BARREL-AGED WINES

Olfactory Perception of Oak-Derived Compounds

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Barrel-aged wine is a complex mixture and its olfactory perception results from the interaction of many aromas. To estimate the role of oak-derived aroma compounds, it is necessary to consider odor activity values (OAV) and to perform a correlation study to assess the impact of individual aroma compounds on the aroma attributes.

Twenty Spanish and French wines, each aged in different types of barrels, were studied using both sensory (descriptive) and chemical (GC-MS) analysis. Paired-sample t-tests were used to assess whether there were consistent differences in the concentrations of oak-derived compounds between wines aged in different barrels and receiving different sensory scores.

Despite their low OAVs in the wines, furanic compounds (fur-fural, furfuryl alcohol, and 5-methylfurfural) increased the perceived overall oak intensity rating and decreased the fruity intensity rating. It is hypothesized that these different compounds indirectly impacted the respective overall oak and fruit intensities. The presence of cis- and trans-whisky lactones, eugenol, and vanillin increased the intensity rating of the vanilla/pastry descriptor, while furfural and 5-methylfurfural diminished it.

Although the volatile phenols (guaiacol, 4-methylguaiacol, eugenol) are described as smoky and spicy in their pure state, no reliable links were found between these compounds and their respective sensory descriptors in wines. Samples described as having higher olfactory persistence were richer in relatively heavy volatiles, such as trans- and cis-whisky lactone, maltol, eugenol, and vanillin, than their paired samples. This would explain their retronasal persistence.

Research on the compounds responsible for specific aromas in wine begins by identifying the key compounds that remind the taster of these odors. However, only a few compounds, that are aromatic in their pure state, can be perceived in wine.

Wine has been characterized as a sensory buffer, because it is a solution in which the addition or omission of several odorant compounds can occur without any significant changes in the overall aroma perception.[6]

Sensory buffer components include ethanol, fermentation products, and odor compounds that cannot break through this buffer. Together these components provide wine with a generic wine flavor with no specific notes. The compounds that can break through this buffer, referred to as impact compounds, confer certain specific aromas to wine, such as raspberry, grapefruit, and smoke.

The odor activity value (OAV) of an aroma compound is the ratio between the concentration of the compound in the wine to its sensory threshold. This indicates the likelihood of the compound being an impact compound. A taster cannot predict the perceptible intensity of the aroma. This is because, depending on their chemical structure, the effects of the matrix can strongly affect the volatilization of odor components.[8,9]

Barrel maturation adds more complexity to wine because of the leaching of several strong odorant chemicals from the wood and their subsequent transformation in the wine. Important wood chemicals, that can be impact molecules, include the naturally present cis- and trans-methyl-octalactone (whisky lactones, with a coconut flavor in their pure state), trans-2-nonenal (sawdust smell), volatile phenols released via toasting such as guaiacol, 4-methylguaiacol, and eugenol (spicy and smoky smells), and vanillin (vanilla smell).

All of these compounds can be present in barrel-aged wines in concentrations above the sensory threshold. Their concentrations depend on the chemical composition of the barrels wood, the nature of the wine, and the length of time the wine has spent in contact with the wood.

Decreased vanillin concentration during barrel fermentation and maturation in the presence of yeast lees has been reported.[5,13,14] The products of transformation were low odorant vanillyl alcohol and vanillyl ethyl ester.

The trans-2-nonenal concentration decreases during stave maturation and more dramatically during toasting.[4] In a wine medium, this compound, like any other aliphatic aldehyde, can interact with tannins and sulfur dioxide. Thus, its contribution is perceived only in wines characterized by pronounced green sawdust off-flavors.

Furfurylthiol and 5-methyl-2-furanmethanthiol (coffee-smelling compounds with very low odor thresholds of 0.4 and 50 ng/L, respectively) have been identified as products of transformation of furfural and 5-methylfurfural, respectively, in a wine medium.[2,16,17]

However, the importance of these compounds in matured wine is difficult to measure as they are very unstable. Wine is a complex mixture and its olfactory perception is the result of the interaction of odors. Therefore, to estimate the role of a particular odor compound, it is important to consider both the OAVs and to perform a correlation study to discern the impact of flavor compounds on aroma attributes. Several studies dealing with wood-derived compounds have already been undertaken.

In Pinot Noir wine, it was observed that there was either a positive correlation between cis-methyl-octalactone and sensory descriptors such as toasty, coconut, woody, and vanilla, or there was a negative correlation with the pharmaceutical, hay, and clove descriptors.[12]

In another study, a partial least squares model was applied to the sensory and chemical results of 57 Spanish wines. An excellent correlation between the woody-vanilla-cinnamon descriptor and cis-methyl-octalactone was found.[1] This correlation was less pronounced with vanillin and eugenol. In addition, cis-methyl-octalactone contributed to the intensity of the sweet-candy-cocoa descriptor and vanillin to the fruity descriptor.

In a more recent study on white (Chardonnay) and red (Cabernet Sauvignon) wine was aged in different barrels. The cis-methyl-octalactone concentration correlated positively with the coconut, berry, coffee, and dark chocolate descriptors in red wine. However in white wine, the same concentration correlated positively only with coconut.[15] The vanillin concentration in white wine was not directly correlated to the vanilla descriptor, however it was correlated to the cinnamon and smoky descriptors.

Other compounds, including guaiacol, 4-methylguaiacol, 4-ethylphenol, furfural, and 5-methylfurfural, contributed to the intensity of the smoky descriptor. In red wine, the vanilla descriptor is linked to numerous wood compounds such as volatile phenols, gamma-lactones, and furanic compounds while the smoky descriptor is correlated to furfuryl alcohol.

The above studies highlight several important phenomena. Odorant molecules often enhanced the intensity of a descriptor differently from the aroma of the respective chemicals in their pure state. It was also observed that compounds that were judged unimportant because of their low OAV, such as furanic compounds, in fact, did have sensory impact.

However, the conclusions of these studies cannot be widely extended. Two were characterized by small sample sizes of only one[12] and two[15] wines. Therefore, the scientifically rigorous conclusions reached in their studies apply only to those wines. With a different wine matrix, the conclusions could be different. The third study used a much broader experimental design (57 wines), which allowed for more general conclusions about the correlations.[1]

In this study, a statistically different approach was used to add to the work done in these previous studies. A range of different wines, each aged in different types of barrels, was compared using both sensory and chemical analysis. Paired-samples t-tests were used to assess whether there is any evidence of systematic differences in the concentrations of aroma compounds between wines aged in different barrels and which were evaluated differently in the sensory tests.

Comparisons were made using paired tests on the same wine matrix. Thus, the differences in sensory perception and chemical composition reported in the study were solely due to the impact of different barrels and not to the wine itself.

This study is limited to only wood-derived compounds and descriptors directly linked to characteristics of these compounds.

Materials and Methods

Wines – Twenty different French and Spanish wines were selected and aged in barrels for 6 to 12 months (Table I). Each wine was aged in new barrels of various types produced from French oak wood (Seguin-Moreau Cooperage, Merpins, France). Variations between barrel types included grain width, bending technique (water or fire), and toasting levels and technique(s) (such as toasting barrel body and/or barrel heads).

The aim of the study was to compare the sensory perceptions of identical wines that were aged in different barrel types that contribute different wood-derived compounds. At least three barrels per set were used for trials.

Sensory analysis – Sensory analyses were performed by a tasting panel of 10 to 14 people comprising professional enologists from the internal staff of the Seguin Moreau Cooperage and invited winemakers. Panelists were asked to assign quantitative scores from 0 to 10 (0 as lowest and 10 as highest) to the following descriptors: fruity, vanilla/pastry, toasty/smoky, spicy, overall woody, and olfactory persistence. The aromas were assessed by nose, while olfactory perception was scored by retro-nasal perception.

Training sessions were conducted using wines characterized by different intensities of individual descriptors (e.g. fruity) after a panel consensus on these wines. The overall woody descriptor was chosen by tasters to describe all olfactory sensations brought about by the wood.

Sensory sessions were organized by series, with the same wine aged in different barrel types. Either one single session per day or a maximum of three sessions per day were organized for the long (eight to nine different barrel types per wine) and short series (two to three different barrel types per wine), respectively. Twenty sessions corresponded to 20 different wine matrixes used in the study.

Tasting sessions were conducted in standard tasting rooms by a session observer. Before the sensory analysis, each bottle was examined for possible off-flavors and rejected if there was an abnormal odor. The tasters scores were normalized by subtracting the average intensity score of a specific descriptor among the individual series for the same taster from the score of that descriptor intensity for the specific wine. Normalized values were used for statistical analysis.

The training was for identification and scoring individual aromas. On the other hand normalizing the scores was done to measure each tasters range of scores within each set/matrix of wines.

Independent samples t-tests were performed to find the statistical difference between the intensity rating for each descriptor when the same wine was aged in different barrels. Thus, in a three-variable experiment, three comparisons were possible (barrel type-A compared to barrel type-B, barrel type-A compared to barrel type-C, and barrel type-B compared to barrel type-C). In all, 161 pair comparisons were performed.

Chemical analysis

Nineteen wood-derived volatile compounds found in wines were quantified by gas phase chromatography mass spectrometry. These included furanic and pyranic compounds (furfural, 5-hydroxymethylfurfural [5HMF], 5-methylfurfural [5MF], furfuryl alcohol, maltol, and ethylmaltol).

When wine is aged in a barrel, furfural can be converted into furfuryl alcohol. Therefore, we calculated the total furfural concentration as the sum of furfural and furfuryl alcohol.

In addition, two aromatic aldehydes (vanillin and syringaldehyde), nine volatile phenols (guaiacol, 4-methylguaiacol, eugenol, isoeugenol, o-cresol, m-cresol, phenol, syringol, and allylsyringol), and two whisky lactone isomers (trans- and cis-whisky lactones) were examined.[11]

Results and Discussion

Seventy-nine different wine samples were analyzed (Table II). The high variability reflects differences in chemical composition of barrel wood and in extraction and transformation of wood compounds during wine maturation.

The wine matrix (alcohol concentration and pH) and maturation conditions (temperature, dissolved oxygen, and redox potential) can affect extraction of oak compounds.[7] In addition, the wines in different series were sampled at different stages of their maturation, from 6 to 12 months).

The independent samples tests performed on each pair of samples during the sensory sessions allowed us to find pairs with different intensity ratings for descriptors at $p< 0.05$ (Table III).

Both samples in a pair represented the same wine aged in different barrels. Thus, if one sample had a significantly higher intensity rating ($p < 0.05$) for a certain descriptor (e.g. fruity) than the other sample found in the same pair, the difference in the sensory perception and chemical composition was due solely to the barrel and not to the wine itself.

Paired-sample t-tests were run for all chemical variables. In this way we could determine if there were corresponding consistent differences in the chemical composition and the perceived sensory character of the wines aged in different barrels. This design allowed the effect on the aroma of the starting wine itself to be isolated from the effect of the wood. It also allowed a degree of generalization because a range of different wines were analyzed.

The concentrations of specific compounds in samples with higher perceived intensity of a specific descriptor were subtracted from those in their paired samples where this intensity was lower. Thus, the values of differences could be positive or negative.

A positive difference indicates that a higher concentration of that chemical increased the perception of the respective descriptor, while a negative difference decreased it. The standard deviations of differences between samples and their statistical significance were calculated to check whether the differences were consistent across all pairs.[11]

The compounds found to have significant differences only are presented in Table III. No significant difference with any descriptor was found for the following compounds: guaiacol, phenol, ethyl maltol, o-cresol, m-cresol, isoeugenol, syringol, 4-allyl-syringol, syringaldehyde.

Relatively few pairs were judged different for any descriptor. Thus, regardless of the variation in oak-derived chemical composition between wine samples, only certain pairs were different enough to be distinguished in sensory analysis.

Fruity descriptor – No wood-derived compounds reminded tasters of a strictly fruit aroma. Thus, it was not surprising that no wood-derived compounds enhanced the fruity expression. On the contrary, the more fruity samples were characterized by systematically lower concentrations of furfural, total furfural, furfuryl alcohol, and 5-methylfurfural, all typically released through barrel toasting. These compounds have relatively high sensory thresholds: 20 to 65 mg/L for furfural; 35 to 45 mg/L for furfuryl alcohol, and 45 to 52 mg/L for 5-methylfurfural in white and red wines.[3]

The differences in these compounds found between more or less fruity samples, when compared to their perception thresholds, could not be explained by their direct sensory impact. There are several possible explanations.

First, regardless of the low probability of a direct impact by these compounds, they could enhance the action of other compounds acting as masking agents for the fruity character. Second, the aforementioned compounds could be markers for either some unknown potent odorant that masks the fruity character or for some process that occurs in wood during heating and which leads to the loss of fruity character. Finally, it could be that they are precursors of other, more potent, odorant molecules and could mask the fruity aroma.

Examples of such products of

transformation include thiols, which possesses a strong coffee aroma,[2,16,17] and furfuryl ethyl ester, which possesses a kerosene-like aroma.[14] Both compounds can mask fruity aromas. It is possible that any or all of these phenomena occur simultaneously.

Overall oak aroma descriptor – The most potent contributors to an overall oak aroma are compounds related to barrel toasting: vanillin, 5-hydroxymethylfurfural, and total furfural. Cis-whisky lactone was also among these contributors. However, unlike the other three compounds, its concentration was not systematically higher in the more intensely oaky samples (p = 4%).

The role of furanic compounds can be explained in the same way as for the fruity descriptor: they enhanced the oaky flavor and acted as markers and/or precursors for potent odorants perceived as an oak barrel aroma. Vanillin and cis-whisky lactone can also be regarded as direct contributors and/or possible enhancers of this descriptor.

Vanilla/pastry descriptor – The cis- and trans-whisky lactones, eugenol, and vanillin are associated the vanilla descriptor. 4-methyl-guaiacol, furfural, and 5-methylfurfural concentrations were systematically lower in more intense vanilla samples. Based on its high significance (p = 0.01) in the t-test, cis-whisky lactone was the most important contributor to this descriptor. As in a previous study,[1] vanillin contributed toward the intensity of this descriptor, but less significantly.

Trans-whisky lactone and eugenol have a high perception threshold in wine: much higher than the average difference found between the paired samples. Their significance may be explained by a correlation with cis-whisky lactone in wood.[10] The role of furfural and 5-methylfurfural was similar as for fruity descriptor: they used different mechanisms to mask the vanilla/pastry flavor.

Toasty/smoky descriptor – Maltol and 5-hydroxymethylfurfural were associated with the toasty/smoky descriptor. The hypothesis of an indirect impact (enhancer, marker, and precursor) seems most plausible here as well, because maltol has a high perception threshold. None of the volatile phenols studied here (such as guaiacol and 4-methylguaiacol) were perceived as smoky in their pure state. Their weak contribution could be explained by the low variation in their concentrations compared with the perception thresholds in wine.

Spicy descriptor – The impact of wood compounds on the spicy descriptor is rather difficult to explain. However, there was no association between spicy and the concentration of eugenol, described in its pure state as spicy/clove.

Olfactory persistence descriptor – The samples described as having a greater olfactory persistence had higher concentrations of trans- and cis-whisky lactones, maltol, eugenol, and vanillin than their paired samples. This group of compounds was characterized in general by low perception thresholds and a pleasant aroma. In addition, vanillin, whisky lactones, and maltol belong to the group of heavy volatiles (high boiling points)[18] with long release in the buccal cavity during tasting. This fact can also explain retronasal persistence.

Conclusion

Twenty different wines, each aged in nine different barrel types, were studied using both sensory (descriptive) and chemical analysis. Comparisons were made using paired tests on the same wine matrix. Thus, the differences in sensory perception and chemical composition reported in the study were solely due to the impact of different barrels and not to the wine itself.

Furanic compounds (furfural, furfuryl alcohol, and 5-methylfurfural) increased the overall oak intensity and decreased the fruity intensity. The presence of cis- and trans-whisky lactones, eugenol, and vanillin raised the intensity of the vanilla/pastry descriptor, while furfural and 5-methylfurfural diminished it. Thus, furanic compounds, often judged as unimportant because of their low OAVs, definitely had a strong sensory impact.

An indirect impact as markers, enhancers, or precursors of some unknown or known odorants (such as furfyl thiol, 5-methyl-2-furanmethanthiol, or furfuryl ethyl ester) masked the fruity and vanilla/pastry aromas.

Some volatile phenols (guaiacol, 4-methylguaiacol, and eugenol) described as smoky and spicy in their pure state and which have low sensory thresholds, in their pure state, were not consistently linked to their respective sensory descriptors in wines.

Samples described as having a higher olfactory persistence were richer than their paired samples in relatively highboiling wood compounds such as trans- and cis-whisky lactone, maltol, eugenol, and vanillin, explaining their retronasal persistence. ■

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TABLE I: Origin of the wine samples studied.

Wine n.	Vine area	Variety	Vintage	N. of barrel types	N. of pairs of comparison	Origin of difference between barrel types
1	Rhone Valley	Syrah	2004	3	3	M, M+, and ML toasting
2	Bordeaux	Cabernet Sauvignon	2005	9	36	M, M+, and ML toasting, toasted and non-toasted heads, medium and tight grain
3	Bordeaux	Cabernet Sauvignon	2005	9	36	M, M+, and ML toasting, toasted and non-toasted heads, medium and tight grain
4	Bordeaux	Cabernet Sauvignon	2005	3	3	M, M+, and ML toasting
5	Bordeaux	Cabernet Sauvignon	2005	3	3	M, M+, and ML toasting
6	Bordeaux	Cabernet Sauvignon	2006	3	3	M, M+, and ML toasting
7	Bordeaux	Cabernet Sauvignon/ Merlot	2007	3	3	M, M+, and ML toasting
8	Languedoc	Chardonnay	2006	2	1	water bending / fire bending
9	Languedoc	Chardonnay	2006	2	1	water bending / fire bending
10	South-West	Merlot/Tannat	2006	4	6	M, M+, and ML toasting; toasted and non-toasted heads
11	South-West	Merlot/Tannat	2007	4	6	M, M+, and ML toasting; toasted and non-toasted heads
12	Somontano*	Cabernet Sauvignon/ Tempranillo	2006	8	28	M, M+, and ML toasting; toasted and non-toasted heads
13	South-West	Merlot	2006	5	10	M, M+, and ML toasting, medium and tight grain
14	Bordeaux	Merlot	2007	3	3	M, M+, and ML toasting
15	Bordeaux	Merlot	2007	4	6	M, M+, and ML toasting, medium and tight grain woods
16	Bordeaux	Merlot	2007	2	1	M, ML toasting
17	Burgundy	Chardonnay	2007	3	3	M, ML toasting; water and fire bending
18	Burgundy	Chardonnay	2007	3	3	M, ML toasting; water and fire bending
19	Burgundy	Chardonnay	2007	3	3	M, ML toasting; water and fire bending
20	Burgundy	Pinot Noir	2007	3	3	M, ML toasting; water and fire bending
			TOTAL	79	161	

Note: Origin of wine – France, excepting * - Spain.; M – medium toasting; M+ - medium plus toasting; ML – medium long toasting

TABLE II: Summary of chemical analysis of wines under experimentation (79 samples analyzed).

	Concentration (µg/L)			
	Min	Max	Avg	SD
Furfural	6	5967	1043	1753
5-Methyl-furfural	1	822	179	185
Furfuryl alcohol	80	23536	2840	3425
Guaiacol	6	40	17	7
Trans-whisky-lactone	1	186	30	37
Cis-whisky-lactone	48	1001	255	160
Maltol	0	169	71	39
4-Methyl-guaïacol	3	22	10	5
Phenol	3	122	13	17
Ethyl maltol	0	7	2	2
o-Cresol	0	4	2	1
m-Cresol	1	158	7	21
Eugenol	4	60	28	14
Isoeugenol	1	127	17	21
Syringol	11	488	65	74
5-Hydroxy-methylfurfural	19	3979	665	838
4-Allyl-syringol	4	300	46	52
Vanillin	13	506	201	104
Syringaldehyde	71	1441	612	337
Total furfural	187	15770	3882	4728

TABLE III: Differences and significances of paired-samples T-test.

Compound	Fruity (22)*	Overall woody (19)	Vanilla/ pastry (28)	Toasty/ smoky (22)	Spicy (17)	Olfactory persistence (23)
	Difference					
Furfural	-	n.s.	-	n.s.	n.s.	n.s.
5-methyl-furfural	-	n.s.	---	n.s.	n.s.	n.s.
Furfuryl alcohol	-	n.s.	n.s.	n.s.	n.s.	n.s.
***Trans*-whisky-lactone**	n.s.	n.s.	+	n.s.	+	+
***Cis*-whisky-lactone**	n.s.	+	+++	n.s.	n.s.	++
Maltol	n.s.	n.s.	n.s.	+	-	+
4-Methyl-guaiacol	n.s.	n.s.	-	n.s.	n.s.	n.s.
Eugenol	n.s.	n.s.	++	n.s.	n.s.	++
5-Hydroxy-methylfurfural	n.s.	++	n.s.	+	n.s.	
Vanillin	n.s.	+++	+	n.s.	n.s.	+
Total furfural	-	+++	n.s.	n.s.	n.s.	n.s.

Notes:

*** –** Number of pairs significantly different after sensory test for descriptor given.

"+" – significant positive difference between concentration of compound in wine having higher sensory score and having lower sensory score, i.e. positive correlation between compound and descriptor.

"-" – significant negative difference between concentration of compound in wine having higher sensory score and having lower sensory score, i.e. positive correlation between compound and descriptor.

Number of "+" or "-" shows the statistical strength of correlation.

n.s. –- non-significant difference (p > 5%).