How extraction of oak volatiles and ellagitannin compounds affect sensory profile of wine aged with French winewoods

BY Kleopatra Chira and Pierre-Louis Teissedre, University of Bordeaux, INRA, ISV, Villenave d’Ornon, France

Oak barrels have long been used in fine winemaking—initially for easy handling of wine during production, storage and transport. Oak became accepted for its positive effects on wine development, namely to provide pleasant aromas and the stabilization of red wine color.

During aging in oak barrels, wine composition changes because of the addition of phenolic compounds and other molecules extracted from the wood. 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.

The sensation imparted by ellagitannins was described as astringent at relatively low concentrations spanning from 0.2 to 6.3 μmol/L, whereas bitterness was strongly dependent on ellagitannin structure (a bitter taste was perceived at threshold concentrations between 410 and 1,650 μmol/L). C. Chira et al. has observed that ellagitannin levels in a model wine solution are 45% of the total astringency variance.

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 β-methyl-γ-octalactone, furfural and its derived compounds, phenolic aldehydes such as vanillin and syringaldehyde, and volatile phenols such as eugenol, guaiacol and ethyl- and vinylphenols. Their extraction from oak barrels depends mainly on wine composition and the quantity of compounds that are potentially extractable during the contact time between wine and oak.

At present, alternatives to the oak barrel are being evaluated to carry out the wine-aging process. This practice was approved and legislated by the European Community in 2006. (CE 2165/2005 and CE 1507/2006)

Different shapes of oak wood pieces are used: chips, cubes or beans, powder, shavings or granulates, dominoes and blocks or segments. Factors such as amount of wood added and contact time between wood and wine affect both sensory and chemical wine characteristics — especially their wood-related volatile composition.

Until recently, it has not seemed very logical to establish an aging period in barrels or with wood pieces through legislation. Hence, it would be important to know more about influence of the wine composition in the process of the extraction of oak compounds.

A study of wine volatile and non-volatile composition along with a tasting assessment would be a more efficient method to establish the optimum contact time between wine and oak wood.

The objectives of this study were to define the chemical (ellagitannins and volatile composition) and sensory characteristics of wine treated with winewoods (staves) representing different toasting methods with the aim to monitor the kinetic levels of extraction of the above compounds for 12 months.

The staves are samples (100 x 11 x 0.12 cm) from Quercus robur oak species from a forest in the center of France. After 24 months of natural seasoning in the Tonnellerie Nadalié seasoning park (Ludon-Medoc, France), they were submitted to different toasting processes according to the desired final product using a revolving drum and oak fire (Table I).

The toasting level impact on both volatile and non-volatile compounds and sensory perception were studied in parallel. The relationship between the chemical composition and the sensory assessment of oak also was investigated.

Oak wood volatile composition

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; the aldehyde

<table>
<thead>
<tr>
<th>Table I: Tasting Characteristics</th>
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<tr>
<td><strong>Oak toast</strong></td>
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<tr>
<td>LT (light toast)</td>
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<td>MT (medium toast)</td>
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<tr>
<td>M+ (medium-plus toast)</td>
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<td>Noisette</td>
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<td>Special (medium toast with watering)</td>
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The levels of oak volatile compounds extracted were quantitatively different depending on contact time and type of toast (Figure 1).

Figure 1 shows that furfural and 5-methylfurfural reached maximum concentration either at three or six months of aging. In all samples furfural content was exhausted after 12 months. “Noisette” was toasted two hours longer than medium toast (MT) staves and produced more furfural and methyl-furfural. “Special” (oak wood heated using medium temperature with watering) produced almost double the methyl-furfural compared to MT. Hence, along with the length of toasting time, the watering process has an important impact on furanic compounds.

Regarding phenolic alcohols, the maximum extraction of guaiacol occurred...
during the first nine or 12 months (Figure 1), and the concentration of 4-methyl-guaiacol was less important.

An increase in eugenol content is observed during the maceration time for all wines. Wines with light toast (LT) staves presented the highest concentrations after 12 months. Both lactones increased linearly in concentration in wine during the oak maturation period (Figure 1), the rate of extraction increasing further from six to nine months, meaning that once a portion of wood is wetted, dissolution of lactones occurs rapidly.

Among phenolic aldehydes, vanillin is considered to have the most important influence on wine aroma. Just as happens with furanic aldehydes, for short aging 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.2 The biggest concentration is found in the wine with Noisette staves (305.81 µg/L). Similarly to furanic compounds, the longer MT toasting time increases vanillin content.

**Oak wood total ellagitannin concentration**

The total ellagitannin level, expressed as milligrams (mg) per liter of released ellagic acid of wine, revealed a large diversity of concentration from 6.31 to 26.07 mg of released ellagic acid per liter (L) of wine (Figure 2). Such differences were expected since ellagitannins undergo thermolytic degradation during the toasting process.4

Figure 2 shows that, for almost all wines, a maximum extraction of ellagitannins is obtained after two to three months. In all wines, after nine and 12 months of contact, the overall concentration of ellagitannins decreased with time. After 12 months, a 10%–20% decrease was observed in ellagitannins levels for
wines with LT oak staves, a 30% loss for wines with Noisette, a 50%–60% reduction for wines with MT, MT+ oak staves and the largest decrease of about 70% for wine with Special staves.

In the first months of a Merlot trial in Medoc, France, ellagitannins were extracted at a rate faster than the rate of the condensation reactions between ellagitannins and other nucleophilic wine constituents (catechin, epicatechin and ethanol). When most of the ellagitannins have been extracted from the first millimeter of the wood, the red wine goes deeper into the wood to extract more ellagitannins (consequently, at a slower rate).

Not only do the toasting stages diminish the quantity of these compounds but also the watering procedure. Wine with light toast staves extracts not only more ellagitannins but also faster than other samples. The above decreases during the maceration time can be attributed to the high reactivity of ellagitannins toward other wine constituents.8,10

Oak wood sensory evaluation

Figure 3 shows the average intensity of each gustatory and olfactory attribute during the different contact times of the control wine and the same wine treated with oak wood for 12 months. The wines treated with toasted oak staves become less astringent and bitter during the contact time. This reduction in the astringency sensation could be attributed to their lower ellagitannins levels and the chemical complex formation between wine tannins, polysaccharides and peptides brought out by the oak.

For all wines treated with toasted oak staves, it is observed that vanilla, spicy and woody characters increase along with sweetness. The woody aroma of wines aged with staves toasted in the “Special” method was more intense after two months of contact, afterward it did not intensify significantly. Therefore, judges found that woody, spicy and vanilla aromas do not fluctuate during the maceration time for wines treated with Special toasted staves. The watering process during the toasting process influences not only the level of hydrosoluble tannins but also a wine’s sensory profile.

Correlations between sensory and chemical results

The analysis of variance revealed that the toasting method has a significant impact (p less than 0.05) on chemical composition and sensory perception of oak wood extract. Each sensory descriptor was correlated with the chemical concentration of the oak wood compounds of interest (Table I).

This procedure allowed us to measure the extent to which sensory and chemical variables are correlated. Based on the correlation analysis, astringency and bitterness intensify significantly with ellagitannins concentration (R = 0.828 and p = 0.001 for astringency, while R = 0.607 and p = 0.003 for bitterness).

The highest astringency and bitterness was perceived for wines with LT oak staves (4.98 for astringency and 4.67 for bitterness) containing 27.33 mg/L of released ellagic acid. The above established correlations permit characterization of tannin quality when ellagitannin levels are known. Additionally, astringency and bitterness were more per-
ceived in the wines with significant levels of furanic compounds (Table II).

Table II: Pearson correlations between chemical and sensory data. Marked correlations are significant at p < 0.05. NS = not significant difference; *, **, *** Significant differences at 5, 1 and 0.1% respectively.

On the other hand, wines with a high content of guaiacol, methyl-guaiacol, syringol and syringaldehyde were characterized as less astringent. This observation is logical as toasting decreases ellagitannins and increases the concentration of volatile compounds that enhance the oak aroma.

Judges perceived the sweetness perception more intensely 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 II).

Vanillin is the principal marker of vanilla smell. Lactones can be regarded as direct contributors and/or possible enhancers of this descriptor.

Overall woody character is positively correlated to guaiacol, methyl-guaiacol, eugenol, syringaldehyde, lactones and vanillin levels, which is reasonable since oak evaluation is complex and influenced by the presence of various odor-active wood extractives.

For example, whisky lactone is an attribute that accounts for a woody and coconut character, while high concentrations of this compound are associated with wine with an intense vanilla perfume. Perceived spicy intensity is closely related to eugenol content, which is logical, since pure eugenol is described as clove-like.

In our experiment, it is also linked positively to the presence of other odorous chemicals such as lactones, vanillin and methyl guaiacol, suggesting that in a complex medium such as wine the above volatile compound values may influence spicy aroma by means of additive or synergistic effects. An important reduction in spicy and vanilla aroma occurs in wine with a significant level of furfural.

Conclusions
The results have shown that each oak stave adds unique and special characteristics. Each sample shows a different ability to extract the 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) and on the watering process during toasting. These differences were reflected by perceived sensory differences.

In general, volatile phenols such as eugenol and guaiacol, along with aldehydes, phenols (vanillin) and lactones, showed an increasing tendency with longer maceration time. Ellagitannins are extracted faster during the first three months; after six months a decrease is observed.

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Wine aged with “Special” toasted staves presented the lowest ellagitannin concentration in comparison with other wood-toasting trials. Concerning sensory evaluation, oak wood contact time enhances vanilla, spicy, and woody characters and sweetness perception. For all the studied samples, with the exception of wines with “Special” oak staves, vanilla and spicy flavors amplify linearly during the storage time.

Wine treated with “Special” oak staves did not show substantial changes in the evolution of aromas during maceration time. Wine storage with winewood has a sweetening effect and, in parallel, decreases the astringency and bitterness sensation. Astringency and bitterness are related significantly to ellagitannin levels ($R = 0.828$, $p = 0.001$ for astringency, and $R = 0.607$ and $p = 0.005$ for bitterness).

A model like this satisfactorily predicts the sensation intensity of both astringency and bitterness if ellagitannin levels are known. Additionally, relationships between volatile oak compounds and sensory perception are found. The above correlations obtained between sensory descriptive evaluation performed by a trained panel and wine chemical characterization resulted in a useful tool applicable to wine development.

Trials kits are available for customers. These kits use 3-liter bag-in-box wines in which sample sticks (samples of staves) are soaking for two weeks to permit evaluation of different toasting profiles.

**Bibliography**