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Maturing Wines in Oak Barrels. Effects of Origin, Volume, and Age of the Barrel on the Wine Volatile Composition

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The influence of the oak origin, volume, and age of the barrel on the volatile composition of a red wine after six months of maturation have been studied. Major volatile compounds in wine were determined using liquid−liquid extraction with dichloromethane, and those volatiles being due to the oak were determined by extraction with pentane-ether using deuterated analogues as internal standards. The results show that there is little difference between the volatile composition of wines matured in French oak barrels and those matured in American oak barrels. The concentration of *cis*-oak lactone was the most significant difference. The greatest instrumental and sensory differences were found between new and used barrels, with important decreases in lactones and vanillin concentration in used barrels. When studying the effect of the volume of the barrel, the characteristics of the wine matured in 220-L new barrels versus those matured in 1000-L new barrels were very different. In new barrels, the larger the volume of the barrel, the lower the concentrations of those compounds responsible for the oaky aroma. These differences were less evident when comparing wines matured in used barrels with different capacities.

KEYWORDS: Oak; wine; volatile compounds; maturation

INTRODUCTION

Oak barrels have been commonly used in the aging of wine and spirits because of the positive effects they have. These include increased color stability, spontaneous clarification, and a more complex aroma.

The complexity of the aroma is increased because of the extraction of certain compounds present in the wood which are transferred to wine during the aging period, although reactions involving only wine compounds and evaporation of volatile compounds can also occur during oak barrel maturing (*1*). Among the compounds released from the wood, and from a sensory point of view the most important compounds, are the oak lactones (*cis*- and *trans*-β-methyl-γ-octalactones) that are present in green wood (*2*, *3*). They arise from lipid oxidation and are known to increase in concentration after the wood is toasted (*4*). Other important compounds are guaiacol and 4-methylguaiacol, with a smoky aroma, which are lignin degradation products formed during toasting, and vanillin, a compound normally present in green wood but whose concentration is increased by seasoning and toasting (*4*, *5*).

Other compounds that can be of sensory importance are the furfuryl compounds (furfural, 5-methylfurfural, and furfuryl alcohol). These compounds with a pleasant aroma are formed from the degradation of hemicellulose during toasting (*6*−*8*).

Some economic factors may influence the decision about buying a particular sort of barrel. One factor which is important from the economic and sensorial point of view is the origin of the wood. From the economic point of view, French oak barrels cost twice or more the price of American oak barrels. The difference is partly due to the greater losses involved in coopering French oak because of its more irregular grain structure and higher wood porosity. Thus, the wood must be split instead of sawn (*9*). When splitting it is impossible to obtain more than 25% of staves from a log, but sawing can give more than 50% (*10*). Because of the economic importance of the choice, a number of studies regarding the differences in extractable compounds have been done (*9*−*20*). The quantitative differences among some of these compounds have also been used in the classification of wood samples according to wood origin (*21*).

Another important factor is the expected life of a barrel, i.e., the time it can be used while still having the positive effects of oak aging on wine. Although several factors such as wine composition, barrel maintenance techniques, or barrel fermentation can affect the extraction process, the age of the barrel is of primary importance in the quality of wines (*22*), and decides the renovation rate of barrels in a winery. Because the pool of extractable compounds in oak is finite, the rate of extraction and the amounts of compounds extracted diminish as the barrel

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is used in successive years (*22*, *23*). In addition, as the barrel becomes older, it might become populated with undesirable microorganisms such as Brettanomyces which may produce sensorially significant concentrations of ethyl phenols with their unpleasant medicinal and horsy aromas. The most important of these are 4-ethylphenol and 4-ethylguaiacol, which are formed from grape-derived *p*-coumaric and ferulic acids (*24*). Inadequate sanitation, high wine pH, and low SO_2 concentrations are among the conditions that encourage the growth of Brettanomyces.

In the past, barrels and vats with a volume of 5 to 10 hL were used. They also acted as storage cooperage following fermentation. However, wooden fermentors and storage tanks have been largely replaced by more durable and easily cleaned tanks whose standard construction materials includes stainless steel, epoxy-lined carbon steel, fiberglass, and cement. Wooden cooperage is primarily retained for maturing wines in which an oak character is desired. Many of the benefits of barrel use come from the relatively large surface area of the barrels. Production economy favors a larger size, whereas ease of movement and earlier maturation favors a smaller size. A compromise between these opposing factors has led to the widespread adoption of barrels with a capacity of between 200 and 1000 L. Although much of the literature focuses on the value of maturing wine in small oak barrels, many fine wines are aged in mid-size to large (more than 1000 L) oak barrels. Large oak barrels also can be used for longer time whereas small barrels are usually replaced after a few years. In Murcia region (southeast Spain), most of the wineries use higher volume barrels and not the typical 220-L barrels. The four kinds of maturation effects attributed to barrels (evaporation, extraction, oxidation, and component reaction) would be intensified with increased wood surface in contact with a unit of beverage (*25*).

The objective of this study is to ascertain the effects of wood origin, barrel age, and barrel size on the volatile compounds of Monastrell wines elaborated in southeast Spain, with special attention paid to economic and sensorial aspects.

MATERIALS AND METHODS

Barrels. The twelve new and twelve used barrels used in this experiment were made of American white oak and French oak (*Quercus petraea* from the Allier forest in France) and were obtained from the same cooperage firm in Spain with the same specifications. The used barrels had been used three times for the aging of Monastrell wine. The following barrels were used in the experiment: 220-L French oak barrels (3 new and 3 used); 220-L American oak barrels (3 new and 3 used); 500-L American oak barrels (3 new and 3 used); 1000-L American oak barrels (3 new and 3 used).

Wine. The wine used in this experiment was a 1998 Monastrell red wine vinified by Bodegas San Isidro in Jumilla, Murcia. All barrels were filled with the same wine. Wine samples were taken after six months of aging. Duplicated samples were taken from each barrel.

Aroma Compounds Determination. For the determination of the aroma compounds two different methods were followed. The method described by Cocito et al. (*26*) and Gómez-Plaza et al. (*27*) was used for the determination of alcohols, esters, acids, and furfuryl compounds. Thus, 50 mL of wine in a 200-mL spherical flask with 15 mL of dichloromethane were extracted by means of ultrasound over 10 min at 20 °C. 2-Octanol was used as internal standard. After separation, the organic layers were dried with anhydrous sodium sulfate and transferred to a tube. Second and third extractions were performed with 5 mL of dichloromethane, and the organic layers were collected in the same flask. The extracts were concentrated to a final volume of 0.5 mL under a N_2 stream and analyzed using a GC−MS system. The concentrated extract was injected into a Hewlett-Packard 5890 gas chromatograph (Hewlett-Packard, Avondale, PA). Separations were performed using an HP-20 M column (25 m × 0.25 mm, Hewlett-Packard). The oven temperature was programmed from 60 °C to 210 °C (4 °C/min). Helium was used as the carrier gas at an average linear velocity of 60 cm/s. The injector was maintained at 200 °C and the FID detector was set at 250 °C. For the identification of compounds, a Hewlett-Packard 5890 gas chromatograph coupled with a Hewlett-Packard 5971 A mass-selective detector was used. The chromatographic separations were performed under the same conditions described above.

The determination of guaiacol, 4-methylguaiacol, *cis*- and *trans*-oak lactone, vanillin, 4- ethylphenol, and 4-ethylgualacol in wine was made by gas chromatography−mass spectrometry using deuterated analogues of guaiacol, 4-methylguaiacol, *cis*- and *trans*-oak lactone, vanillin, and 4-ethylphenol as internal standards, following the method described by Pollnitz and Spillman (*5*, *28*, *29*). A solution of precisely known concentration of internal standards $^2H_3$-guaiacol (5 μg/mL), $^2H_3$-4-methylguaiacol (5 μg/mL), $^2H_4$-*cis*-oak lactone (19 μg/mL), $^2H_4$-*trans*-oak lactone (31 μg/mL), $^2H_3$-vanillin (25 μg/mL), and $^2H_4$-4-ethylphenol (25 μg/mL) in ethanol was prepared. The deuterated standards were donated by Dr. Pollnitz and Dr. Sefton from the Australian Wine Research Institute.

The standard solution (100 μL) was added to the sample (5 mL of wine) in a screw-cap vial using a glass syringe (100 μL, Hamilton). The organic solvent (2 mL of pentane/ether, 2:1) was added, and the mixture was shaken briefly. A portion of the organic layer was then placed in a vial ready for instrumental analysis.

Samples were analyzed with a gas chromatograph coupled with a mass spectrometer (Thermoquest Tracer-MS). The gas chromatograph was fitted with a 30 m × 0.25 mm Carbowax 20M column. The carrier gas was helium, and the flow rate was 1.1 mL/min. The oven temperature was started at 90 °C and then increased to 240 °C at 10 °C/min. The injector was held at 250 °C, and the transfer line was set at 280 °C. The injection volume was 2 μL, and the splitter, at 30:1, was opened after 30 s.

Aroma Compounds Quantification. For quantification of the compounds, mass spectra were recorded in the selective ion monitoring (SIM) mode. The ions monitored in SIM runs were the following (underlined ions are those used for quantification): (1) *m*/*z* 81, 109, *124*, and *127* for guaiacol and $^2H_3$-guaiacol; (2) *m*/*z* 95, 123, *138*, and *141* for 4-methylguaiacol and 4-methyl-$^2H_3$-guaiacol; (3) *m*/*z* 90, *99*, *101*, 114, 118, 128, 132, 156, and 160 for *cis*- and *trans*-oak lactones and *cis*- and *trans*-$^2H_4$-lactones; (4) *m*/*z* 107, 111, *122*, and *126* for $^2H_4$-4-ethylphenol and 4-ethylphenol; (5) *m*/*z* 122, 137, and *152* for 4-ethylguaiacol; and (6) *m*/*z* 151, *152*, *155*, and 156 for vanillin and $^2H_3$-vanillin.

Statistical Analysis. A multivariate analysis of variance (MANOVA) was performed to study the effects of oak origin, barrel volume, and barrel age on all the constituents measured in the wines. Significant differences among wines and for each variable were assessed with analysis of variance (ANOVA). These statistical analyses were performed using Statgraphics 2.0 Plus.

Sensory Analysis. For the descriptive analysis, five trained judges were selected. The intensity of each attribute was rated on a scale of zero to ten, with a score of zero indicating that the descriptor was not perceived. Data from all judges for all samples were used in the analysis. An analysis of variance was run to test the significance of the effect of oak origin, volume, and age of the barrel.

RESULTS AND DISCUSSION

Figures 1 through **3** show the concentrations of the studied compounds and the effect of the variables (origin, volume, and age of the barrels) on those compounds. Higher alcohols, acids, esters, and furfuryl compounds (the sum of furfural and furfuryl alcohol) were determined by liquid−liquid extraction by means of ultrasound, while guaiacol, 4-methylguaiacol, 4-ethylguaiacol, 4-ethylphenol, vanillin, and oak lactones were determined by the stable isotope dilution method developed by Spillman et al. (*28*) and Pollnitz et al. (*24*, *29*) because of the greater accuracy and simplicity of this method.

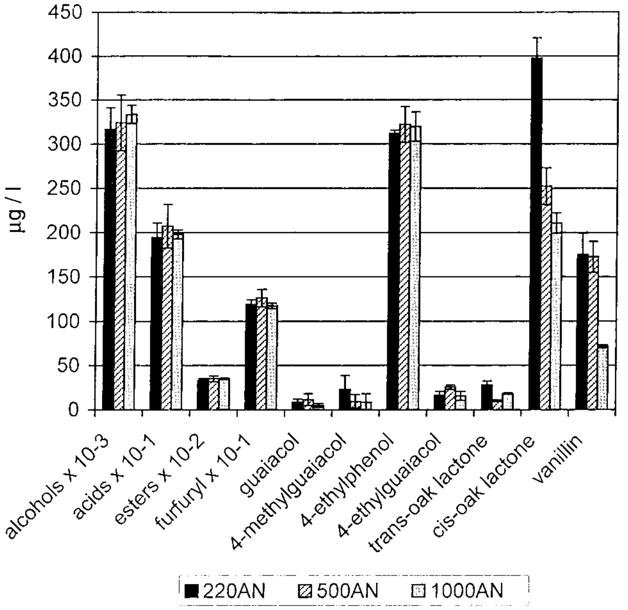

Figure 1. Concentrations of volatile compounds in wines from 220-, 500-, and 1000-L new American oak barrels.

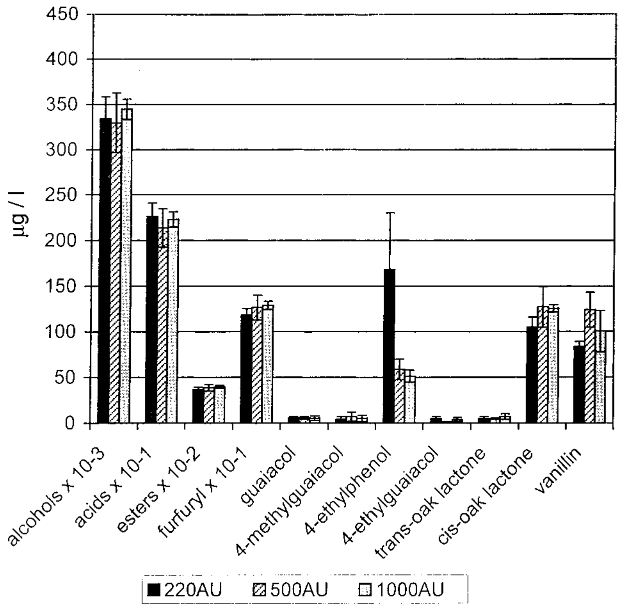

Figure 2. Concentrations of volatile compounds in wines from 220-, 500-, and 1000-L used American oak barrels.

Some important differences were found among wines matured in identical barrels. Winemakers are aware that the same wine stored in barrels from the same oak and purchased as a simple batch from the same cooper may differ significantly from barrel to barrel (*12*). It has even been found that great variability exists between trees from the same forest (*20*, *30*). Barrels are constructed from wood from several trees and this is likely to reduce variation among barrels (*31*). Nonetheless, Towey and Waterhouse (*32*) reported a coefficient of variation of 31% in the level of lactones among barrels from the same lot and procedence.

Table 1 shows the three-way analysis of variance used to study the effect of barrel volume, origin, and age on the volatile compounds of the wine.

New Barrels versus Used Barrels. The analysis of variance (**Table 1**) revealed that, among those compounds which are formed during fermentation, only acids and esters showed significant differences, appearing at higher levels in wines aged in used barrels. Such differences could be due to the lower degree of evaporation taking place in used barrels because of their lower porosity: most of the pores having been plugged by previous deposits of mineral salts and color pigments. However, Towey and Waterhouse (*22*) found that the volatile compounds which are fermentation products are not affected by oak aging.

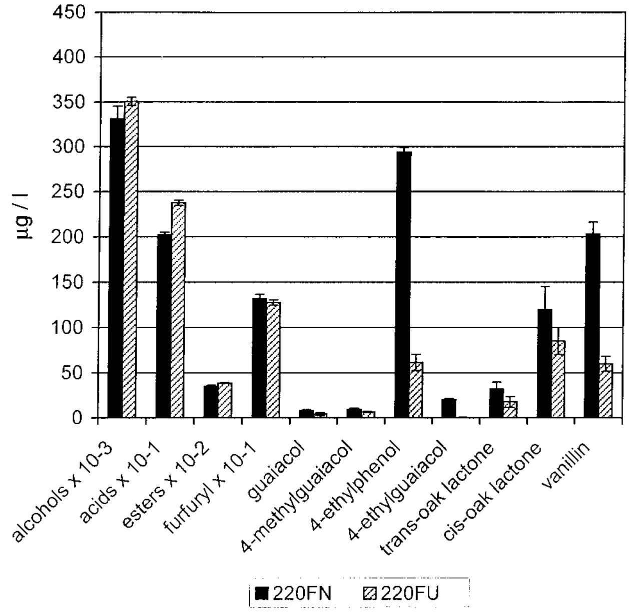

Figure 3. Concentrations of volatile compounds in wines from 220-L new and used French oak barrels.

Among those compounds extracted from wood, no significant differences were found in furfuryl compounds and 4-methylguaicol. All the other compounds appeared in lower concentrations in wines from used barrels. The most significant decrease was seen in the concentration of lactones, which are one of the most important compounds from the sensory point of view.

We also found differences in 4-ethylphenol and 4-ethylguaiacol levels, which were lower in wines from used barrels. Several authors have reported that these compounds appear in higher concentrations in wines stored in used barrels because their formation is due to the action of the Brettanomyces/Dekkera yeast that commonly develops in used barrels (*33*). However, this alteration may also occur in new barrels (*34*) or even in anaerobic conditions (bottled wines). The higher ethyl phenols content in wines from new barrels (although below the detection limit mentioned by Chatonnet et al. (*34*)) could have been due to improper sanitation of the new barrels accompanied by low levels of free SO_2. New barrels were merely rinsed with hot water and filled with the wine, whereas the used barrels were sanitized by burning sulfur inside them, a treatment which has been proved a better system for avoiding yeast contamination.

The results of the sensory analysis (**Figures 4** to **6**) pointed to significant differences between almost all the descriptors when wines from new and used barrels were compared. Woody, vanilla, spicy, and cedar notes were higher in wine from new barrels, and pharmaceutical, herbaceous, and horsy notes were higher in wines from used barrels, even though lower concentrations of the ethyl phenols were found in wines from used barrels.

Effect of the Volume of the Barrel. Very small differences were found in those compounds arising from fermentation.

Table 1. Mean Values for Volatile Compounds According to Multivariate Analysis of Variance[a]

effects		alcohols	acids	esters	FC	G	4-MG	TL	CL	EP	EG	V
volume	220 L	333.2	2.1	35.8 a	1.2 a	6.5	10.7	20.6 b	176.8 b	208.8 b	10.4	130.4 b
	500 L	334.5	2.1	37.7 ab	1.3 b	8.2	5.4	11.6 b	115.2a	159.1 a	12.9	149.3 b
	1000 L	346.8	2.1	38.2 b	1.3 b	4.7	4.2	16.9 a b	93.6 a	154.3a	9.4	87.1a
origin	American	331.4	2.1	36.2	1.2 a	6.9	9.5	12.2 a	203.2 b	205.2 b	11.2	121.3
	French	344.9	2.2	38.2	1.3 b	6.1	4.0	20.6 b	54.2 a	142.8a	10.6	123.4
age	new	330.4	2.0 a	35.3 a	1.3	8.0 b	10.2	23.2 b	195.7 b	287.5 b	19.4 b	154.2 b
	used	345.9	2.3 b	39.2 b	1.3	4.9 a	3.3	9.6 a	61.4 a	60.6 a	2.3 a	90.5 a

[a] Results for alcohols, esters, acids, and furfuryl compounds (the sum of furfural and furfuryl alcohol, FC) are expressed in mg/L. Results for other compounds are expressed as μg/L: G, guaiacol; 4MG, 4-methyl guaiacol; TL, *trans*-oak lactone; CL, *cis*-oak lactone; EP, 4-ethyl phenol; EG, 4-ethyl guaiacol; V, vanillin. Different letters mean significant differences ($p < 0.05$).

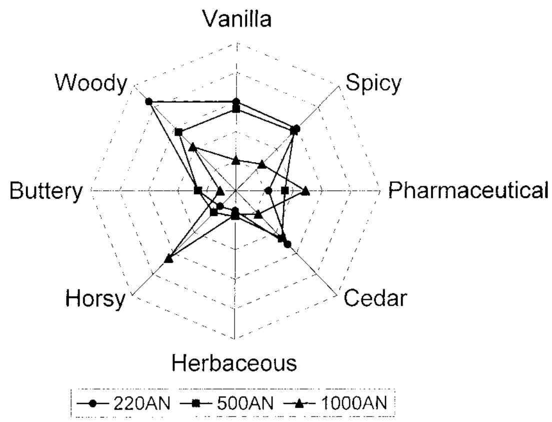

Figure 4. Sensory scores of wines from 220-, 500-, and 1000-L new American oak barrels.

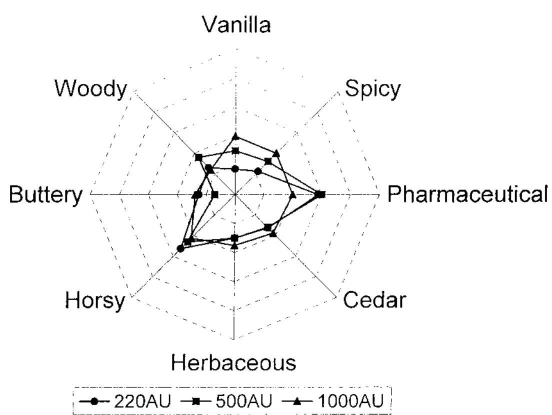

Figure 5. Sensory scores of wines from 220-, 500-, and 1000-L used American oak barrels.

Among those compounds extracted from the wood, significant differences were found in oak lactones, 4-ethylphenol, and vanillin, with the highest differences being between wines matured in 220-L and 1000-L barrels: the concentrations of these compounds were lower in the latter.

Figure 1 compares wines matured in new barrels of different volumes. The decrease in *cis*-oak lactone and vanillin when volume increases is clearly observed; similar results were also found by Puech (*35*). However, it is important to know what happens in used barrels (**Figure 2**). Comparing wines from new and used 220-L American oak barrels, we found that the difference in *cis*-oak lactone concentration was 74%, but when the same compound was analyzed in new and used 500- and 1000-L American oak barrels, the decreases in *cis*-oak lactone concentration were 50% and 41%, respectively. Jackson (*36*) stated that the larger the volume of the barrel, the longer the life of the barrel. Even more, the concentration of *cis*-oak lactone found in wines from 220-L used barrels is lower that those found in 500- and 1000-L used barrels.

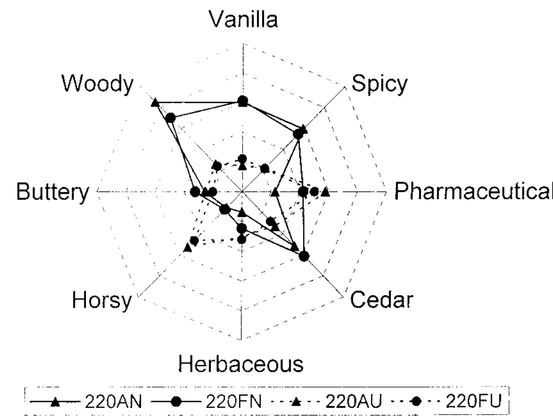

Figure 6. Sensory scores of wines from new and used 220-L American and French oak barrels.

The sensory analysis pointed to no differences in descriptors except for the woody character for wines from new barrels. Very little differences were found between 220- and 500-L new barrels. When comparing used barrels, no significant differences were found in any of the descriptors, and even more, scores were slightly higher in the larger volume barrels.

American versus French Oak. The compounds mainly affected by oak origin are the oak lactones. The cis isomer has a threshold value of 92 μg/L (*6*). This is the important isomer, with aromas described as oaky, coconut, or vanilla at low concentration (*6*). Again, the main difference was observed in *cis*-oak lactone, and, as it can be seen in the figures, it was almost four times higher in wines from American oak barrels than in wines from French oak barrels, although the differences were less in old barrels (20% of difference). Vanillin levels were higher in wines matured in French oak barrels. Different results have been described by other authors (*12*, *15*), who found higher levels of vanillin in wines stored in American oak barrels; although the findings of Spillman et al. (*28*) reflected ours.

The sensory analysis (**Figure 6**) showed no differences in descriptive characters: woody character was higher in wines from American oak barrels but the differences were not significant when the analysis of variance was applied to the sensory scores (data not shown).

We can conclude that the volatile compounds of wines are affected by the variables involved in the oak maturation period. Small new barrels endowed wines with a higher content in oak volatile compounds. Maturation in American oak barrels led to higher levels of *cis*-oak lactone than in French oak barrels, although no significant differences were found in woody character in the sensory analysis. It should also be pointed out that although oak volatile compounds were extracted more intensely in small new barrels, larger barrels proved to be more interesting as they became older, since the results showed that, from the aromatic point of view, extended life could be expected from larger volume barrels.

ABBREVIATIONS USED

AN, New American oak barrels; AU, used American oak barrels; FN, new French oak barrels; FU, used French oak barrels.

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