (MT TH) presented lower ellagitannin concentration. Thus, not only the pyrolytic toasting stage diminishes the quantity of these compounds but also the watering process. This observation confirms previous studies [30] that have showed the watering impact during toasting on ellagitannins levels of oak winewoods.

Forest origin effect

The significant post hoc comparisons among forest origins are shown in Fig. 4. Wine in French pedunculate oak barrels (Lim) was richer in ellagitannins, followed by wine matured in French oak Ct (*Q. robur and Q. petraea*), Co (*Q. petraea*), Al (*Q. petraea*). Sla (*Q. robur and Q. petraea*) oak barrels did not present significant differences with respect to French Al barrel. Ao barrels (*Q. alba*) showed the lowest ellagitannin levels. Ellagitannin concentration in the Slavonia barrels is found to be halfway between French *Q. robur,* French *Q. petraea* and the American *Q. alba*. This may be explained by the different manners of ellagitannin release by different geographical wood origin [40, 41].

Sensory evaluation

One-way ANOVA of the initial raw sensory data (from all assessors) revealed that the six attributes studied (spicy, woody, vanilla aroma, sweetness, bitterness taste and astringent mouthfeel) were significant at $p \leq 0.05$ in wine

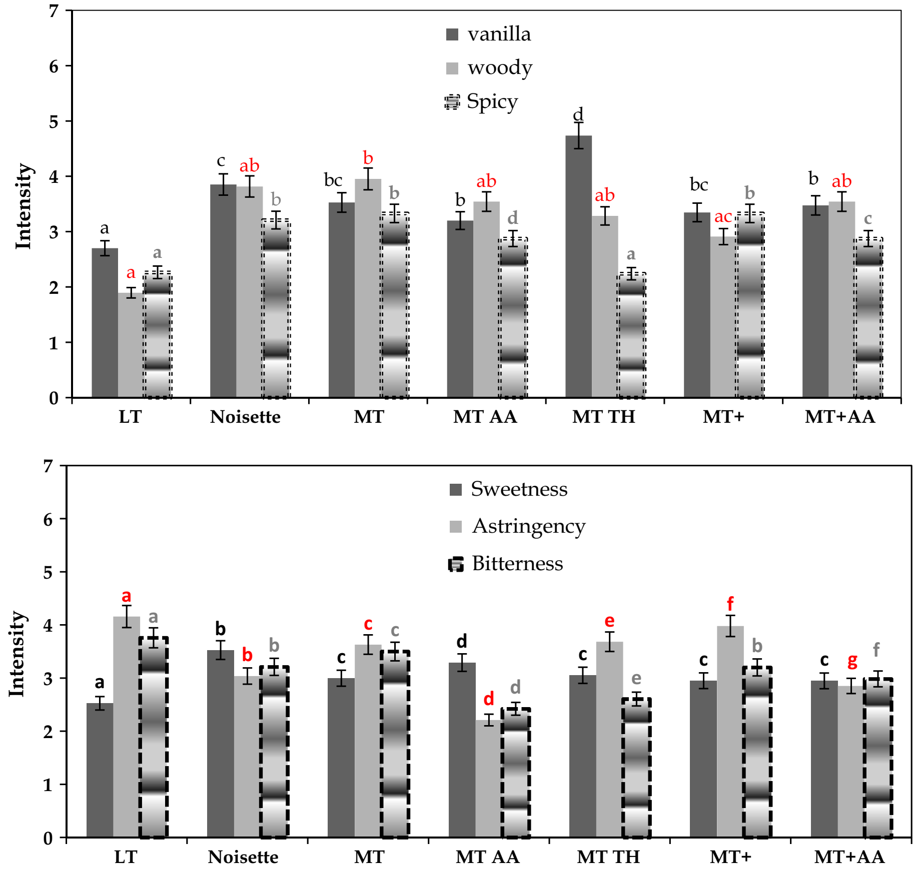

Fig. 5 Sensory evaluation of wines aged in barrels representing different toasting processes: light toast (LT), medium toast (MT), medium toast with watering (MT AA), medium toast with toasted head (MT TH), medium plus toast (MT+) and medium plus toast with watering (MT+AA). The *bars* represent the mean values of two repetitions; *error bars* show the standard deviation; ANOVA toasting effect for wine samples; *a*, *b*, *c*, etc., of each column show the significant differences between toasting processes (Duncan's test, $p \leq 0.05$)

differentiation according to barrels toasting method and forest origin. Figures 5 and 6 show the average intensities of each gustatory and olfactory attribute.

Toasting process effect

Wine aged in MT TH and Noisette barrels were characterized with more intense vanilla aroma than the other wine. Toasting barrel head pieces increase vanillin extraction and in parallel vanilla smell. Vanillin is the principal marker of vanilla and along with lactones can be regarded as direct contributors and/or possible enhancers of this descriptor [6]. Regarding, overall woody aroma, significant differences were found between LT and MT wine, whereas among the different medium toast processes (Noisette, MT, MTAA, MT+, MT+AA) judges found slight differences. Wine contact with Noisette and MT barrels led to an increase in spicy aromas, but when wine is in contact with LT barrels, spicy flavor is slightly reduced. In parallel, LT sample presented less eugenol concentration, and pure eugenol is described as clove-like [42]. After tasting the wine, the sample matured in LT barrels was perceived less sweet and at the same time bitter and more astringent than the other samples. This last observation may be attributed to their higher ellagitannin levels. The wine aged in MT AA barrels was characterized as less astringent and bitter, showing that watering process during toasting influences not only the levels of hydrosoluble tannins but also wine sensory profile.

Forest origin effect

As far as forest origin is concerned, Merlot wine in Sla and Ao barrels released more vanilla, less woody and a medium spicy intensity compared to Merlot wine in French oak. Notably, wine in *Q. petraea* barrels (Co and Al) was characterized with less vanilla notes compared to *Q. alba* (Ao). However, wine in *Q. petraea* (Co and Al) was identified as having more vanilla notes than wine aged in Ct (*Q. robur* and *Q. petraea*) and Lim (*Q. robur*) barrels. Wine in Co and Ct barrels revealed a deep spicy note, and wine aged in Ao and Sla barrels was perceived sweeter than the wine in European barrels. In terms of astringency, the attributed notes for all the samples fluctuated between three and four which correspond to mellow tannins. Particularly, Ao and Sla were noted with lower intensities of astringency than the wine in European oak. This finding can be explained by their lower ellagitannin levels. In establishing order of preference, there was no clear favorite wine depending on

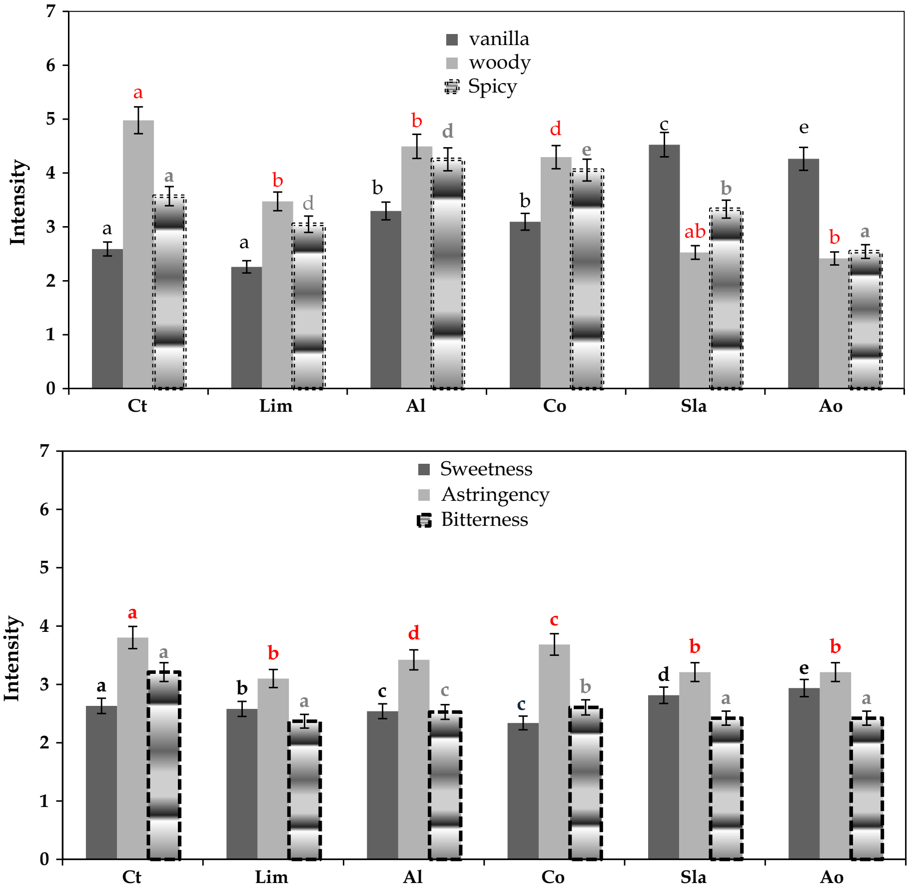

Fig. 6 Sensory evaluation of wines aged in barrels representing different forest origins: Centre (Ct), Limousin (Lim), Allier (Al), Colbert (Co), Slavonia forest (Sla) and American (Ao). The *bars* represent the mean values of two repetitions; *error bars* show the standard deviation; ANOVA forest origin effect for wine samples; *a*, *b*, *c*, etc., of each column show the significant differences between forest origins (Duncan's test, $p \leq 0.05$)

forest origin and toasting method. Wine aged in Slavonia or French oak woods alternated in first place together with wine aged in American oak wood, according to the personal preference of each judge. There was only one agreement: The wine aged in Ct barrels at 12 months of aging was always the woodiest one, whereas the wine aged in Lim barrels presented the lowest scores in vanilla.

Toasting and origin effect

To develop and examine in detail the above observations, to compare statistically the two studied effects, the results of one-way ANOVA results are given in Table 3. One can observe high critical F values (F specifies the significance level of p value) for all the studied variables among forest origins and toasting process. The F values in one-way analysis of variance are used to assess whether the expected values of a quantitative variable within several predefined groups differ from each other. More particularly, here, it is used to compare the two effects, to assess whether forest origin or toasting process is on average superior, or inferior, to the others versus the null hypothesis that both effects yield the same mean response. More the F value is high, more the effect is important. Thus, they are some parameters that are influenced more by toasting process than by forest origin. Furfural, guaiacol, methyl guaiacol, vanillin and ellagitannins are more influenced by toasting than by forest origin. Whiskey lactone and eugenol are more influenced by forest origin increased from *Q. robur* (Lim) to *Q. petraea* (Co and Al) to *Q. alba* (Ao). In relation to French and American oak wood *Q. robur* and *Q. petraea* of Slavonia oak wood produce a very low proportion of these compounds.

With regard to sensory analysis, judges perceived that vanilla aroma, bitterness taste and astringency sensation are greatly influenced by toasting process, whereas overall woody, spicy aroma and sweetness are considerably affected by forest origin. Generally, vanilla aroma increases from wine matured in LT barrels to MT and finally to MT TH barrels (Fig. 5). Astringency intensity builds up almost linearly from medium toast with watering barrels (MT AA, MT+AA) to Noisette, to MT, to MTTH, to MT+ and finally to LT. Regarding forest origin, one can see (Fig. 6) spicy and overall woody notes grew from *Q. alba* (Ao), to *Q. robur* (Lim) and finally to *Q. petraea* (Al and Co). Barrels made from Slavonia oak wood have an intermediary place between American oak and French oak.

Table 3 ANOVA significance values studied effects by one-way variance analysis

	Variable	Effect of toasting		Effect of forest origin	
		F values	*p* values	*F* values	*p* values
Chemical data	Furfural	9,387.54	<0.000	2,664.73	<0.000
	Methyl furfural	183,157	<0.000	506,267	<0.000
	Trans-WL	1,400.45	<0.000	84,200	<0.000
	cis-WL	1,258.72	<0.000	1,453,793	<0.000
	Guaiacol	22,321	<0.000	1,543.910	<0.000
	Methyl guaiacol	14,720.5	<0.000	13,728.1	<0.000
	Eugenol + isoeugenol	417.85	<0.000	3,116.8	<0.000
	Vanillin	164,579	<0.000	14,216	<0.000
	Ellagitannins	512.60	<0.000	226.84	<0.000
Sensory data	Vanilla	55.16	<0.000	37.604	<0.000
	Woody	59.908	<0.000	141.93	<0.000
	Spicy	23.171	<0.000	211.84	<0.000
	Sweetness	26.11	<0.000	1,301.7	<0.000
	Astringency	79,038	<0.000	150.10	<0.000
	Bitterness	4,555	<0.000	104.36	<0.000

The relationship between the compounds

In order to visualize the above results to see if the studied quantified compounds are correlated with one another and if they can be separated according to forest origin and toasting method, PCA was performed. Figure 7A, B summarizes these correlations. The weightings on the principal components suggest that principal components may be interpreted as representing either (A) variables influenced by toasting process, or (B) variables influenced by oak wood origin. These suggested interpretations of the principal components are provided on the axes of the Figures.

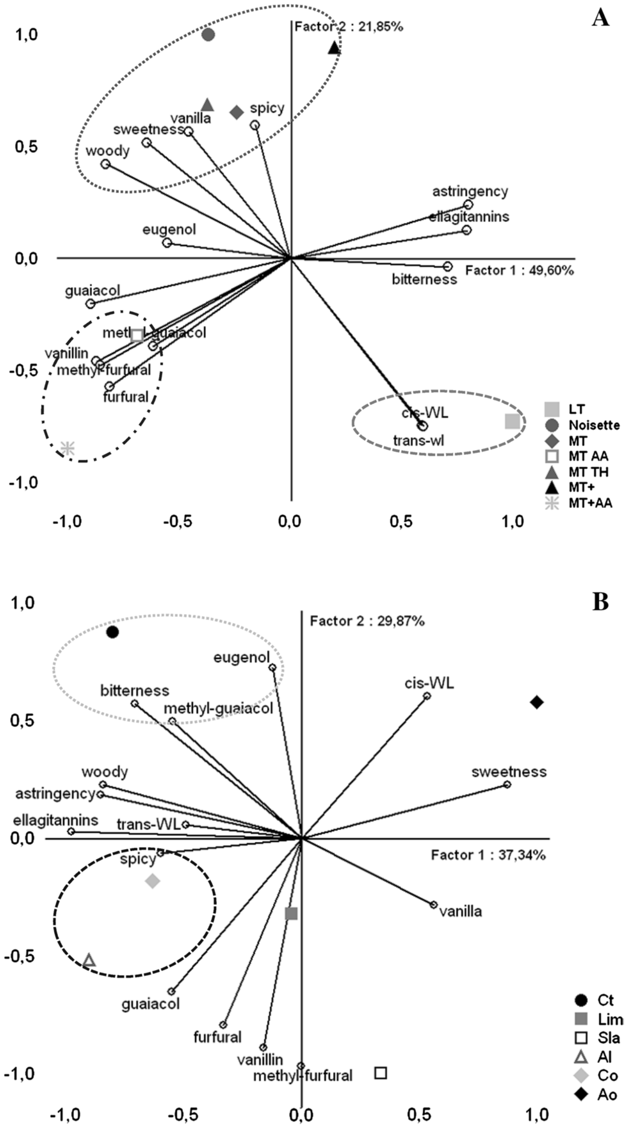

Fig. 7 Projection of composition data on principal component 1 and 2 for Merlot wine emphasis on barrel toasting process effect (**A**), emphasis on oak forest origin (**B**)

In PC1 (49.60 % of variance) and PC2 (21.85 % of variance), wine is clearly differentiated into three groups according to the toasting process: (a) LT barrel (b) Noisette, MT, MT+, MTTH barrels and (c) MTAA, MT+AA barrels (Fig. 7A). Thus, based on chemical and sensory data, the wine sample can be differentiated according to the toasting method as well as the watering process used during toasting. PC1 represents strongly and negatively furanic, guaiacol, methyl guaiacol, eugenol, vanillin compounds, and sweet taste and woody aroma. Astringency and bitterness are represented positively by PC1. Both trans-WL and cis-WL are expressed strongly and negatively by PC2, whereas vanilla and spicy are positively illustrated by PC2. Wine from LT barrels is situated on the positive part of PC1 and showed a good correlation with whiskey lactones. Noisette, MT, MT+ and MTTH samples are grouped with positive PC2 scores and related to overall woody, vanilla and spicy aroma and to sweet taste. Wine from MTAA and MT+AA barrels is scored on the negative side of PC2, showing a good affinity with furanic compounds, vanillin and methyl guaiacol. That may be explained by the similar mechanisms of formation of these substances. Indeed, all studied aromatic aldehydes originate from lignin decomposition; thus, the amplitude of this process influences to almost the same extent each aldehyde formation. These differences in chemical composition, together with the differences in the ultra structure and porosity of wood, contribute to a differentiation of wine in relation to the kind of toasting used during barrel construction.

In Fig. 7B, one can observe very evident differences for practically all of the variables among forest origins. PC1 (37.34 % variance explained) and PC2 (29.87 % variance explained) showed that the main separation was between wine treated with Ao barrels the ones aged in Sla barrels and the ones being in Ct. Wines coming from Al and Co barrels are not very well separated, whereas wine from Lim seems to be isolated from the other samples. Wine from Ao barrels is clearly differentiated from all other samples, with positive scores to *cis*-whiskey-lactone and sweet taste. Wine aging on Ct barrels scored negatively on PC1, showing a higher affinity with loadings for eugenol, methyl guaiacol, ellagitannin compound for bitterness taste and astringency mouthfeel sensation. Finally, wine matured in Co and Al discloses a good relationship with spicy flavor and can be differentiated from the other French barrels. American oak (*Q. alba*) and Slavonia oak have quite a different composition compared with European species.

Ellagitannin concentration is closely correlated to astringency mouthfeel, reflecting the relationship between them; $R = 0.599$, $p < 0.001$ (Fig. 7A), $R = 0.811$, $p < 0.001$ (Fig. 7B). These results confirm ellagitannin importance to astringency perception; higher levels of ellagitannin compounds result in astringency increase.

Conclusion

All of the data obtained reveal that the kind of wood used for wine aging induces important modifications. Wines with different characteristics were obtained from the same wine, after 12 months of aging, in relation to the kind of oak wood used in the barrel-making process as well as in relation to the kind of toasting process used.

Different rates of extraction have been observed, depending on the toasting method used. The toasting method had a significant influence on all the studied variables. A comparison study between forest origin and toasting showed that the toasting affects more considerably furanic, guaiacol, vanillin and ellagitannin concentration. Wine samples can be differentiated according to the toasting method as well as the watering process used during toasting. Watering process during toasting enhances furanic compounds, vanillin and oak lactones extraction, whereas toasting barrel head pieces may lead to eugenol degradation. Toasting head pieces along with watering process decrease ellagitannin concentration. Regarding sensory analysis, vanilla aroma, bitterness taste and astringency sensation are considerably influenced by toasting process. Wine matured in medium toast, particularly in Noisette characterized with more vanilla flavor, and wine being in light toast barrels were perceived less sweet and at the same time bitter and more astringent than the other samples. Astringency intensifies significantly with ellagitannin concentration $R = 0.599$, $p < 0.001$.

The forest origin of wood used induces important changes on all the studied variables ($p \leq 0.05$) but especially on whiskey lactone and eugenol concentration. Slavonia oak wood had an intermediate position between the two French oak Allier and Colbert in extracting furanic compounds. American oak extract more eugenol followed by French and Slavonia. French oak *Q. robur* (Colbert and Allier) extract more eugenol than French oak *Q. petraea* (Limousin). American white oak wood is richer in *cis*-whiskey lactone than either French oak or Slavonia oak. Ellagitannin concentration in the Slavonia barrels was found to be halfway between French *Q. robur,* French *Q. petraea* and the American *Q. alba.* The astringency intensity increased with ellagitannin concentration ($R = 0.811$, $p < 0.001$). Spicy and overall woody notes grew from American oak to Slavonia oak and finally to French oak with wine being in Colbert and Allier barrels characterized more spicy and wine in Centre barrels more woody. Additionally, sweetness decreases from American oak to Slavonia and finally to European. Barrels made from Slavonia oak wood have an intermediary place between American oak and French oak. Moreover, Slavonia oak wood from *Q. robur* and *Q. petraea* can be considered suitable for barrel production for quality wine; wine aged in barrels made of these oak woods showed characteristics similar and intermediate to those of the same wine aged in French and American oak woods.

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Conflict of interest None.

Compliance with Ethics Requirements This research was financially supported by Nadalié cooperage. All procedures followed were in accordance with the ethical standards of the responsible committee on human experimentation (institutional and national) and with the Helsinki Declaration of 1975, as revised in 2008.

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