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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

impact 70 eV, eMV = 2 kV). One-microlitre samples of organic extract were injected in splitless mode. The column was a BP21 column (50 m × 0.32 mm, 0.25 μm; SGE, Ringwood VA, Australia); carrier gas was helium (pressure: 70 kPa); temperatures were: injector, 250 °C; detector, 280 °C; oven, 60 °C for 1 min programmed at 3 °C/min to 240 °C, the final step lasting 40 min; the splitless time was 30 s with a split flow of 30 ml/min.

The compounds were identified by comparing their retention times and mass spectra with those of pure reference standards. Working in 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 (1-dodecanol). The concentrations of each substance were measured by comparison with calibrations made with pure reference compounds analysed under the same conditions. The corresponding calibration was made for each compound, and linear regression coefficients between 0.980 and 0.999 were obtained.

2.5. Red wine sample preparation prior to 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 were loaded in hydrolysis tubes for the determination of total ellagitannin concentration.

2.6. Total ellagitannins

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) as previously described (Peng, Scalbert, & Monties, 1991). Each sample was analysed in triplicate, and each reaction mixture was subjected to HPLC–UV using a Lichrospher 100 RP 18 column (250 × 4.6 mm, 5 μm; Merck, Darmstadt, Germany). The mobile phases used were solvent A [H_2O/H_3PO_4 (99.9/1)] and solvent B [methanol/H_3PO_4 (99.9/1)], and the gradient elution was 0–35% of B in 5 min, 35–45% of B in 25 min and 45–100% of B in 5 min. The flow rate was set at 1 mL/min with detection set at 370 nm.

2.7. Sensory analysis

The sensory assessment was done by a committee of 20 expert judges from the Oenology faculty of Bordeaux. The judges were specially trained in the employment of scales and aroma descriptors according to ISO 8586-2 (2008). The attributes selected were grouped in two families: olfactive descriptors related to wood–wine interaction (vanilla, spicy, overall woody) and gustative descriptors (sweetness, astringency and bitterness). The "overall woody" descriptor was chosen by tasters to describe all olfactory sensations brought about by the wood.

Panellists attended 16 training sessions over a period of 2 months. The training period included a first general phase a second and a third, product-specific training phase. The general phase was dedicated to the recognition of sensations and aromas perceived. Aqueous solutions of vanillin (120 μg/L), eugenol (60 μg/L), furfuryl-thiol (0.8 ng/L) and oak wood chips (5 g/L medium-toast) were proposed for vanilla, spicy and overall woody character, respectively. Aqueous solutions of quinine sulfate (0.25 g/L), aluminium sulfate (3 g/L), and sucrose (4 g/L) were used to set bitterness, astringency and sweetness. During this session, the discriminative ability of participants was assessed. 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. In order to improve panel performance, scaling training (ranking of solution according to concentration of descriptor) was used. Four sessions were used for the olfactory attributes and four for the gustatory attributes. Wine model solutions containing different concentrations of table sugar (0–24 g/L) for sweetness (Jackson, 2009), quinine sulfate (0–30 mg/L) for bitterness, aluminium sulfate (0–4 g/L) for astringency, vanillin (0–320 μg/L) for vanilla, eugenol (0–500 μg/L) for spicy and medium toast oak wood chips (0–10 g/L) for woody were presented to the panel to aid them to discriminate between the different concentrations. After this second phase, the discriminative ability of participants was assessed.

The third phase (seven sessions) was allocated to familiarise 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 (0–10 g/L) and of different toast (light, medium, heavy) were added to 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 to red wine. After the training sessions, the judges were homogenised and became familiar with intensity rating of spicy, woody, vanilla, sweetness, bitterness, and astringency using a 7-point scale.

In the formal sessions, the panellists were provided with 30 mL of wine in coded standard clear wine glasses (ISO-3591, 1997), covered with a watch-glass to minimise the escape of volatile components, and coded with random three-digit numbers. Assessment took place in a standard sensory-analysis chamber (ISO-8589, 1988), 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 .

Wines were sniffed and tasted. In every session the expert judges had to start with evaluation of the orthonasal odour (first without moving the glass, then moving it gently) and then, after a short break they evaluated the perception. The experiment was carried out in duplicate.

2.8. Data analysis

Statistical data analysis was performed using analysis of variance (ANOVA) with Statistica V.7 software (Statsoft Inc., Tulsa, OK). 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 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.

3. Results and discussion

3.1. Oak wood volatile composition

The most potent contributors to overall oak aroma are compounds related to barrel toasting: vanillin, furfural and 5-methylfurfural. The role of furanic compounds can be explained in the same way as for the fruity descriptor: they enhanced the oaky flavour and acted as markers and/or precursors for potent odorants perceived as an oak barrel aroma (Prida & Chatonnet, 2010). Vanillin and *cis*-whiskey lactone can also be regarded as direct contributors and/or possible enhancers of this descriptor. 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 isoeugenol; and the phenolic aldehydes, vanillin and syringaldehyde. The levels of wood volatile compounds extracted were quantitatively different depending on the contact time and type of winewood (Table 2). The control wine, wine without winewoods, possessed lower levels of oak wood aroma compounds. Oak lactones, vanillin as well as guaiacol can be already present in slight levels in wine, but their concentration increases with oak wood contact (Arfelli, Sartini, Corzani, & Fabiani, 2011). Schreier (1979) postulated that furfural in aged wines does not originate exclusively from oak but can also be formed from wines during bottle ageing from hexoses and pentoses in the wine. It is interesting to note that guaiacol and syringol are the only compounds extracted after 12 months. These observations seem to indicate that the other compounds like furfural could be implicated in the aldehyde-generating reactions with flavanols or anthocyanins, producing new colour or colourless pigments (Es-Safi, Cheynier, & Moutounet, 2002).

For short ageing periods, the extraction of furanic compounds from wood is greater than their conversion, and so they accumulate in the wine. Table 2 shows that furfural and 5-methylfurfural reached their maximum concentrations either at 3 or 6 months of ageing. However, for longer ageing periods, the conversion of furanic aldehydes into their corresponding alcohols could surpass their extraction from wood, so that the concentration of these compounds decreases in the wine (Cerdán, Goñi, & Azpilicueta, 2004). Thus, in all the samples, furfural contents were exhausted after 12 months. The wine with LT+ winewoods presented the most important concentrations in furanic compounds (1360 and 278 μg/L for furfural and 5-methylfurfural, respectively) after 3 months of contact, followed by wines with MT^{+} (844 and 48.6 μg/L for furfural and 5-methylfurfural, respectively, after 6 months of contact), following by wines with Noisette (809 and 160 μg/L for furfural and 5-methylfurfural, respectively, after 3 months of contact), wines with Special (783 and 134 μg/L for furfural and 5-methylfurfural, respectively, after 3 months of contact) and MT (682 and 72.4 μg/L for furfural and 5-methylfurfural, respectively, after 3 months of contact). Noisette was toasted 2 h more than MT and presented more furfural and 5-methylfurfural. Moreover, Special (oak wood heated using medium temperature with watering), contained almost two times as much 5-methylfurfural compared to MT, suggesting that along with the prolongation of toasting method, the watering process also has an important impact on furanic compounds.

Among phenolic aldehydes, vanillin was considered to have the most important influence on wine aroma. Just as happens with the furanic aldehydes, for short ageing periods, vanillin accumulates in wine because at the beginning its extraction is high, due to the difference of concentration between the wine and the wood (Cerdán & Ancín-Azpilicueta, 2006; Gómez-Plaza, Pérez-Prieto, Fernández-Fernández, & López-Roca, 2004). Vanillin levels were at a maximum after 9 months of contact in wines with LT+ and Noisette winewoods and after 12 months of contact for the other wines. Its highest concentration was found in the wine with Noisette winewoods (306 μg/L). Similar to furanic compounds the prolongation of MT toasting increases vanillin content. For eugenol and iso-eugenol, an increase in content is observed during maceration for all the wines. Wines with LT and LT+ staves presented the highest concentrations after 12 months.

For phenolic alcohols, the maximum extraction of guaiacol occurred during the first nine or 12 months (Table 2) and the concentration of 4-methylguaiacol was less important throughout the period studied. At the end of the oak period (12 months) the highest values of guaiacol were found for wines with MT (98.5 μg/L) and MT+ (139 μg/L) winewoods. In the other samples, their concentrations were at all times below their thresholds in wine, which Boidron et al. (1988) gave as 75 and 65 μg/L for guaiacol and 4-methylguaiacol, respectively, in red wine.

Both lactones increased linearly in concentration in wine during the oak maturation period (Table 2), the rate of extraction increasing further from 6 to 9 months, meaning that once a portion of wood is wetted, decomposition of lactones occurs rapidly (Boidron et al., 1988; Spillman et al., 1998). After 12 months, the wines matured with LT and LT+ winewoods showed the highest concentration of *cis*-lactone (396 μg/L LT, and 285 μg/L LT+) and *trans*-lactone (209 μg/L LT, and 140 μg/L LT+), whereas the wines with HT winewoods showed the lowest concentrations (10.9 μg/L *cis*-lactone, and 26.9 μg/L *trans*-lactone). The *cis* isomer is regarded as one of the most important volatile components of oakwood that are extracted into wine during barrel ageing. This compound was generally found at concentrations above its perception threshold (46 μg/L) in our study (Wilkinson, Elsey, Prager, Tanaka, & Sefton, 2004). Light-toasted winewoods released more oak lactones to wines than did toasted, probably due to thermodegradation of these heat-sensitive compounds or their loss by volatilisation when the oak wood is subjected to very high temperatures or even charring (Singleton, 1995).

3.2. Oak wood total ellagitannin concentration

The ellagitannin level and composition in each oak winewood were 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. 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 milligrams per litre of ellagic acid released from wine, revealed a large diversity of concentrations ranging from 6.31 to 26.1 mg/L. The HT and the LT wines, respectively, contained the lowest and highest ellagitannins concentrations (Fig. 1). Such differences were expected, since ellagitannins undergo thermolytic degradation during the toasting process (Doussot, De Jéso, Quideau, & Pardon, 2002; Mosedale, Puech, & Feuillat, 1999).

Their extraction rates appeared to be faster during the first 3 months. The same trend was observed by Michel et al. (2011) in red wine aged in contact with oak staves. Fig. 1 shows that for almost all the wines a maximum extraction of ellagitannins is obtained after 2–3 months; after 9 months of contact a decrease is observed. In a red wine aged with LT winewoods a maximum concentration of ~27.3 mg/L was observed after only 1 month; with LT+ winewoods the highest concentration of ~23.7 mg/L was achieved after 2 months; with MT a maximum concentration of ~25.7 mg/L is shown after 3 months; with MT+ the highest levels of ~19.0 mg/L were monitored after 2 months; with HT winewoods a maximum concentration of ~9.16 mg/L was observed after 3 months; with Noisette winewood a maximum concentration of ~25.5 mg/L was obtained after 2 months, and with Special the highest levels of ~16.7 mg/L were monitored after 3 months. In all wines, after 9 and 12 months of contact, the overall concentration of ellagitannins decreased. Particularly, after 12 months, a 10–20% decrease was observed in ellagitannins levels for wines with LT and LT+ winewoods; a 30% loss was monitored for wines with Noisette; a 50–60% reduction was noticed for wines with MT, MT+, HT winewoods; and finally the most important decrease of ~70% was noted for wines with Special winewoods. Wines with Special winewoods not only possessed lower important ellagitannins concentrations but they showed as well the greatest decrease in these compounds during time. Thus not only the pyrolytic toasting stages diminish the quantity of these compounds but also the

Table 2
Evolution of oak volatile concentration (μg/L) in Merlot wine during 12 months (T1 = 1 month, T2 = 2 months, T3 = 3 months, T6 = 6 months, T9 = 9 months, T12 = 12 months).

	Control						LT						LT+					
	T1	T2	T3	T6	T9	T12	T1	T2	T3	T6	T9	T12	T1	T2	T3	T6	T9	T12
Volatile compounds																		
Furanic derivatives																		
Furfural	84.25	84.25	67.9	57.98	50.55	nd	132.4	284.3	339.2	387.5	32.66	nd	392.77	818.75	1358.5	35.31	190.95	nd
5-Methylfurfural	1.12	1.12	nd	nd	0.72	nd	23.5	51.7	69.26	102.5	26.73	nd	99.24	184.11	278.1	52.12	226.13	43.7
β-Methyl-γ-octalactone																		
trans	3.01	3.01	2.92	2.74	3.05	nd	23.01	44.48	57.99	106.8	194.17	208.8	18.32	30.79	33.83	67.75	115.28	140
cis	3.52	3.52	3.24	3.11	4.09	nd	43.5	83.07	104.8	208.4	362.53	396	31.49	59.1	60.63	133.48	228.26	286
cis/*trans* Ratio	0.86	0.86	0.90	0.88	0.75	0.00	1.89	1.87	1.81	1.95	1.87	1.90	1.72	1.92	1.79	1.97	1.98	2.05
Volatile phenols																		
Guaiacol	42.95	42.95	7.04	2.64	21.92	10.85	26.29	35.28	8.76	6.63	22.5	16.18	36.1	33.43	11.59	11.78	32.99	32.3
Methylguaiacol	nd	nd	nd	nd	nd	nd	3.13	3.4	3.72	3.65	4.14	4.96	8.48	11.53	11.4	16.8	22.68	30.7
Eugenol + isoeugenol	1.4	1.4	0.8	0.73	0.78	nd	4.25	6.83	4.65	9	17.5	19.07	3.73	5.83	3.87	7.58	13.7	19.1
Syringol	133.46	133.46	37.75	12.1	76.21	43.23	94.35	121.9	49.55	32.19	88.88	67.81	133.59	126.4	53.53	45	126.08	102
Phenolic aldehydes																		
Syringaldehyde	12.76	12.76	17.04	10.71	10.87	nd	42.76	67.56	82.18	107.2	177.58	202.7	205.1	313.71	317.25	520.31	717.89	862
Vanillin	19.4	19.4	27.58	nd	10.78	nd	34.13	56.79	64.14	68.6	82.7	93.76	108.51	149.69	169.43	199.77	240.96	230
	MT						MT+						HT					
	T1	T2	T3	T6	T9	T12	T1	T2	T3	T6	T9	T12	T1	T2	T3	T6	T9	T12
Volatile compounds																		
Furanic derivatives																		
Furfural	478.57	275.25	682	39.41	187.02	nd	158.4	126.1	317.5	844.9	129.29	nd	104.9	141.6	129.95	284.71	862.1	nd
5-Methylfurfural	30.44	32.76	72.41	28.67	45.61	9.98	8.17	8.52	21.22	48.57	19.64	nd	5.9	8.64	12.52	26.72	53.72	nd
β-Methyl-γ-octalactone																		
trans	7.81	13.87	14.98	25.54	42.47	50.45	4.91	7.9	6.88	12.89	20.31	23.47	4.04	6.49	4.64	7.19	12.39	10.9
cis	13.19	22.48	23.93	42.39	78.57	88.95	7.97	10.52	11.57	20.25	37.81	47	5.83	7.93	7.98	13.89	24.56	26.9
cis/*trans* Ratio	1.69	1.62	1.60	1.66	1.85	1.76	1.62	1.33	1.68	1.57	1.86	2.00	1.44	1.22	1.72	1.93	1.98	2.46
Volatile phenols																		
Guaiacol	72.32	44.92	20.6	27.76	60.4	98.49	44.58	40.25	21.15	32.86	73.09	138.7	45.99	42.99	22.97	37.13	70	111
Methylguaiacol	8.33	10.21	11.41	17.59	23.73	30.43	4.73	6.65	7.66	11.41	17.64	26.63	6	8.61	8.61	16.15	26	31.6
Eugenol + isoeugenol	2.39	3.31	2.16	3.51	6.67	7.27	2.15	2.43	1.79	2.59	4.62	5.43	1.84	1.82	1.09	1.49	2.31	nd
Syringol	265.47	195.54	110.8	133.44	278.46	357.42	169.8	179.7	111.4	157.6	334.75	487.6	174.4	199.03	144.03	214.15	363.62	465
Phenolic aldehydes																		
Syringaldehyde	170.4	271.87	276.6	433.75	646.03	747.16	88.3	128.7	118.5	204.4	305.44	404.2	67.24	95.17	111.47	160.45	227.91	275
Vanillin	92.09	107.28	108.9	130.48	173.76	236.54	45.67	64.73	49.06	66.04	77.47	124.5	36.31	53.38	74.9	64.52	67.3	76.8
	Noisette						Special											
	T1	T2	T3	T6	T9	T12	T1	T2	T3	T6	T9	T12						
Volatile compounds																		
Furanic derivatives																		
Furfural	271.73	608.97	808.5	32.28	313.9	nd	248.3	229	782.5	32.47	62.78	nd						
5-Methylfurfural	57.25	110.81	159.7	7.27	97.59	29.62	47.46	52.65	134	2.73	47.68	25.55						
β-Methyl-γ-octalactone																		
trans	5.78	7.03	7.67	12.50	21.65	20.97	11.59	16.21	16.96	36.26	53.6	74.92						
cis	14.33	19.94	21.78	42.99	77.77	87.83	19.75	30.54	65.63	69.17	112.47	153.5						
cis/*trans* Ratio	2.48	2.84	2.84	3.44	3.59	4.19	1.70	1.88	3.87	1.91	2.10	2.05						
Volatile phenols																		
Guaiacol	37.39	32.16	13.97	16.56	43.06	40.47	37.85	44.1	13.27	20.79	41.34	63.17						
Methylguaiacol	7.92	10.89	10.69	17.91	23.81	33.76	4.86	7.89	7	13.99	17.4	28.16						
Eugenol + isoeugenol	2.11	2.62	1.75	3.02	5.66	6.18	3.58	4.48	2.65	5.36	8.46	10.93						
Syringol	139.68	127.72	67.6	75.82	188.25	146.08	136.2	167.2	65.63	94.31	175.9	212.9						
Phenolic aldehydes																		
Syringaldehyde	229.7	327.4	369.1	672.31	963.56	991.77	157.7	241.2	222.1	415.4	489.83	676.6						
Vanillin	109.74	143.85	166	243.59	305.81	291.86	69.19	107.2	93.82	137.2	128.84	186						

nd Not detected.
LT (Light Toast), LT+ (Light Plus Toast), MT (Medium Toast), MT+ (Medium Plus Toast), HT (Heavy Toast), Special (Medium Toast with watering).

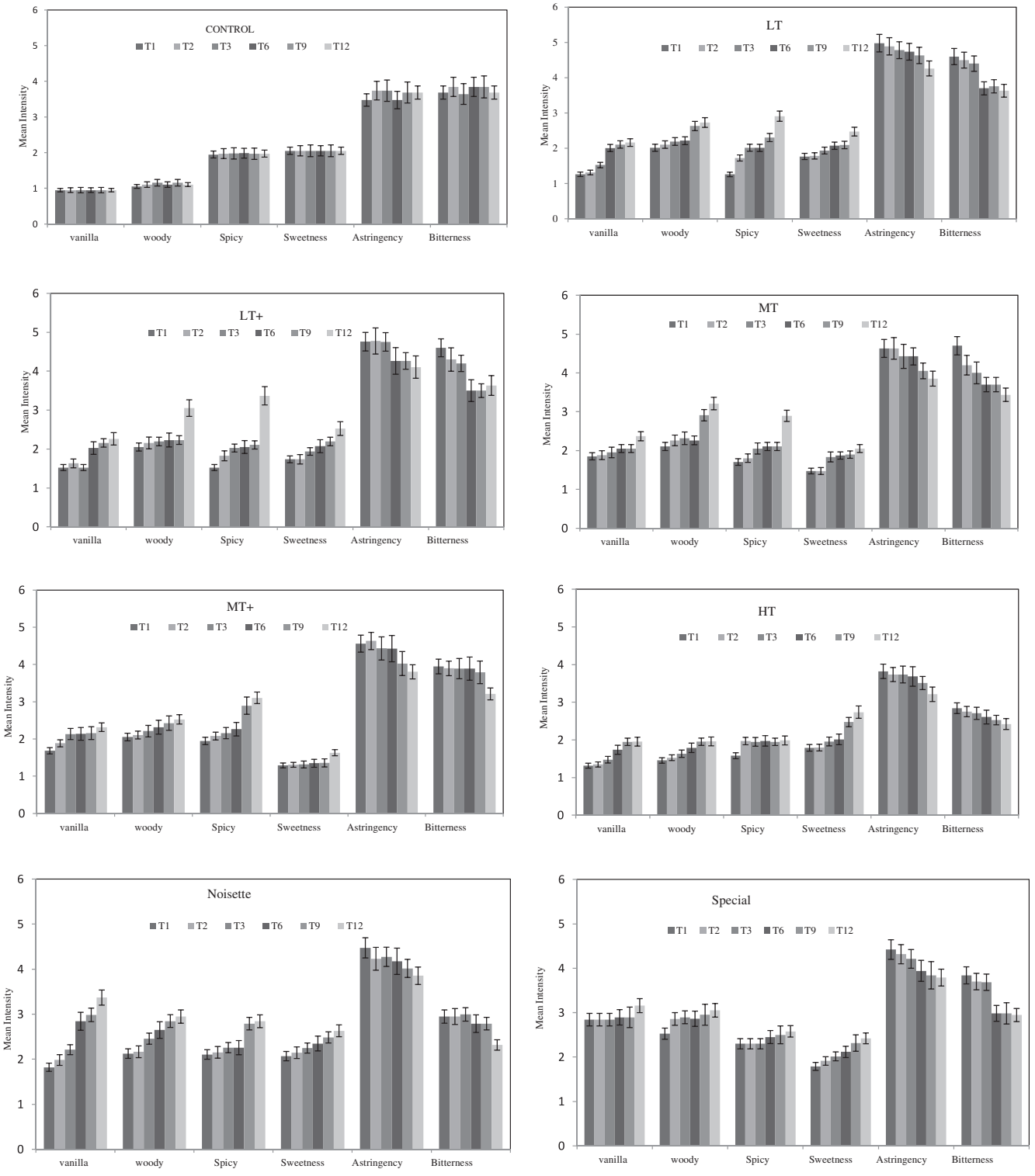

Fig. 1. Evolution of ellagitannins during different contact times (T1 = 1 month, T2 = 2 months, T3 = 3 months, T6 = 6 months, T9 = 9 months, T12 = 12 months). Bars represent the standard deviation.

watering procedure. Moreover, wines with lightly toasted wine-woods extract not only more ellagitannins but also faster than the other samples.

The above decreases during maceration time can be attributed to the high reactivity of ellagitannins toward other wine constituents (Jordão, Ricardo-Da-Silva, Laureano, Mullen, & Crozier, 2008; Quideau et al., 2005). In the first month, the red wine extracts ellagitannins at a rate faster than the rate of the condensation reactions between ellagitannin and the other nucleophilic wine constituents (e.g., catechin, epicatechin, and ethanol). Then, when most of the ellagitannins have been extracted from the first millimetre of the wood, the red wine solution needs to go deeper into

the wood to extract more ellagitannins, consequently at a slower rate.

3.3. Oak wood sensory evaluation

Fig. 2 shows the average intensities of each gustatory and olfactory attribute during the different contact times of the control wine and those of the same wine treated with oak wood for 12 months (T1 = 1 month, T2 = 2 months, T3 = 3 months, T6 = 6 months, T9 = 9 months, T12 = 12 months). Control wine was perceived as less woody with less vanilla flavour, due to the lower levels of oak volatile compounds that were found in this wine. No significant variations were noticed for the control wine during this time. Moreover, Fig. 2 shows that wines treated with toasted oak winewoods were generally more astringent and with stronger bitterness than the control wine. This may attributed to their higher levels of ellagitannins. For all the wines treated with toasted oak winewoods, it is observed that vanilla, spicy and woody characters along with sweetness built up, whereas astringency and bitterness intensity decreased during the contact time. This reduction in the astringency sensation could be caused by the formation of a chemical complex either between wine tannins, polysaccharides and peptides brought out by the oak wood or even due to reactions between ellagitannins and wine molecules. Chemical transformations with time, involving wine native phenolic compounds, can probably contribute to this (Chira, Pacella, Jourdes, & Teissedre, 2011).

Wines with MT+, Special, Noisette and MT winewoods were perceived as more woody; at the same time wines with MT and MT+ presented guaiacol levels above their threshold perceptions. Wines with Noisette and Special were characterised with more vanilla flavour; wines with LT, LT+, MT+, MT and Noisette winewoods were more spicy. In parallel, wines with LT and LT+ winewoods contained the highest levels of eugenol. Wines treated with oak wood were generally more astringent and with stronger bitterness than the control wine. Notably wines with LT were perceived more astringent and bitter, whereas wines with HT winewoods were less astringent and bitter.

Particularly, on a scale from 0 to 7 and from the first to twelfth month vanilla intensity varied from 1.3 to 2.2 for wines with LT winewoods; from 1.5 to 2.3 for wines with LT+ winewoods); between 1.9 and 2.4 for wines with MT winewoods; between 2.1 and 2.3 for wines with MT+ winewoods; between 1.8 and 3.4 for wines with Noisette winewoods; between 1.8 and 3.2 for wines with Special winewoods. It seems that for all the wines vanilla flavour builds up linearly during maceration time ($r^2 = 0.921$ for LT, $r^2 = 0.857$ for LT+, $r^2 = 0.831$ for MT, $r^2 = 0.877$ for MT+, $r^2 = 0.938$ for HT, $r^2 = 0.967$ for Noisette, $r^2 = 0.450$ for Special) probably due to the linear accumulation of vanillin. Vanilla intensity for wines with Special winewoods increased to a lesser extent, compared to the other wines.

Similar to vanilla aroma, spicy character intensifies linearly during the maceration time. Thus from the first to the twelfth month, wines with lightly toasted winewoods demonstrated the highest increase (55–60%) followed by wines with medium toasted winewoods (30–40%), highly toasted winewoods (~20%) and finally by wines with Special (~8%) (medium toast with watering process).

Relating to overall woody aroma, all wines were perceived to be woodier at the end of maceration period (after 12 months). The woody aroma of wines with Special winewoods was more intense after 2 months of contact; afterwards it did not intensify significantly; woody, spicy and vanilla aromas did not fluctuate during the maceration time for wines treated with Special winewood. Thus, the watering process during toasting process influences not only the levels of hydrosoluble tannins but also the wine's sensory profile.

3.4. Correlations between sensory and chemical results

ANOVA revealed that the toasting method has a significant impact ($p < 0.05$) on chemical composition and sensory perception of oak wood extracts. Afterwards correlations between sensory descriptors were assessed. The PCA factor loading plot for the sensory variables and the means for oak volatile compounds and ellagitannins is shown in Fig. 3. This figure is complemented by the Pearson correlation coefficient values presented in Table 3. The first two principal components captured 60.38% of the sample variance. The first PC (Principal Component) is strongly and negatively correlated with sensory descriptors (vanilla, woody, spicy, sweetness) and methylguaiacol and vanillin, whereas it is strongly positively correlated with astringency and bitterness. Concerning the second principal component, it represents strongly and negatively ellagitannins, lactones and eugenol. In parallel, it represents positively the guaiacol. Apparently winewoods can easily be discriminated according to their toasting

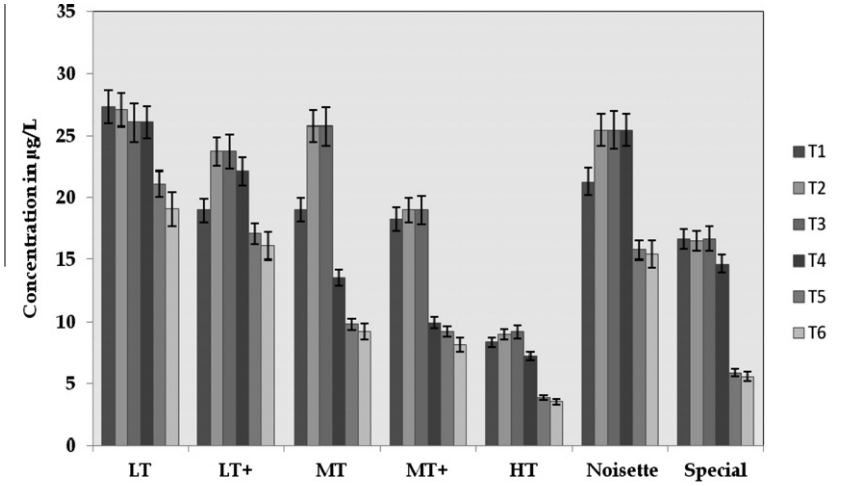

Fig. 2. Evaluation of wine treated with different winewoods during different contact times (T1 = 1 month, T2 = 2 months, T3 = 3 months, T6 = 6 months, T9 = 9 months, T12 = 12 months). Bars represent the standard deviation.

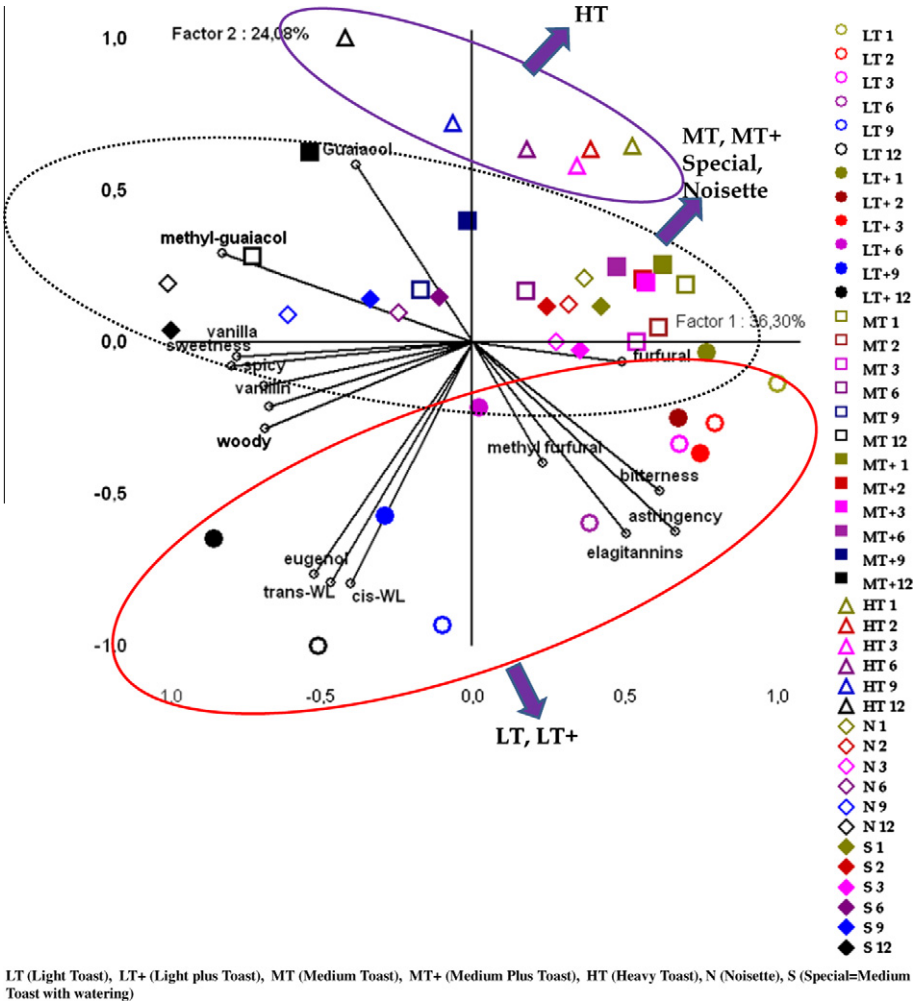

Fig. 3. Principal component analysis (PCA) representation of chemical and sensory data for wine solutions treated with different oak chips during different contact times (T1 = 1 month, T2 = 2 months, T3 = 3 months, T6 = 6 months, T9 = 9 months, T12 = 12 months).

Table 3
Pearson correlations between chemical and sensory data. Marked correlations are significant at $p < 0.05$.

Chemical composition	Sensory descriptor					
	Vanilla	Woody	Spicy	Sweetness	Astringency	Bitterness
Elagitannins	−0.290 NS	−0.085 NS	−0.278 NS	−0.180 NS	**0.828*****	**0.607*****
Furfural	−**0.352***	0.011 NS	−**0.437***	−0.315 NS	**0.430***	**0.393***
5-Methylfurfural	−0.034 NS	0.050 NS	−0.139 NS	0.114 NS	**0.403***	0.264 NS
trans-Whiskey lactone	**0.351***	**0.582****	**0.388***	**0.353***	0.166 NS	0.196 NS
cis-Whiskey lactone	**0.445***	**0.636*****	**0.458****	**0.537****	0.056 NS	0.024 NS
cis/*trans* Ratio	**0.502****	**0.385***	**0.417***	**0.758*****	−0.327 NS	−**0.525****
Guaiacol	0.065 NS	−0.035 NS	0.025 NS	−0.158 NS	−0.431*	−0.207 NS
Methylguaiacol	**0.484***	**0.396***	**0.420***	0.467 NS	−**0.658*****	−**0.546****
Eugenol	**0.428***	**0.636****	**0.462****	**0.314***	0.139 NS	0.154 NS
Syringol	0.028 NS	−0.108 NS	0.004 NS	−0.169 NS	−**0.526****	−0.292 NS
Syringaldehyde	0.684 NS	**0.691*****	**0.601*****	**0.547****	−**0.382***	−**0.373***
Vanillin	**0.595*****	**0.657*****	**0.509****	**0.551****	−0.212 NS	−0.279 NS

Bold values indicate significant differences.
*, **, *** Significant at 5%, 1% and 0.1%, respectively; NS, not significant.

method (light, medium, heavy). LT and LT+ winewoods have similar behaviour pattern; they show a good correlation with ellagitannins, astringency, and bitterness, as well as with the volatile lactones and eugenol. The wines having medium toast winewoods (MT, MT+, Noisette and Special) are grouped together, showing a good affinity with furfural, vanillin, guaiacol, methylguaiacol and at the same time they are correlated to sensory descriptors like vanilla, sweetness, smokey, grilled and spicy. Wines with heavily toasted winewoods are very well separated from wines with light and medium toast and they have a great distance from chemical and sensory studied parameters. Obviously the medium toasted winewoods had the most significant

sensory effect, making wines more sweet, spicy, woody and vanilla. Wines with lightly toasted winewoods scored highest for bitter taste and astringent mouthfeel, possibly due to increased ellagitannins extracted at this toasting intensity.

Each sensory descriptor was correlated with the chemical concentration of the oak wood compounds of interest (Table 3). This procedure allowed us to measure the extent to which sensory and chemical variables are correlated. Based on the correlation analysis, astringency and bitterness intensified significantly with ellagitannins concentration ($r = 0.828$, $p = 0.001$ for astringency and $r = 0.607$, $p = 0.003$ for bitterness). The highest astringency and bitterness were perceived for wines with LT oak wood winewoods (4.98 for astringency and 4.67 for bitterness) containing 27.3 mg/L of released ellagic acid. The above established correlations permit tannin quality characterisation when ellagitannins levels are known. Additionally astringency and bitterness were more perceived in the wines with higher levels of furanic compounds (Table 3). On the other hand, wines with high contents of guaiacol, methylguaiacol, syringol and syringaldehyde were less astringent. This observation is logical as toasting decreases ellagitannins and at the same time increases the concentration of volatile compounds that enhance the oak wood aroma.

An important interaction between sweetness perception and oak volatile compounds was also highlighted. Judges perceived the sweetness perception more intense in wines with higher levels of lactones, eugenol and vanillin compounds. The levels of these compounds are correlated positively with the perceived intensity of vanilla aroma (Table 3). Vanillin is the principal marker of vanilla aroma; lactones can be regarded as direct contributors and/or possible enhancers of this descriptor (Spillman, Pollnitz, Liacopoulos, Skouroumounis, & Sefton 1997). The above observations suggest that judges perceived sweeter the wines with more intense vanilla flavour.

Woody overall character is positively correlated to guaiacol, methylguaiacol, eugenol, syringaldehyde, lactones and vanillin levels, which is reasonable since oak wood sensation is complex and influenced by the presence of various odour-active wood extractives (Boidron et al., 1988; García-Carpintero, Gómez Gallego, Sánchez-Palomo, & González Viñas, 2012; Sauvageot & Feuillat, 1999). For example, whiskey lactone is an attribute that accounts for a woody and coconut character (Boidron et al., 1988; Spillman et al., 1998, 1997), while high concentrations of this compound are associated with wine with an intense vanilla perfume (Boidron et al., 1988; Feuillat, Keller, Sauvageot, & Puech, 1999; Spillman et al., 1998).

Perceived spicy intensity is closely related to eugenol content, which is logical, since pure eugenol is described as clove-like (Feuillat et al., 1999). In our experiment, it is also linked positively to the presence of other odorous chemicals, like lactones, vanillin, methylguaiacol, and syringaldehyde, suggesting that in a complex medium such as wine the above volatile compounds may influence spicy aroma by means of additive, or synergistic effects. An important reduction in spicy and vanilla aroma takes place in wines with significant levels of furfural.

4. Conclusion

The results described here have shown that each oak winewood added unique and special characteristics to wine, and in addition each sample showed a different ability to extract compounds (volatile and non-volatile). Different rates of extraction have been observed, depending mainly on the origin of the compounds in the wood (toasting or present in natural wood) as well as on the watering process during toasting. The above differences were reflected by perceived sensory differences.

In general, volatile phenols, such as eugenol and iso-eugenol, guaiacol methylguaiacol along with phenolic aldehydes (vanillin) and lactones, showed a tendency to increase with increasing maceration time. The extraction rate of furanic compounds was maximum after 3 or 6 months of maceration; after 12 months these compounds were exhausted. Ellagitannins are extracted faster during the first 3 months, after 6 months an important decrease is observed. Wines with Special winewoods had lower ellagitannins concentrations and they demonstrated the greatest decrease.

Concerning sensory evaluation, oak wood contact time enhances vanilla, spicy, woody, characters and sweetness perception. For all the studied samples, with the exception of wines with Special winewoods, vanilla and spicy flavours increase linearly during storage. Wines treated with Special winewoods did not show substantial changes in the evolution of aromas during maceration time. Moreover, wine storage with winewoods had a sweetening effect and in parallel decreased the astringency and bitterness sensation. Astringency and bitterness were related significantly to ellagitannin levels ($r = 0.828$, $p = 0.001$ for astringency and $r = 0.607$, $p = 0.005$ for bitterness). A model like this satisfactorily predicts the sensation intensity of both astringency and bitterness if ellagitannins levels are known. Additionally, relationships between volatile oak compounds and sensory perception were found, confirming that both oak volatile compounds and ellagitannins influence wine perception. The general tendencies observed were that lactones levels induced positively the sweetness sensation whereas furanic and guaiacol compounds influenced the sensations of bitterness and astringency. Spicy and vanilla perception was significant positively related to the presence of eugenol, syringaldehyde and vanillin, as well as to the presence of other odorous chemicals. The above correlations obtained between sensory descriptive evaluation performed by a trained panel and wine chemical characterisation resulted in a useful tool applicable to wine development.

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