Contribution of oak wood ageing to the sweet perception of dry wines

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Abstract

Background and Aims: Winemakers observe a sensory effect of oak ageing in wines, but the phenomenon has never been fully described. The aim of this study was to determine the influence of the ageing container on the sweetness of dry wines and to evaluate the contribution of oak volatile compounds.

Methods and Results: Both white and red dry wines were produced in various containers, such as stainless steel and oak tanks, and new and used oak barrels. Sensory analysis (with or without use of nose clip) by a panel of experts showed an increase in sweetness when wine was in contact with wood. Oak volatile compounds (vanillin, β-methyl-γ-octalactone and eugenol) were quantified by gas chromatography-mass spectrometry and then added to the wines to obtain the same concentration in all samples. To determine the contribution of the volatiles to the increase in sweetness, the treatments were tasted again after such an 'oak aroma' homogenisation. The ranking of the wines remained unchanged.

Conclusion: These results demonstrate that the sweet perception of dry wines depends on the nature of the ageing container, and that these oak volatiles are not involved in this phenomenon.

Significance of the Study: This work confirms an empirical observation and establishes for the first time the sweetening effect of oak ageing, strongly suggesting the existence of sweet non-volatile compounds in oak wood.

Keywords: *GC-MS, oak ageing, oak aroma, sensory analysis, wine sweetness*

Introduction

The production of great wines and numerous spirits, such as cognac, armagnac, whiskey and rum, involves maturation in wood barrels. Contact with wood can occur during winemaking and ageing or only during ageing (spirits). While various tree species are used for cooperage, oak wood is the most widespread, in particular sessile oak (*Quercus petraea L.*), pedunculate oak (*Quercus robur L.*) and American oak (e.g. *Quercus alba L.*) (Ribéreau-Gayon et al. 2006).

During ageing, wine and spirits undergo several physicochemical modifications resulting in considerable evolution of their composition, colour, stability and sensory properties (Singleton 1974, Garde-Cerdán and Ancín-Azpilicueta 2006). In practice, both an aromatisation and an increase in flavour, particularly sweetness, are observed. Consequently, the length of maturation is a key factor in the quality of spirits and contributes notably to their commercial value.

Sensory modifications are in part due to oxidation-reduction phenomena because an oak barrel allows oxygen exchange with the ambient atmosphere (Ribéreau-Gayon 1933). For example, during ageing on white wine lees, there is a potential redox gradient from the bottom to the top of the barrel. The stirring of lees into the wine (batonnage) prevents the development of such a redox gradient (Chatonnet 1991). It is also well known that several reactions concomitant with oxidation phenomena (e.g. polymerisation and condensation), especially with phenolic compounds (e.g. flavan-3-ols and anthocyanins) lead to the formation of more stable pigments (Bakker and Timberlake 1997), explaining that the colour of red wine aged in oak barrels is more stable (Cano-López et al. 2006). Moreover, the interaction of anthocyanins with procyanidins can also influence the taste of wine because they can form the terminal subunits, thus preventing further polymerisation (Monagas et al. 2005). According to Vidal et al. (2004), they have been found not to contribute significantly to astringency. More generally, the phenolic compounds extracted from the wood during barrel ageing (ellagitannins, phenolic acids and wood aldehydes) play also an important role in protecting barrel-aged wine colour. In this way, some authors have studied the effect of adding oak chips, during the microoxygenation of red wines, on the colour and phenolic composition of wines, but they have also concluded that microoxygenation had much more impact on wine colour than oak had (Gómez-Plaza and Cano-López 2011). Cano-López et al. (2010) showed that microoxygenation improves wine colour in a way similar to oak barrel ageing. They do not evolve similarly, however, during bottle ageing. After 6 months in the bottle, microoxygenated wines were chromatically different from wines aged in new barrels, showing a more evolved colour.

Barrel ageing, moreover, leads to a release of volatile and non-volatile compounds present in oak wood (Puech et al. 1999, Ribéreau-Gayon et al. 2006). These molecules of different chemical nature can be native in oak heartwood or appear during the cooperage process (oak seasoning and toasting).

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Some of them have sensory properties that are likely to modify the aroma and taste of wine.

In recent years, a focus of research has been the identification and quantification of aromatic compounds released during barrel ageing. Vanillin (vanilla), β-methyl-γ-octalactone (coconut), volatile phenols (spicy) and 2-furanmethanethiol (toasted) are considered to be the key molecules associated with oak ageing including ageing in barrels (Chatonnet et al. 1991, Tominaga et al. 2000) and treatment with oak chips (Fernández de Simón et al. 2010).

In addition, various non-volatile compounds have been identified in oak extracts, including phenolic acids, such as ferulic and gallic acids, hydrolysable tannins (called ellagitannins) such as castalagin, vescalagin and roburin, coumarins such as scopoletin, lignans such as lyoniresinol, and polymeric compounds (Lapierre et al. 1983, Moutounet et al. 1989, 1992, Ribéreau-Gayon et al. 2006). Nevertheless, the molecular origin of taste modification remains largely unknown. Contrary to aromatic compounds, only few non-volatile molecules from oak have been characterised using classical sensory analysis or electronic tongue (Puech et al. 2007, Schmidtke et al. 2010). Some oak molecules are known to elicit bitterness and astringency (Quinn and Singleton 1985, Puech et al. 1999, Glabasnia and Hofmann 2006), suggesting that modification of the taste balance occurs during maturation in barrel or in contact with oak alternatives (Michel et al. 2011). Indeed, several researchers have shown that sweetness and bitterness are antagonist tastes (Calvino et al. 1990, Drewnowski 2001, Nurgel and Pickering 2006) involving a neural inhibition mechanism (Lawless 1979), and interactions between astringency and sweetness have also been reported (Brannan et al. 2001, Sáenz-Navajas et al. 2012a). Considering that taste–taste and taste–tactile sensation interactions have a large influence on the perception of wine quality (Sáenz-Navajas et al. 2012b), it appears paradoxical that winemakers observe an increase in sweetness during oak maturation whereas oak barrels release bitter and astringent compounds. This phenomenon could be explained by the existence of highly sweet compounds potentially extracted from oak wood, but knowledge of such molecules is substantially lacking.

To verify the empirical observation of winemakers, we analysed the influence of type of ageing container on wine sweetness. Because numerous interactions between aromatic molecules and taste perception have already been reported, the volatile key compounds were quantified by gas chromatography-mass spectrometry (GC-MS) in wine aged in different containers, and their influence on perceived sweetness was evaluated by sensory analysis.

Materials and methods

Wines

A white Bordeaux (France) wine from the 2007 vintage was used in this study. After pressing, a Sauvignon must (63 000 L) was settled at 200 NTU, inoculated with Zymaflore X5 yeast strain (100 mg/L, Laffort SA, Bordeaux, France), and the assimilable nitrogen content was adjusted to 200 mg/L. When one third of the alcoholic fermentation had been completed, the must was placed in four containers: a stainless steel tank (400 L), a tank made exclusively of new oak called 'new oak tank' (5000 L), two 1-year-old oak barrels (225 L) and two new oak barrels (225 L). The barrels and tank were made from French oak wood (*Quercus petraea*) that was chosen by the cooperage as representative of its production. The temperature was limited to 24°C during fermentation. When two thirds of the fermentation had been completed, the fermenting yeast in each container was subjected to a control of yeast implantation using polymerase chain reaction delta (Legras and Karst 2003); the genetic fingerprints of the six containers (1 SST, 1 NOT, 2 1OB, 2NOB) were identical and similar to that of the Zymaflore X5 yeast strain. After the end of alcoholic fermentation, the wines were aged on total lees. The level of free sulfur dioxide was maintained at 30 mg/L during the overall experiment and just before the bottling to avoid malolactic fermentation. The samples were removed from their respective containers after 5 months of ageing and constituted the four treatments of series W1 (Tables 1 and 2): W1-SST (stainless steel tank), W1-NOT (new oak tank), W1-1OB (1-year-old barrels) and W1-NOB (new oak barrels).

The red wine used was a Crozes-Hermitage (Rhone Valley, France) from the 2008 vintage. This Syrah wine (10 000 L) was first produced in stainless steel tank (14 000 L). After the end of malolactic fermentation, the wine was placed in four containers (series R1, Tables 1 and 2): one new oak tank (R1-NOT, 8500 L), two 2-year-old oak barrels (R1-2OB, 225 L), two 1-year-old oak barrels (R1-1OB, 225 L) and two new oak barrels (R1-NOB, 225 L). The barrels and tank were made from French oak wood (*Quercus petraea*) that was chosen by the cooperage as representative of its production. The level of free sulfur dioxide was maintained at 30 mg/L during the overall experiment and just before the bottling. The wines were bottled after 12 months of ageing. Each duplicate of the barrel treatments was individually bottled for chemical analysis (Table 2).

The wines were analysed for alcohol content, pH, titratable acidity and volatile acidity (Organisation Internationale de la Vigne et du Vin 2007). Alcohol content was measured after distillation by vapour training with an electronic densimeter

Table 1. Containers in which the two wines were aged.

Origin of wine	Volatile addition†	Series	Type of ageing container				
			Stainless steel tank	New oak tank	2-year-old barrels	1-year-old barrels	New oak barrels
White Bordeaux 2007	No	W1	W1-SST	W1-NOT	—	W1-1OB	W1-NOB
	Yes†	W2	W2-SST	W2-NOT	—	W2-1OB	W2-NOB
Red Crozes-Hermitage 2008	No	R1	—	R1-NOT	R1-2OB	R1-1OB	R1-NOB
	Yes†	R2	—	R2-NOT	R2-2OB	R2-1OB	R2-NOB

†Addition of vanillin, *cis* and *trans* β-methyl-γ-octalactone and eugenol to obtain the same concentration in all samples of a series. —, non-existent treatments.

Table 2. Basic composition of the two wines used in the study.

Wine	White Bordeaux 2007						Red Crozes-Hermitage 2008						
Treatments	**W1-SST**	**W1-NOT**	**W1-1OB**		**W1-NOB**		**R1-NOT**	**R1-2OB**		**R1-1OB**		**R1-NOB**	
Samples (Duplicates)†			Barrel 1	Barrel 2	Barrel 1	Barrel 2		Barrel 1	Barrel 2	Barrel 1	Barrel 2	Barrel 1	Barrel 2
Ethanol (% v/v)	12.32 ± 0.05	12.27 ± 0.05	12.31 ± 0.05	12.28 ± 0.05	12.29 ± 0.05	12.28 ± 0.05	13.08 ± 0.05	13.11 ± 0.05	13.10 ± 0.05	13.09 ± 0.05	13.11 ± 0.05	13.11 ± 0.05	13.12 ± 0.05
Glycerol (g/L)	6.0 ± 0.1	5.8 ± 0.1	5.9 ± 0.1	5.9 ± 0.1	5.9 ± 0.1	6.0 ± 0.1	6.8 ± 0.1	6.9 ± 0.1	6.9 ± 0.1	6.8 ± 0.1	6.9 ± 0.1	6.8 ± 0.1	6.8 ± 0.1
Glucose + fructose (g/L)	0.17 ± 0.05	0.18 ± 0.05	0.14 ± 0.05	0.17 ± 0.05	0.17 ± 0.05	0.18 ± 0.05	0.24 ± 0.05	0.21 ± 0.05	0.24 ± 0.05	0.25 ± 0.05	0.22 ± 0.05	0.25 ± 0.05	0.2 ± 0.05
Residual sugars (g/L)	0.5 ± 0.1	0.6 ± 0.1	0.5 ± 0.1	0.5 ± 0.1	0.6 ± 0.1	0.6 ± 0.1	1.0 ± 0.1	1.1 ± 0.1	1.0 ± 0.1	1.1 ± 0.1	1.1 ± 0.1	1.2 ± 0.1	1.1 ± 0.1
pH	3.18 ± 0.05	3.17 ± 0.05	3.14 ± 0.05	3.18 ± 0.05	3.19 ± 0.05	3.17 ± 0.05	3.56 ± 0.05	3.54 ± 0.05	3.53 ± 0.05	3.51 ± 0.05	3.57 ± 0.05	3.53 ± 0.05	3.57 ± 0.05
Total acidity (g/L)‡	6.7 ± 0.1	6.8 ± 0.1	6.6 ± 0.1	6.7 ± 0.1	6.7 ± 0.1	6.8 ± 0.1	5.6 ± 0.1	5.6 ± 0.1	5.5 ± 0.1	5.6 ± 0.1	5.5 ± 0.1	5.7 ± 0.1	5.6 ± 0.1
Volatile acidity (g/L)‡	0.19 ± 0.06	0.21 ± 0.06	0.22 ± 0.06	0.24 ± 0.06	0.23 ± 0.06	0.24 ± 0.06	0.42 ± 0.06	0.47 ± 0.06	0.45 ± 0.06	0.41 ± 0.06	0.45 ± 0.06	0.48 ± 0.06	0.49 ± 0.06
Free sulfur dioxide (mg/L)	28 ± 2	27 ± 2	29 ± 2	29 ± 2	28 ± 2	27 ± 2	26 ± 2	28 ± 2	29 ± 2	28 ± 2	25 ± 2	27 ± 2	26 ± 2

†For sensory analysis, duplicates were blended (50 % of each barrel). ‡Total acidity and volatile acidity are expressed in g/L of tartaric acid and acetic acid, respectively.

(Anton Paar DMA 35 N, Anton Paar GmbH, Graz, Austria). Wine pH was measured with a pH meter (pH 538 Multical®, WTW; Wilheim, Germany) standardised to pH 7.0 and 4.0. Glycerol and glucose + fructose were assayed by an enzymatic method (Boehringer kits, R-Biopharm, Darmstadt, Germany). Residual sugars and volatile acidity were determined chemically by colorimetry (460 nm) in continuous flux (Sanimat, Montauban, France). Sulfur dioxide was measured with a titrator (Iodomatic Oeno-20, Oeno-Bio, Saint-Martin-Le-Vieil, France).

Quantitative analysis of volatile oak compounds in wines.

Extraction procedure

The extraction procedure was based on the method described by Cutzach et al. (1998) and Prida et al. (2007). Wine samples (100 mL) were spiked with 100 µL 3-octanol (100 mg/L in EtOH) as an internal standard. Wines were extracted three times with 10, 5, 5 mL CH_2Cl_2 (magnetic stirring: 10, 5, 5 min; 750 rpm). The three organic phases obtained were blended, dried over anhydrous sodium sulfate and concentrated to 0.5 mL under a nitrogen stream. Two microlitres of the extract were injected into the gas chromatograph (GC) with a mass spectrometer (MS) detector.

GC-MS

The organic extract was analysed with a trace GC fitted to a DSQ II quadrupole MS (Thermo Fisher, Paris, France) in the electronic impact 70 eV mode. The MS interface temperature was set at 230°C. The carrier gas was helium N60 (Air Liquide, Floirac, France). Two microlitres of organic extract were injected in splitless mode (injector temperature 230°C; column head pressure 159 kPa purge flow 50 mL/min) on a BP20 capillary (50 m, 0.22 mm internal diameter; 0.25 µm film thickness) (SGE, Interchim, Montluçon, France). Temperature was as follows: 45°C for 1 min, then increasing temperature to 230°C at 3°C/min and final isothermal for 15 min. Analysis was performed in selected ion monitoring mode after checking the retention time of quantified compounds with that of the standards in total ion current mode. Vanillin, β-methyl-γ-octalactone and eugenol were quantified by means of the ratio of the areas of their main characteristic ions *m/z* 151, *m/z* 99, *m/z* 164, respectively, with those of 3-octanol (*m/z* 83).

Sample preparation and sensory analysis

For both series (white and red), other treatments were prepared by adding volatile compounds in order to obtain similar concentrations in each sample of a given series. In practice, 1 h before tasting, the level of vanillin, oak lactone and eugenol was adjusted to the highest measured concentration, i.e. corresponding to the wine aged in new oak barrels (W1-NOB and R1-NOB, respectively). As commercial oak lactone is a mix of cis/trans isomers (1/1), we decided to adjust the level of the highest odorant form, e.g. *cis* form. In this way, two complementary series of four samples were prepared (W2 and R2, Table 1).

Bottles were opened 1 h before tasting, and the barrel duplicates were blended (50/50) to provide the corresponding treatment used for sensory analysis. The samples (20 mL) were presented at 18°C for the red wines and 12°C for the white wines in normalised dark glasses coded with random numbers. All the tasting sessions took place in a specific room equipped with individual booths and air-conditioned at 20°C. All the panellists (32 tasters aged from 22 to 61 years) were wine-tasting specialists or winemakers and had been previously informed of the nature and risks associated with the investigation. Four tasters were absent or ill for the red series.

Sensory analysis (W1, W2, R1 and R2) consisted of independent ranking tests. For each series, four glasses corresponding to each of the four containers in which the wines were aged (Table 1) were presented to the assessors, who were asked to classify the samples from less to more sweet. The order of presentation was imposed to the taster by the individual tasting sheet. With four samples, the number of possible combinations is 4! = 24. In our tests, the panel contained 32 tasters, so each combination was used once (24 tasters), and the eight other combinations were randomly chosen among the 24 possibilities. In this way, we attempted to minimise order effects. Each combination was randomly attributed to each taster. This procedure follows the recommendation of ISO 8587 (International Organization for Standardization 2006). Wines of series W1 were also tasted by the panellists fitted with a nose clip (FIM Medical, Lyon, France) to remove the retronasal pathway.

Statistical analysis

Results obtained from sensory tests were statistically interpreted according to the norms published by the International Organization for Standardization (2006). The test implemented here consisted of a ranking test with no preordained order, so the Friedman test was used. For each assessor, a value between 1 and 4 was attributed to each sample, depending on the response of the assessor (1 for samples designated as the least intense, 4 for the most intense). The sums of the ranks were obtained for each sample, then the parameter F was calculated using Friedman test specifications and compared with a χ^2 value in order to determine whether the result of the test was significant ($F \geq \chi^2$) or not ($F < \chi^2$).

The concentration of volatile compounds was expressed as a means ± standard deviation. The effects of the containers on each variable were tested by one-way analysis of variance (ANOVA). If significant effects were found at a 95% confidence interval, ANOVA was followed by a Duncan post hoc test to identify differences between groups. Statistical analyses (ANOVA and Duncan's post hoc tests) were performed using Statistica V.7 (Statsoft Inc., Tulsa, OK, USA).

Panel training before wine evaluation

Prior to the formal evaluation of wines, the panellists undertook training sessions. For these sessions, two unoaked commercial wines were used: a white Bordeaux 2008 (12.1% ethanol, 5.4 g/L glycerol, 0.30 g/L glucose + fructose) and a red Bordeaux 2008 (12.4% ethanol, 6.2 g/L glycerol, 0.43 g/L glucose + fructose). For each wine, three sweet compounds, i.e. sucrose, aspartame and neohesperidin dihydrochalcone, were separately added at four concentrations resulting in six training series (three compounds; two wines) with four treatments for each series (Table 3). One session was dedicated to white wines,

Table 3. Red and white wines with sucrose, aspartame and NHDC added separately for panel training.

	Compound		Concentration†	
Sucrose	+2	+4	+8	+16
Aspartame	+10	+20	+40	+80
NHDC‡	+0.5	+1	+2	+4

†g/L for sucrose and mg/L for aspartame and NHDC. ‡Neohesperidin dihydrochalcone. NHDC, neohesperidin dihydrochalcone.

another to red wines. The panellists were asked to classify the wines according to the intensity of sweetness.

This ranking test with a preordained order was interpreted using a Page test according to the norms published by the International Organization for Standardization (2006).

Results and discussion

Influence of the type of ageing container on wine sweetness

The data presented in Table 4 showed that the training tests were significant for the three compounds, for both wines. The implementation of such tests trained the panellists to perceive differences of sweetness because of various non-volatile compounds in a complex mixture as dry wine. Their performance demonstrates their ability to classify wines according to sweet intensity.

White wines. During the filling of the containers, the must was homogenised, and the turbidity was identical across the treatments. Consequently, the fermentation kinetics were similar between the replicates and between the treatments; no significant difference of the basic physico-chemical parameters (ethanol, glycerol, residual sugars, pH, total and volatile acidity) was observed (Table 2) with regard to the standard errors of the analytical methods used. Yeast lees increase perceived sweetness (Marchal et al. 2011a), so the lees ageing conditions had to be similar in the different treatments. For this reason, a small stainless steel tank (400 L) was used to allow wine ageing on lees without the appearance of reduction off-flavours generally encountered with ageing in large tanks (Lavigne 1996).

Each taster was forced to assign 1 to the less sweet wine and 4 to the sweetest one. The sum of the ranks was calculated and conveyed the sweet perception of each treatment in comparison with other treatments (but not the absolute rating of sweetness because the different ranks attributed by the tasters may not be equidistant). The Friedman test used for statistical analysis was significant at 1% (Table 5), which means the tasters were able to distinguish different categories of treatments according to their sweet perception. Moreover, the highest sum of ranks (corresponding to treatment designed most often as the sweetest one) was observed for treatment W1-NOB, followed by treatments W1-1OB, W1-NOT and finally, W1-SST. Therefore, in this experiment, the type of container generates differences in wine sweetness during the ageing process. Moreover, the ranking of the treatments showed that wines aged in contact with oak wood were classified as sweeter by the panellists.

Red wines. During fermentation on skins, the size and volume of the container strongly influences the extraction of grape compounds and the taste of wine. Consequently, contrary to the white wines, the red wines used in this study were placed in various containers after the end of malolactic fermentation to avoid such influences. Hence, the experiment concerned only wine ageing and not the entire production process. With regard to the standard errors of the analytical methods used, no significant difference of the basic physico-chemical parameters (ethanol, glycerol, residual sugars, pH, total and volatile acidity) was observed between the replicates and between the treatments (Table 2). As for the white wines, the Friedman test was also significant in this experiment (at 5%), indicating the ability of the tasters to distinguish categories of wines according to their sweetness intensity. The highest sum of ranks was obtained for sample R1-NOB, then R1-1OB, R1-2OB and R1-NOT, so the treatments mainly designated as the sweetest were aged in contact with oak wood.

These results were similar to those obtained for the white wines, demonstrating that the type of the ageing container used in this work appeared to influence the sweet taste of the wine. Indeed, the wines designated as sweetest were most often aged in oak containers, especially new oak barrels. The taste quality of a white wine depends on a balance between acidity and sweetness, whereas the balance of a red wine is more complex than that in white wines and involves more sensations, such as bitterness or astringency. This could cause a difference of sweetness perception between white and red wines.

Table 4. Results of the panel training with the selected sweet compounds.

Compounds	Wine	R_1†	R_2†	R_3†	R_4†	L‡	L′‡§
Sucrose	White	61	69	80	110	879	4.84**
	Red	60	78	86	96	858	3.55**
Aspartame	White	53	72	90	105	887	5.33**
	Red	49	70	86	115	907	6.55**
NHDC	White	57	79	89	95	862	3.80**
	Red	52	74	91	103	885	5.21**

*Significant at 5% ($L' \geq 1.645$); **Significant at 1% ($L' \geq 2.326$). †$R_1 \ldots R_4$ are the sums of ranks for treatments 1 to 4. ‡L and L′ were calculated as described in ISO 8587 (International Organization for Standardization 2006) for page test: $L = \sum_{i=1}^{p} i\mathrm{Ri}$ and $L' = \frac{12L - 3n \cdot p \cdot (p+1)^2}{p \cdot (p+1) \cdot \sqrt{n \cdot (p-1)}}$ (n is the number of panellists and p is the number of concentrations). §Significance: ns, non-significant (L′ < 1.645). NHDC, neohesperidin dihydrochalcone.

Table 5. Ranking of perceived sweetness in wines aged in various containers.

Wine	Treatments and sums of ranks				F†	Result‡
White Bordeaux 2007	W1-SST	W1-NOT	W1-1OB	W1-NOB	30.63	Significant at 1%
	52	73	90	105		
Red Crozes-Hermitage 2008	R1-NOT	R1-2OB	R1-1OB	R1-NOB	10.93	Significant at 5%
	52	71	74	83		

†F was calculated as described in ISO 8587:2006 (2006) for Friedman test. $F = \frac{12\sum_{i=1}^{p} R\mathrm{i}^2}{n \cdot p \cdot (p+1)} - 3 \cdot n \cdot (p+1)$ with n represents the number of tasters, p represents the number of modalities/treatments and Ri represents the sum of the ranks for the modality i. ‡With four treatments, the test was significant at 5% when F > 7.81 and at 1% when F > 11.34.

The experiment has involved one white wine and one red wine only, and it could be interesting to repeat it with more wines and other cultivars because the nature of the non-volatile matrix might strongly influence the sweetness perception. Moreover, the oak wood used in this study was chosen by the cooperage as representative of its production, and it would be interesting to evaluate more specifically the influence of other oak species and origins.

In addition, the fact that white wine treatments were partially fermented in different containers and that yeast biomass can influence perceived sweetness (Marchal et al. 2011a) could create an artefact in the assumption that yeast biomass was strongly different between treatments. Nevertheless, the must was inoculated in a common stainless steel tank and placed in several containers corresponding to the different treatments after about one third of the fermentation had been completed. Albertin et al. (2011) have shown that at this fermentation step, fermenting yeast has reached a maximum population size and the concentration of yeast is then stable up to the end of alcoholic fermentation. Moreover, an examination of the yeast population, after two thirds of the fermentation had been completed in each container, revealed that the genetic fingerprint of all the containers was identical and similar to that of Zymaflore X5 yeast strain used for inoculation. Considering all these data and the fact that the fermentation times and temperatures were exactly similar in various treatments, we assume that the difference of yeast biomass (content or quality) was too small in this trial to explain the modifications of perceived sweetness. Repetition of such experiments is expected to confirm these results.

Thus, it appears that the sweetness of both white and red dry wines was modified by the type of ageing container and the increased contact with oak wood in this experiment. Despite the well-known release of bitter and astringent compounds typical of oak ageing, the sweet perception increased during maturation suggesting the liberation of sweet molecules able to develop taste interactions and consequently to impact the taste balance. As this modification of sweet taste was similar in white and red wines, although the composition of their non-volatiles is largely different, these results suggest that the phenomenon was due to the extraction and release of sweet compounds from oak to wine, and not due to the modification of grape-derived components during oak maturation.

Smell and taste are two distinct sensations, respectively, perceived by the olfactory and gustatory systems. In particular, sweet perception involves taste receptors located in the mouth that are stimulated by non-volatile compounds (Temussi 2006). Taste in physiological terms, however, is almost never experienced alone when eating but in association with olfactory stimulation through the retronasal pathway (Schiffman 2000) because dishes always contain both volatile and non-volatile compounds. These sensory associations induce interactions between smell and taste. Indeed, from a phenomenological point of view, tasters often use the term 'sweet' to describe an olfactory sensation (Dravnieks 1985). Even if the sweet sensation does not result from the stimulation of the taste receptors by aromatic compounds (Labbe et al. 2006), the expression 'sweet odour' has a sensory reality as a result of a multimodal representation. The fact of smelling an odour usually associated with a sweet taste suggests the taste itself to the taster (Stevenson et al. 1998). This has been reported for various fruity scents such as strawberry (Bate-Smith 1968). Moreover, volatiles are able to modify the gustatory perception of non-volatile molecules through synergistic effects. For example, the sweet intensity of a sucrose solution is increased by addition of vanillin (Clark and Lawless 1994, Small et al. 2004).

Table 6. The ratio of volume to internal area for a barrel and oak vats.

	Volume (L)	Area (m^2)	Ratio
Barrel	225	2.46	91.5
Oak vat	5000	16.6	301.2
Oak vat	8500	22.95	370.4

Previous studies have therefore shown that volatile compounds interact with taste-active molecules and can generate a sweet representation through perceptive and cognitive associations.

Oak wood releases various volatiles during wine ageing; vanillin, β-methyl-γ-octalactone (oak lactone) and eugenol are known to be the key molecules responsible for woody aroma. Moreover, these compounds, in particular vanillin and oak lactone, are often associated with sweet foodstuffs. Consequently, because the type of ageing container had been shown to influence wine sweetness, we decided to measure the concentration of the principle oak volatile compounds released by the containers. The aim was to demonstrate the differential release of oak volatile compounds according to the type of container, and then to evaluate the influence of these molecules on wine sweetness.

Influence of container type on the concentration of vanillin, oak lactone and eugenol in wine

We analysed these compounds in the wines previously tasted (after 6 and 12 months ageing for the dry white Bordeaux wine and red Crozes-Hermitage wine, respectively). First, the *cis* and *trans* isomers of β-methyl-γ-octalactone (oak lactone), vanillin and eugenol were not detected in the initial white wine (W1-SST), without contact with oak wood. For this reason, they were considered as markers of oak wood-aged wines. At the end of ageing, the wine aged in new barrels (W1-NOB and R1-NOB) showed the highest concentration of volatiles, which is one of the most important characteristics of wines aged in these conditions, irrespective of grape cultivar.

The volatile compounds were extracted from the wood according to the age of the barrel and the ratio of barrel capacity/wood area. For all the volatile compounds studied, we found a significant difference ($P < 0.05$) between the concentration found at 6 and 12 months of ageing for the white and red wines, respectively. If we take into account, however, the ratio of the volume to the internal area of a barrel and an oak vat (Table 6), the concentration of the volatile compounds in new barrels and the oak vat was more or less similar. The detection threshold of *trans*- and *cis*-oak lactone is respectively, 460 and 92 μg/L in white Ugni Blanc wine and 320 and 74 μg/L in a red Merlot wine (Chatonnet et al. 1992). More recently, Brown et al. (2006) found a detection threshold level quite different: 24 and 172 μg/L for the *cis*- and *trans*-oak lactone in a white wine; 57 and 380 μg/L for the *cis*- and *trans*-oak lactone in red Shiraz wine. The concentration of *trans*-oak lactone ranged from 58.2 to 181.3 μg/L while *cis*-oak lactone concentration ranged from 82.9 to 463.1 μg/L (Table 7). In the red wine, the concentration of *cis* isomer was above the perception threshold in all the treatments, while the *trans*-isomer concentration was under

Table 7. The concentration of vanillin, eugenol, and oak lactone found in white and red wines aged in various containers.

Wine	Treatments	Vanillin (μg/L)†	Oak lactone (μg/L)†		Eugenol (μg/L)†
			trans	*cis*	
White Bordeaux 2007 W1	W1-SST (stainless steel tank)	Trace	Trace	Trace	Trace
	W1-NOT (oak vat)	28.4 (1.5)^{c}	58.2 (3.3)^{c}	82.9 (2.5)^{c}	5.5 (0.9)^{c}
	W1-1OB (1-year-old barrels)	120.5 (1.8)^{b}	122.8 (5.5)^{b}	210.3 (4.5)^{b}	15.2 (2.1)^{b}
	W1-NOB (new barrels)	148.7 (4.1)^{a}	143.6 (2.3)^{a}	268.6 (12.1)^{a}	20.1 (1.1)^{a}
Red Crozes-Hermitage 2008 R1	R1-NOT (oak vat)	91 (1.4)^{c}	Trace	84.2 (2.9)^{d}	7.8 (0.9)^{b}
	R1-2OB (2-year-old barrels)	92.2 (5.1)^{c}	36.1 (3.1)^{c}	178.3 (3.5)^{c}	9 (1.1)^{b}
	R1-1OB (1-year-old barrels)	214.3 (8.1)^{b}	116.9 (3.7)^{b}	226.2 (5.5)^{b}	22.4 (1.1)^{a}
	R1-NOB (new barrels)	348.5 (19.2)^{a}	181.3 (5.9)^{a}	463.1 (11.1)^{a}	24 (0.9)^{a}

†Data are mean of triplicate determination. Results of variance analysis: for each type of wine, values with different letters within each row are significantly different (Duncan's test, $P < 0.05$).

Table 8. Ranking of perceived sweetness in wines aged in various containers after addition of vanillin, oak lactone and eugenol.

Wine	Treatments and sums of the ranks				F†	Result†
White Bordeaux 2007	W2-SST	W2-NOT	W2-1OB	W2-NOB	29.81	Significant at 1%
	57	66	89	108		
Red Crozes-Hermitage 2008	R2-NOT	R2-2OB	R2-1OB	R2-NOB	9.87	Significant at 5%
	54	69	74	83		

†The test was interpreted as described in ISO 8587:2006 (International Organization for Standardization 2006) for Friedman test. See Table 5.

the perception threshold. Similar observations were made with the white wine, except for the oak vat-aged treatment where the contribution of the *cis* form was low. The concentration of these two isomers are in accordance with that found in other work reporting the influence of the age of barrels on the volatiles found in wines (Fernández de Simón et al. 2008).

Eugenol is one of the most important phenols because of its aromatic properties, with a sensory threshold in wines of 500 μg/L (Chatonnet et al. 1992). In our experiment, however, the concentration of eugenol was systematically below its perception threshold.

The concentration of vanillin was in agreement with that of oak lactone and eugenol distribution in the different vats. The vanillin concentration found in the wines aged with oak ranged from 28.4 to 348.5 μg/L. According to Chatonnet et al. (1992), the perception threshold of vanillin is 400 μg/L for a white wine and 320 μg/L for a red wine. At these thresholds, vanillin can influence the flavour of some (but not necessarily all) wines aged in new oak and probably influences the perception of the 'vanilla' character in wines (Chatonnet et al. 1991, Spillman et al. 1997). In our experiment, the contribution of vanillin to the flavour of the red wine was obvious only in the red wine aged 12 months in new barrels.

At the end of the ageing period, the choice of several vats gave us the opportunity to obtain a natural enrichment in flavour compounds associated with a broad range of concentration of volatiles in the wines.

Role of vanillin, oak lactone and eugenol in the increase in perceived sweetness because of oak ageing of dry wines

Many examples of the interaction between odour and taste have been previously reported. Additionally, we observed a differential concentration of key volatiles in wines aged in various containers. Altogether, these results led us to study the role of the volatile compounds characteristic of oak ageing in the increase in wine sweetness observed in this work. In a first experiment, vanillin, oak lactone and eugenol were added for each series to the various wines until the highest concentration was reached, i.e. in treatments W1-NOB and R1-NOB (Table 7). All the wines obtained in this way contained the same level of key volatile molecules characteristic of each series (W2 and R2). Consequently, the oak aroma due to vanillin, oak lactone and eugenol was evenly distributed between the four treatments of a series. The different samples were tasted by the same panellists and in the same conditions as for treatments W1 and R1.

For both white (W2) and red wines (R2), the Friedman test was significant at 1 and 5%, respectively (Table 8), and the Friedman values were quite close to those obtained for series W1 and R1 (Table 5). The panellists were able to categorise treatments W2 and R2 according to their perceived sweetness, and the order of the sums of ranks was unchanged compared with that for wines W1 and R1. Therefore, a similar difference in sweetness perception was observed between treatments in the series with a different (W1, R1) and equal (W2, R2) level of vanillin, oak lactone and eugenol. These results demonstrated that the concentration of key volatile oak compounds (vanillin,

Table 9. Ranking of perceived sweetness in W1 wines by using a nose clip.

Wine	Treatments and sums of the ranks				F†	Result†
White Bordeaux 2007 with nose clip	W1-SST	W1-NOT	W1-1OB	W1-NOB	26.14	Significant at 1%
	56	69	91	104		

†The test was interpreted as described in ISO 8587:2006 (International Organization for Standardization 2006) for Friedman test. See Table 5.

oak lactone and eugenol) did not affect the perception of sweetness for the wines used in this work. Similarly, addition of vanillin (0, 100, 300 and 900 µg/L) and furfurylthiol (0, 5, 15 and 45 ng/L) to unoaked wines did not affect the perceived sweetness (results not shown). Consequently, the sweetening effect of oak barrel ageing observed in this study is not due to cognitive interactions involving oak key volatile compounds, such as vanillin, oak lactone and eugenol.

The olfactory effect of all volatile compounds coming from grape, lees and oak was then suppressed by tasting the wines of series W1 using a nose clip. Indeed, a nose clip closed the nostrils and consequently prevented orthonasal olfaction, but also retronasal stimulation by suppressing airflow in the nasal cavity (Chen and Halpern 2008, Lim et al. 2008). The samples were tasted by the same trained panellists using a nose clip. The test was significant at 1% (Table 9), and the results were similar to those obtained without a nose clip. So the suppression of the olfactory stimulation of all wine volatiles (both orthonasal and retronasal pathways) did not change the ability of the tasters to distinguish the treatments according to their perceived sweetness.

Jointly, these experiments suggest that the differences of perceived sweetness were not due to volatile compounds. These findings suggest that some non-volatile compounds released from oak wood may have sweetening properties and contribute to the sweet taste of dry wines. Such assumptions are supported by the recent identification in oak wood of new sweet compounds called quercotriterpenosides because of their triterpenoid nature (Marchal et al. 2011b). As for each sensory attribute, it will be important to relate these results concerning sweetness to consumer preference by determining, e.g. the consumer rejection threshold (Yoo et al. 2012).

Conclusion

We showed in this study that wines aged in various containers exhibited variations of sweet taste, whereas their basic analyses (ethanol, glycerol, pH, total acidity, volatile acidity) did not differ. Moreover, we demonstrated that oak volatiles and more generally volatiles were not involved in these differences of perceived sweetness. Similar experiments could be carried out with other types of wine and various oak origins to generalise our conclusions and specify the effects of the taste modifications on the consumer acceptance or rejection. These findings, however, suggest that non-volatile compounds are released during oak ageing and contribute to the increase in sweetness perceived in the wines used for this work. The repeatability of this phenomenon according to the types of wine and oak will be measured, and the study of their interactions with oak bitter compounds will need further investigation to provide a better understanding of the gustatory impact of oak ageing.

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