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Influence of Oak Wood on the Aromatic Composition and Quality of Wines with Different Tannin Contents

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The aromatic compounds associated with aging in wood have been studied in Monastrell wines containing different quantities of tannin. Analyses were carried out before and after a six month period in wooden barrels (225 L) of American and French (Allier) medium-toasted oak. Three classes of wine were made: one from a free-run must; one to which enological tannin was added; and one to which was added the wine obtained by pressing the grape pomaces. The aromas were determined by GC−MS, and the quality was evaluated by a group of expert tasters. The aromatic compounds, some of which came from the wines themselves and others which came from the wood, all increased in concentration by the end of the aging process. The ratio between *cis*- and *trans*-whiskylactone contents in wines reflected the different types of oak wood used. The note "vanilla" used by the tasters is probably not totally due to the presence of vanillin. The wines of best quality were those with a natural tannin content which had been aged in American oak, whereas the wines to which enological tannin had been added but aged in the same wood were considered the worst. An equalizing effect on the sensorial wines qualities was seen to result from the use of French oak. The furfural, eugenol, and *cis*-, and in some cases *trans*-, whiskylactone contents identified the wines from the different classes of wood. The wood from which the barrels were made had a greater effect on wines differentiation than the tannin content.

KEYWORDS: Wine; aroma; aging; oak wood

INTRODUCTION

Aging in wooden barrels followed by a reduction in bottles is a traditional practice in the production of quality wines, and one which is practiced in many wine-making regions of the world. In Spain, the term "crianza" is used to describe wines which have remained at least six months in contact with wood. During this period, the wood permits atmospheric oxygen to pass slowly through its pores, leading to the gentle oxidation of certain components of the wines, a reduction in astringency, and changes in color and taste (*1*, *2*). At the same time, certain components are extracted from the wood into the wines, giving the wines their characteristic aromas and tastes (*3*, *4*). However, for a wine to be suitable for aging in wood it needs to have sufficient structure and body, together with an antioxidant capacity to counterbalance the negative effect of oxygen. Phenolic compounds are mainly responsible for these characteristics (*5*) although they must evolve suitably during the aging process if a quality wine is to be produced (*6*). For this reason, prolonged maceration, or any other process which will increase the quality of phenolic compounds in the must, should render a wine more capable of withstanding the prolonged oxidative aging.

Oak is the most commonly used wood for barrels used for aging quality wines. American or French oak is preferred and depending on the drying or the toasting to which it has been submitted, the aromatic characteristics of the wines will change accordingly. However, several studies have suggested that, with all other things being equal, American oak contributes more cis and trans isomers of whiskylactone (*4*, *7*) than French oak. Both forms of whiskylactone have low thresholds and are usually identified as "woody" or "oak-like" aromas; they have also been used to distinguish between types of oak (*8*, *9*). These compounds, together with other lactones, volatile phenols, and related compounds such as γ-butyrolactone, 4-ethylphenol, 4-ethylguaiacol, guaiacol, eugenol, furfural, and vanillin, form the most important group of aromas in wines obtained by aging in wood. Although several authors have studied the influence of American or French oak on wine quality (*10*−*14*), these seems to be no generally accepted criteria for attributing better sensorial quality to wines aged in one versus the other.

The variety Monastrell represents 90% of the vines destined for D. O. Jumilla (SE Spain) wines. However, the resulting wines cannot withstand long aging periods because of their low

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Table 1. Type of Aged Wines and Nomenclature Used

barrel type	free-run wine (B)	B + enological tannin (T)	B + 15% press wine (P)
American oak (A)	AB	AT	AP
French oak (F)	FB	FT	FP

phenolic content (*15*); therefore, it is unusual for these wines to be aged in barrels. The study described here intends to establish the influence of using American or French oak on the sensorial quality and aromatic composition of three types of wine made from Monastrell, in which the phenolic content has been increased by the addition of wine obtained by pressing the grape pomace or by the addition of enological tannin.

MATERIALS AND METHODS

Vinification and Aging. Monastrell variety grapes picked during the 1999 harvest in a representative district of D. O. Jumilla (Murcia, SE Spain) were used. The wines were made with 10,000 kg of grapes following the traditional red-wine making processes of the region. Maceration was carried out in a 10-hL stainless steel container and lasted 13 days. During this time, the mixture was stirred 10 times to increase the extraction of phenolic compounds: seven "delestages" or emptying and strong refilling of the liquid phase, and three normal stirred. The alcoholic fermentation lasted 9 days and was carried out at 24 °C. The free-run wine was then separated from the solid phase, which was pressed to obtain 65% yield of wine or "press wine". After alcoholic followed by malolactic fermentation, the wines were kept at 10 °C until they were introduced into the barrels for aging. The free-run wine was used as starter, from which the other two wines were prepared. To the first was added 20 g/hL of enological tannin (Tanisouple, AEB, Bordeaux, France), and to the second was added 15% (volume) of pressed wine. In this way, three types of wine were obtained: free-run (B), B + enological tannin (T), and B + pressed wine (P), with the following characteristics (B, T, and P, respectively): alcohol (% v/v) 16.0, 15.9, and 15.8; pH 3.5, 3.5, and 3.4; volatile acidity (g/L acetic acid) 0.58, 0.58, and 0.62; titratable acidity (g/L tartaric acid) 6.21, 6.42, and 6.83; SO_2 total (mg/L) 106, 118, and 127.

The wines were introduced into 225-L new barrels of medium-toasted American (A) or French (F) oak (Magreñán S. L., La Rioja, Spain) where they remained for six months, the time necessary for them to be denominated "crianza". Each experiment was carried out in duplicate. The conditions of the cellar were kept at approximately 14 °C and 88% relative humidity. On a monthly basis the oxidation−reduction potential of the wines was measured by means of a platinum electrode (Crison Instruments S. A., Barcelona, Spain). **Table 1** shows the different types of wine obtained and their nomenclature.

After six months the wines were bottled and taken to the laboratory for chemical and sensorial analysis.

Sensorial Analysis. A sensorial analysis was carried out by a team of seven expert judges belonging to the Tasting Committee of this Regulatory Council. A penalization system of scoring was used in which the lowest points were given to the best-considered attributes: visual appreciation, olfactory appreciation (intensity and quality), taste appreciation (intensity and quality), and harmony (between all the above attributes). The score-scale of each parameter was: 0, excellent; 4−11, very good; 11−20, good; 20−29, normal; 29−36, quite bad; 36−41, defective.

Chemical Analysis. The phenolic content of the wines was evaluated by the total polyphenols index (*16*) and Folin-Ciocalteu's index (*17*), and tannins were measured according to the method described by Montedoro and Fantozzi (*18*).

To analyze the aromatic compounds contributed by the wood, a method based on that described by Waterhouse and Towey (*8*) and Pérez-Coello et al. (*9*), with some modifications, was used. Briefly, 0.5 mL of γ-hexalactone (IS) (Sigma-Aldrich Chemie, Steinheim, Germany) at 1 μL/mL and 15 g of ammonium sulfate were added to 50 mL of wine. Then 8 mL of dichloromethane was added to this solution, which was then centrifuged for three minutes at 2000 rpm to break down the emulsion. The organic phase was separated and filtered through anhydrous sodium sulfate, and the liquid filtrate was concentrated to 0.5 mL in a N_2 stream. A 10-μL aliquot of the concentrate was injected into a stainless steel thermal desorption tube measuring 88.9 mm × 6.35 mm o.d. × 4.50 mm i.d. which contained glass wool previously conditioned for one minute at 300 °C under a N_2 stream flowing at 100 mL/min. The components determined were furfural, guaiacol, *trans*-whiskylactone, *cis*-whiskylactone, 4-ethylguaiacol, eugenol, 4-ethylphenol, vanillin (Sigma-Aldrich), and γ-butyrolactone (Chem Service, West Chester).

Table 2. Phenolic Content of the Wines before Aging in Barrels ($n = 3$)

wine type	total polyphenols index	Folin–Ciocalteu index[c]	tannin[c]
free-run wine (B)	83.36a	3.00a	2.07a
B + enological tannin (T)	88.96b	3.16b	2.12b
B + 15% press wine (P)	82.90a	3.06a	2.09a

[a,b] Mean between treatments followed by the same letter are not significantly different ($\alpha = 0.05$). [c] g/L gallic acid.

The equipment used was a Perkin-Elmer thermal desorption injector (Norwalk, CT) coupled to a HP 6890 gas chromatograph with NIST library (Hewlett-Packard, Palo Alto, CA) and a fused silica capillary column with BP21 stationary phase 50 m length, 0.22 mm i.d., and 0.25 μm film thickness (SGE, Australia). The injection method allowed processing of many samples automatically. Chromatographically pure helium was used as carried gas. The conditions for thermic desorption were as follows: oven temperature, 290 °C; cold trap temperature, −30 °C; transfer line temperature, 190 °C; for gas chromatography 100 °C, then 2.5 °C/min to 180 °C (held 2 min), then 1 °C/min to 200 °C (held 22 min), and a carrier gas pressure of 22.6 psi; for mass spectrometry an energy of 70 eV was used in electron impact (EI) mode. The mass range varies between 40 and 350. The compounds were identified by comparing their mass spectra with those of the NIST library, those of the compounds we ourselves made from chromatographic standards, and data found in the bibliography. Working in the "sim mode" the following ions were used: γ-hexalactone, m/z 85; *cis*- and *trans*-whiskylactone, m/z 99; furfural, m/z 96; guaiacol, m/z 124; 4-ethylguaiacol, m/z 137; eugenol, m/z 164; 4-ethylphenol, m/z 107; vanillin, m/z 152; and γ-butyrolactone, m/z 86. Various standard solutions obtained by weighing the different compounds in a 12% (v/v) ethanol solution adjusted to pH 3.6 with tartaric acid were used for quantification. The corresponding calibration was made for each compound by successive dilutions of the original solutions. Linear regression coefficients between 0.97 and 0.99 were obtained. All the analyses were carried out in duplicate, except wines before aging, which were analyzed in triplicate.

The data were processed using the SPSS Version 9.0 statistical package for windows (*19*). A normalized variance analysis (ANOVA) was made using Duncan's test with a probability of 99.95%. The data were submitted to stepwise discriminant analysis.

RESULTS AND DISCUSSION

Table 2 shows the phenolic content of the three wines before being submitted to aging in barrels. The highest values for the three parameters evaluated were obtained in the wine treated with enological tannin. The phenolic levels recorded were within the range considered acceptable for the aging process to be used (*20*, *21*). The monthly measurements of the redox potential were similar in all three wines (89−100 mV).

Table 3 shows the respective concentrations of the aromatic compounds found in the wines before and after aging. The most abundant was γ-butyrolactone, the levels of which rose during aging with no significant differences being observed between the wines. The wines contained small quantities of guaiacol

Table 3. Mean Aromatic Compounds Content of the Wines (mg/L)[a]

	S_0[b]	A[c]B[e]	A[c]T[f]	A[c]P[g]	F[d]B[e]	F[d]T[f]	F[d]P[g]
furfural	0.011 ± 0.00a	1.206 ± 0.70 b	2.866 ± 0.77 c	1.127 ± 0.61 b	1.903 ± 0.27 bc	1.434 ± 0.96 b	1.998 ± 1.09 bc
γ-butyrolactone	1.225 ± 0.23a	16.969 ± 0.81b	17.463 ± 0.78 b	18.310 ± 0.36 b	16.865 ± 2.03 b	17.538 ± 0.63 b	18.523 ± 0.53 b
guaiacol	0.03 ± 0.01a	0.215 ± 0.09 b	0.268 ± 0.07 b	0.238 ± 0.05 b	0.232 ± 0.08 b	0.276 ± 0.07 b	0.307 ± 0.09 b
trans-whiskylactone	-	0.030 ± 0.00 ab	0.025 ± 0.00 a	0.030 ± 0.01 ab	0.031 ± 0.01 ab	0.037 ± 0.00 b	0.053 ± 0.00 c
cis-whiskylactone	-	0.260 ± 0.05 c	0.194 ± 0.01 b	0.247 ± 0.02 c	0.099 ± 0.05 a	0.072 ± 0.02 a	0.097 ± 0.01 a
4-ethylguaiacol	*	0.006 ± 0.00 cd	0.008 ± 0.00 d	0.007 ± 0.00 e	0.008 ± 0.00 ab	0.009 ± 0.00 a	0.011 ± 0.00 bc
eugenol	-	0.033 ± 0.00 a	0.035 ± 0.00 ab	0.042 ± 0.00 a	0.027 ± 0.00 ab	0.025 ± 0.00 ab	0.030 ± 0.00 b
4-ethylphenol	0.001 ± 0.00a	0.018 ± 0.01 b	0.031 ± 0.01 cd	0.025 ± 0.00 bc	0.042 ± 0.01 d	0.042 ± 0.01 d	0.042 ± 0.00 d
vanillin	0.006 ± 0.00a	0.018 ± 0.01 bc	0.013 ± 0.00 abc	0.010 ± 0.00 abc	0.019 ± 0.01 c	0.007 ± 0.00 a	0.009 ± 0.00 ab
cis/*trans*-whiskylactone		8.667	7.760	8.233	3.194	1.946	1.830

[a] Means between treatments followed by the same letter are not significantly different ($\alpha = 0.05$). * indicates the compound was detected but was not quantified. [b] Mean value of the wines B, T, and P before aging ($n = 3$), and after aging. [c] Barrel of American oak. [d] Barrel of French oak (means of two analyses for each one for both experiments $n = 2 \times 2$). [e] B: free-run wine. [f] T: B + enological tannin. [g] P: B + press wine.

Table 4. Aromatic Notes for the Aged Wines

	AB	AT	AP	FB	FT	FP
woody	X	X	X	X	X	X
vanilla	X	X	X			
spicy	X	X	X	X	X	X
smoky	X			X	X	
toasted	X	X	X	X	X	

Table 5. Mean Scores Obtained by Sensorial Analysis of Wines[a]

	A[c]B[e]	A[c]T[f]	A[c]P[g]	F[d]B[e]	F[d]T[f]	F[d]P[g]
visual appreciation	0.75 a	0.75 a	1.13 c	0.88 ab	0.88 ab	1.00 bc
taste appreciaton[b]	3.55 bc	4.05 d	3.00 ab	3.79 cd	2.71 a	2.50 a
olfactory appreciation[b]	2.00 a	3.19 b	2.06 a	3.57 bc	3.87 c	3.25 b
harmony	3.25 a	6.25 d	3.50 a	5.50 cd	4.88 bc	4.38 b
total	9.55 a	14.24 c	9.69 a	13.74 c	12.34 bc	11.13 ab

[a] Means between treatments followed by the same letter are not significantly different ($\alpha = 0.05$). [b] Mean of both intensity and quality. [c,d] Means of two analyses for each one for both experiments $n = 2 \times 2$. [e] B: free-run wine. [f] T: B+ enological tannin. [g] P: B+ "press wine".

before aging which increased significantly during the time in barrels. Although there were no significant differences in this parameter between the wines submitted to aging, the concentrations of guaiacol were slightly higher in the wines aged in French oak. Although the guaiacol content exceeded the olfactory threshold of 0.075 mg/L according to Chatonnet et al., 1992 (*22*), in all the aged wines, the characteristic smoked aroma was detected by the tasters in only three of the wines tasted (**Table 4**).

Furfural was present in low concentrations before aging, but was one of the most abundant components after being submitted to aging due to its extraction from the wood of the barrels. This particular compound is originated in the heating to which the wood is subjected during the manufacturing stage. Although the type of toasting is the same for all the barrels, furfural levels were particularly high in the wine with added tannins aged in American oak. However, in no wine did this compound exceed its olfactory perception threshold, which is 20 mg/L according to Chatonnet et al. (*22*).

The two whiskylactones were recorded in only the aged wines, which means they came from the wood. The levels of the cis isomer form in the wines aged in American oak were much higher than in those aged in French oak, although in neither case were they below the perception threshold (0.025 μg/L for *cis*-whiskylactone and 0.110 μg/L for *trans*-whiskylactone, according to Towey and Waterhouse) (*14*). A "woody" aroma was noted by all the tasters (**Table 4**). The ratio between cis and trans whiskylactone varied between 7.78 and 8.67 in the wines aged in American oak and between 1.83 and 3.19 in those aged in French oak. Although there are important variations in the whiskylactone ratio in those wines aged in French oak, it could be possible that they are due to the wood. Results are in accordance with those found by other authors (*8*, *9*) and demonstrate the usefulness of this parameter for distinguishing between the wines aged in each type of these two oak woods.

Eugenol was found only in aged wines, confirming its origin from the wood of which the barrels were made. Although the differences were not significant,. the mean values of eugenol were slightly higher in the wines aged in American oak. Eugenol endows wines with a characteristic spicy smell reminiscent of cloves; 4-ethylguaiacol does so, too, but only when its concentration is near the olfactory perception threshold of 0.047 mg/L according to Boidron et al., 1988 (*23*). Only eugenol exceeded its olfactory perception level, 0.015 mg/L according to Chatonnet et al. 1992 (*22*), and so the term "spicy" used by the tasters would have been due to this compound.

Although present in low concentrations before aging, the concentration of 4-ethylphenol increased significantly during this step. Its content was higher in wines matured in French oak and in the wine with added tannin matured in American oak. The perception level of 1.2 mg/L, according to Chatonnet et al. (*22*), was never reached, and its characteristic "leather", "phenolic", or "horse manure" smell was not noted by any of the tasters.

Most of the vanillin content of the aged wines came from the wines themselves before they were submitted to the aging process. Of all the components analyzed, this rose the least during aging. The greatest concentration was observed in the free-run wine regardless of the type of the wood. The word "vanilla" was used only to describe the wines aged in American oak (**Table 4**) despite the fact that the levels were far below the olfactory perception threshold of 0.32 mg/L according to Chatonnet et al. (*22*). Accepting that the vanillin was not degraded during this analysis because our work with standards confirms this, and acknowledging that the "vanilla" note was observed by the tasters, it could be thought that, judging from the results, this aroma (which is very appreciated in wines) may have some other source (*24*). Some authors have related the "vanilla" aroma with the *cis*-whiskylactone content of red wines (*25*, *26*).

Table 5 shows the values obtained in the sensorial analysis of the aged wines. The best-scored attribute in all the wines was the visual phase, and the worst attribute was harmony, although even this parameter was qualified as "very good". The attributes most closely related with aroma were those corre-

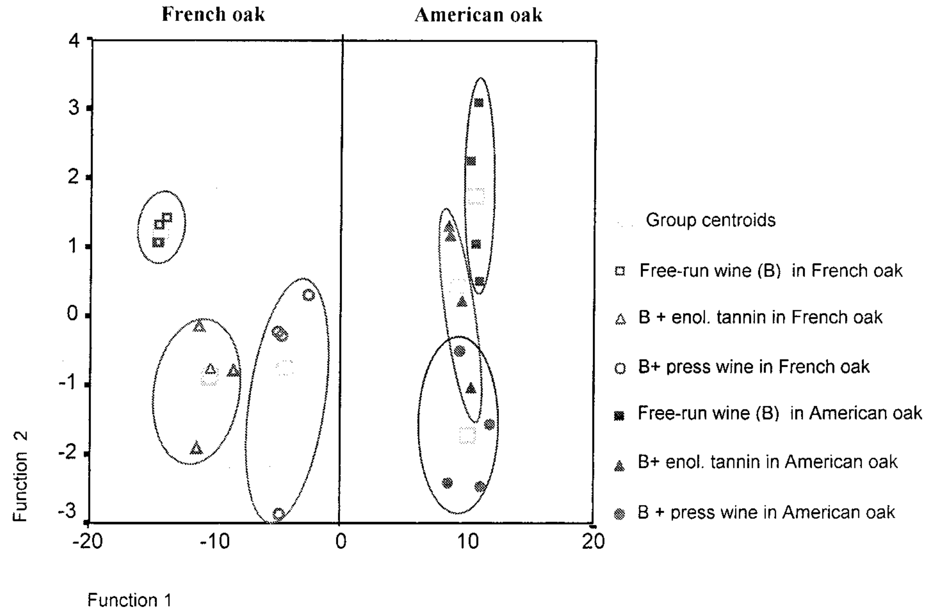

Figure 1. Discriminant analysis according to the type of wine in each barrel.

Table 6. Standardized Coefficients of the Canonic Discriminant Functions with Group Variable TWO[a]

	function			
compound	1	2	3	4
furfural	0.847	0.096	0.736	0.686
trans-whiskylactone	–3.101	0.222	–0.514	0.385
cis-whiskylactone	2.674	–0.610	–0.357	0.422
eugenol	0.716	1.090	0.160	–0.301
accumulated variance %	97.5	98.9	99.7	100

[a] Type of wine in each barrel.

Table 7. Standardized Coefficients of the Canonic Discriminant Functions with Group Variable TWT[a]

	function	
compound	1	2
γ-butyrolactone	–0.660	0.753
vanillin	0.721	0.695
accumulated variance %	88.9	100

[a] Type of wine.

sponding to the olfactory phase and harmony (balance between the rest of attributes). The best-valued wines from both points of view were those containing natural tannin (free-run wine and the wine resulting for the addition of press wine) aged in American oak. However, this type of wood was also responsible for the least appreciated wine: that to which enological tannin had been added. Consequently, American oak aging differentiates the wine made with added enological tannin from wine made with natural tannin, whereas French oak aging equals out the sensorial characteristics of these wines.

A statistical treatment was applied to the aromatic compounds determined, and the results of the sensorial analysis to determine their abilities to differentiate between aged wines. Two group variables were defined: “type of wine in each type of oak” (TWO) and “type of wine according to tannin content” (TWT). The independent variables used were those corresponding to the aromas analyzed and the score in the sensorial analysis. Furfural, *cis*- and *trans*-whiskylactones, and eugenol distinguished between the wines in each type of barrel. **Table 6** shows the coefficients of the four discriminant functions and the contribution of each function for the differentiation. **Figure 1** shows the diagram obtained by representing the first two functions, where it can be seen that function 1 clearly differentiates between the aged wines in each type of wood and between the two types of natural wine in French oak, although the wines aged in American oak are not so clearly differentiated.

When the type of wine was used as group variable without reference to the type of wood, differentiation was possible with only two functions, where the selected variables were γ-butyrolactone and vanillin (**Table 7**). The graphical representation of both functions did not clearly distinguish between the three wines, suggesting that the differentiating effect of the wood, regardless of its type, is greater than the effect of the tannin content.

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