

# Fermentation of White Wines in the Presence of Wood Chips of American and French Oak

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Must obtained from Airén grapes was fermented in the presence of wood chips (4 and 7 g/L) of either French oak (from Vosges, central France, and Allier zones) or American oak. Fermentation yields were higher than in the control fermentations carried out in the absence of wood, and production of volatile substances during fermentation (alcohols, esters, and acetates) was also higher. The volatile substances that leached out of the wood were analyzed by GC-MS-SIR. The results showed that their concentrations depended on the type and amount of the oak; some of these substances were consumed in part by the yeasts during fermentation. A taste panel favorably assessed the wines produced by fermentation in the presence of oak chips, which retained part of the must original fruity aroma.

**Keywords:** Oak compounds; fermentation; white wine

## INTRODUCTION

The aging of wines in wooden barrels made from American oak (*Quercus alba*) and French oak (*Q. petraea* and *Q. robur*) has mainly been applied to red wines. Barrel aging of such wines brings about changes in color and aroma that are highly appreciated by consumers; the changes are produced by several compounds extracted from the wood (Nykänen, 1984).

In certain countries, white wines have occasionally been fermented in oak barrels and left in contact with the lees after fermentation (Marsal et al., 1988; Chatonnet et al., 1992). These practices are now being generally used in order to impart new nuances to wines made from certain grape varieties that do not present fruity aromas.

Wines aging or fermenting in oak barrels contain substances extracted from the wood, such as furan derivatives (furfural, 5-hydroxymethylfurfural) and phenolic and cinnamic aldehydes, with vanillin exerting the greatest effect on wine aroma (Singleton, 1995). The two oak lactone isomers ( $\beta$ -methyl- $\gamma$ -octalactone), whose odor has been described as oak or coconut, have very low perception thresholds (Abbott et al., 1995; Maga, 1996). In barrel fermentation, the yeast cells adsorb the ellagitannins from the wood, thereby reducing adverse astringent effects (Boidron et al., 1988). In white wines fermented in oak barrels, yeasts are also able to convert some of the substances extracted from the wood during alcoholic fermentation. Furfural is reduced to furfuryl

alcohol by the yeasts, which are also able to reduce vanillin to vanillyl alcohol, a substance of less sensory importance given its high perception threshold (50 mg/L; Chatonnet et al., 1992).

The quantities of the substances passing into the wine depend on the type of wood employed (Marco et al., 1994; Towey and Waterhouse, 1996b; Chatonnet and Dubourdieu, 1998), the seasoning and charring treatments that have been applied to the wood (Sefton et al., 1993; Chatonnet et al., 1994; Cutzach et al., 1997), and other factors, such as how long the wood is in contact with the wine, the conditions of temperature and humidity, and so forth. (Puech, 1987; Towey and Waterhouse, 1996a; Mangas et al., 1997; Spillman et al., 1998a).

Fermentation in oak barrels does present certain technical challenges, such as the inability to carry out large-capacity fermentations, the difficult temperature control, the required cleaning of the barrels on completion of fermentation, and so forth. The use of oak chips allows the fermentation in stainless steel tanks to be carried out under the required temperature conditions and the amount of substances leached out of the wood to be controlled.

In a comparative study carried out by Wilker and Gallander (1988), no significant differences were found using sensory analysis between wines aged in the presence of oak chips and wines aged in oak barrels, but no chemical composition data were given.

An alternative to the aging of distillates in oak barrels is the use of oak chips or oak chip extracts (Mosedale and Puech, 1998; Monedero et al., 1998). Steeping oak chips for a time ranging from 1 h to 1 day, depending on the extraction conditions, has yielded results similar

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to those obtained by barrel aging for several years (Giménez-Martínez et al., 1997).

The object of this study was to examine the effect of oak chips on the fermentation of white wines and to determine the influence of oak type and the quantity of oak chips employed on the chemical and sensory characteristics of the wines produced.

## MATERIALS AND METHODS

The wood chips used were obtained from French oak (*Quercus petraea*) from Allier, Vosges, and central France and from American oak (*Quercus alba*) (Tonelería Victoria S. A., Haro, Spain). Oak chips were added to Erlenmeyer flasks containing 2 L of Airén grape must. Erlenmeyer flasks were sealed with Müller valves, which provided a hermetic seal that prevented oxygen from entering while allowing CO<sub>2</sub> to escape.

In a previous step, samples of the white wine were left to steep for 15 days with 14 g/L of oak chips of the four types considered in this study. When sensory analysis was applied, assessors rejected these wines because of their heavy oak flavor predominating over all the other attributes. For this reason, lower concentrations of wood were selected when fermentations were carried out in the presence of wood chips, but macerated wines were useful to evaluate the effect of the fermentation in the volatile wood compounds.

Two different concentrations (4 and 7 g/L) of each type of oak chip were tested in duplicate. All the flasks were inoculated with a laboratory culture of a strain of *S. cerevisiae uvarum* selected at our laboratory among musts from the La Mancha region in Spain. The control fermentations were carried out in the same conditions, but no oak chips were added to the flask.

**Gas Chromatographic Analysis of Volatile Components.** Two microliters of wine containing 2-pentanol as an internal standard was injected in a Perkin-Elmer 8700 gas chromatograph in order to analyze the major volatile components. A methyl siloxane column (HP-101, 50 m × 0.32 mm i.d. × 0.3 μm film thickness) was programmed from 50 °C (7 min) to 130 °C (15 min) at a 6 °C/min rate.

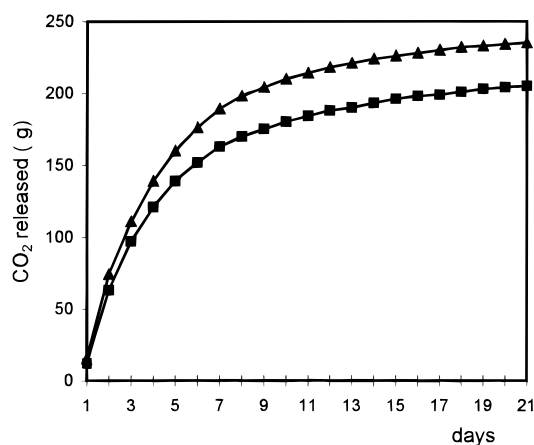
Minor wine components, including those proceeding from wood, were extracted in a continuous liquid-liquid extractor using organic solvents (pentane and dichloromethane). For an internal standard 0.5 mL of 4-nonanol (2 mg/L) was added to 200 mL of wine.

The extracted volatile components were analyzed by GC using a Hewlett-Packard model 5890 chromatograph. 2 μL of extract was injected in splitless mode (1 min). A BP-21 capillary column (50 m × 0.32 mm i.d. × 0.25 μm film thickness) (SGE, Australia) was programmed from 70 °C (7 min) to 90 °C (10 min) at a 1 °C/min rate and then to 190 °C (40 min) at 2 °C/min rate.

To analyze the compounds from the wood present in the extract, a Fisons model GC 8000 gas chromatograph connected to a model MD 800 mass detector in SIR (selected ions register) mode was used. A Supelco SPB-1 methyl silicone column (50 m × 0.25 mm i.d. × 0.25 μm film thickness) was programmed from 80 °C (3 min) to 250 °C (40 min) at a 3 °C/min rate. Transfer line temperature was 225 °C. Ionization was by electron impact (EI mode) at an energy of 70 eV. The following ion masses were selected, taking into account relative ion abundance and low spectral noise: internal standard (2-nonanol), 73; furfural, 96; 5-hydroxymethylfurfural, 126; guaiacol, 124; furfuryl alcohol, 98; eugenol, 164; *cis*- and *trans*-β-methyl-γ-octalactone, 99; vanillin, 152; and syringaldehyde, 182.

For quantitative analysis, response factors were calculated for each compound using standards (Sigma-Aldrich Química S. A., Madrid, Spain).

**Sensory Analysis.** Wine sensory analysis was carried out by a panel of 10 expert assessors ranging in age between 25 and 45, in a standard tasting chamber (ISO 8589, 1988). Samples were presented in standard glasses (ISO 3591, 1977) in random order at a temperature of 10 °C.



**Figure 1.** Mean values of CO<sub>2</sub> loss vs fermentation time for musts fermented with (▲) and without (■) oak chips (control wines).

An unstructured 10-cm scale, in which 0 was "attribute not perceptible", and 10 was "attribute highly perceptible" was used. The attributes selected were fresh aroma, unripe fruit aroma, unripe fruit flavor, oak aroma, oak flavor, acidity, aftertaste, and overall impression.

## RESULTS AND DISCUSSION

**Effect of Oak Chips on Fermentation.** Fermentation was monitored by the CO<sub>2</sub> produced, calculated as weight loss in the flasks. The mean value of the total amount of CO<sub>2</sub> released by the wines fermented in the presence of the oak chips was 223.3 g/L (standard deviation: 5.2), and the amount of CO<sub>2</sub> produced by the control wines was 203.7 g/L (standard deviation: 3.9). These values are statistically different according to the Student's *t* test.

Curves of CO<sub>2</sub> loss versus time are shown in Figure 1.

The volatile components were analyzed when fermentation was complete. Table 1 shows the mean concentrations for the volatile components in the wines fermented in the presence of the oak chips and in the control wines. The Student *t*-test was performed in order to evaluate the significance of the differences between the mean values for the two types of wine.

The volatile components that presented significant differences between the experimental and control wine batches were alcohols (isobutanol, 2-methyl-1-butanol, 3-methyl-1-butanol, and 2-phenylethanol), acetates (isoamyl acetate and 2-phenylethyl acetate), and certain esters (ethyl caprylate, ethyl caproate, and ethyl lactate). All those substances were present in higher concentrations in the wines fermented in the presence of the oak chips. Since all these substances are products of yeast metabolism, the results are a consequence of the higher fermentation yields obtained when the must was fermented in the presence of the oak chips.

This effect can be attributed to the action of the oak chips as a carrier for the yeast cells, exerting an effect similar to that of immobilized cells. Other workers have also observed that the presence of suspended solids in the liquid during fermentation, such as bentonite, glass balls, gluten pellets, and delignified cellulosic material, increased fermentation yields (Guenette and Duvnjak, 1996; Iconomou et al., 1996; Bardi et al., 1997). Lenzi (1986) noted that the use of immobilized yeasts during fermentation increased the production of such fermentation components as propanol and isoamyl alcohols.

**Table 1. Volatile Compounds in Airén Grape Wines Fermented with and without Oak Chips (Control Wines) (mg/L)**

compound	control wines (n = 8)		Airén grape wine fermented with oak chips (n = 16)	
	X	SD	X	SD
1-hexanol	0.23	0.006	0.16	0.02
cis-3-hexen-1-ol	0.52	0.02	0.47	0.11
trans-3-hexen-1-ol	0.10	0.009	0.09	0.01
benzyl alcohol	0.09	0.02	0.08	0.02
benzaldehyde	0.02	0.005	0.04	0.007
methanol	51.14	1.51	47.48	3.98
2-phenylethanol <sup>a</sup>	9.96	0.49	14.94	1.53
1-propanol	23.56	3.93	20.92	2.05
ssobutanol <sup>a</sup>	20.93	1.47	28.28	1.97
2-methyl-1-butanol <sup>a</sup>	15.38	1.51	18.19	1.38
3-methyl-1-butanol <sup>a</sup>	85.51	3.92	123.48	8.34
acetaldehyde	35.15	10.91	29.72	5.56
ethyl acetate	22.57	18.34	22.75	13.8
isoamyl acetate <sup>a</sup>	1.43	0.12	3.66	0.35
2-phenylethyl acetate <sup>a</sup>	0.12	0.03	0.31	0.01
hexyl acetate	0.04	0.05	0.04	0.008
ethyl butyrate	0.38	0.08	0.37	0.08
4-OH-ethyl butyrate	1.97	0.36	2.08	0.48
ethyl caprylate <sup>a</sup>	0.82	0.05	0.98	0.08
ethyl caproate <sup>a</sup>	0.39	0.06	0.66	0.07
ethyl lactate <sup>a</sup>	0.96	0.01	1.34	0.26
butyric acid	0.45	0.07	0.44	0.03
isobutyric acid	0.42	0.007	0.53	0.10
isovaleric acid	0.46	0.03	0.54	0.08
caproic acid	3.07	0.18	3.17	0.47
caprylic acid	2.22	0.38	2.03	0.44
capric acid	5.62	0.78	5.26	0.96

<sup>a</sup> There were significant differences in component concentrations between the two wines according to Student's *t*-test.

From a technical standpoint, the higher fermentation yields obtained in the case of the wines fermented in the presence of the oak chips are advantageous since several components produced in higher levels by yeast metabolism, such as 2-phenylethanol, esters, and acetates, have an interesting impact on wine aroma.

**Wood Components Leaching into the Wine.** Table 2 lists the concentrations of the wood components present in the wines macerated with 14 g/L of oak chips and in the wines fermented in the presence of the chips (4 g/L and 7 g/L) analyzed by GC-MS-SIR. The values vary according to the type of oak and increase with the amount of the oak chips employed.

Quantities of guaiacol were similar for all the different types of oak tested, whereas eugenol concentrations were somewhat higher in the wines that were in contact with wood chips of oak from central France (Pérez-Coello et al., 1999).

Levels of the *cis* isomer of oak lactone were higher than those of the *trans* isomer in all the samples; in addition, the *cis* isomer has the lower perception threshold of the two forms (Abbot et al., 1995). Other workers have used the ratio between the two isomeric forms as an indicator for differentiating between oak wood types, because the ratio remains constant irrespective of the treatments applied to the wood (seasoning, charring, etc.) (Waterhouse and Towey, 1994; Masson et al., 1995a,b). Table 2 shows that the ratio value was highest in the samples in contact with the wood chips made from American oak. The ratio values for the French oaks were lower, and in the case of the oak wood from central France, were around 1. These findings are consistent with the results of the wood chip analyses using direct thermal desorption and injection of water/ethanol extracts (Pérez-Coello et al., 1997).

Furfural concentrations were higher in the wine samples fermented with American oak but lower than other authors found in charred or toasted oak wood, since the toasting procedure increases the formation of furfural and 5-hydroxymethylfurfural from cellulose (Marsal and Sarre, 1987; Towey and Waterhouse, 1996a).

Furfural is reduced to furfuryl alcohol by the action of yeasts (Marsal et al., 1988; Chatonnet et al., 1992). This is an interesting issue, since high levels of furfural are not desirable in a white wine. This effect is observable in the samples analyzed in this study (Table 2). The wines fermented in the presence of 4 g/L and 7 g/L of oak chips had lower concentrations of furfural and higher concentrations of furfuryl alcohol than the wines macerated with oak chips only after fermentation. The increase in furfuryl alcohol was less than was to be expected, because it could probably be converted to the corresponding ether and other compounds (Spillman et al., 1998 b).

Vanillin levels were higher in wines macerated with oak chips than in the samples fermented in the presence of chips, since vanillin can be metabolized by yeasts producing vanillyl alcohol (Chatonnet et al., 1992; Spillman et al., 1997, 1998b). However, vanillin contents in all wines fermented with 7 g/L of chips are above their organoleptic perception threshold (0.5 mg/L; Maga, 1984).

**Sensory Profiles of the Wines Fermented in the Presence of Oak Chips.** Table 3 presents the mean scores awarded by the assessors for the different attributes.

Scores for the attributes fresh aroma, unripe fruit aroma, and unripe fruit flavor were lower in the samples

**Table 2. Volatile Wood Extractives in White Airén Wines Macerated and Fermented in Contact with American or French Oak Chips (μg/L)**

compound	Airén wines macerated with 14 g/L of oak chips				Airén wines fermented with 7 g/L of oak chips				Airén wines fermented with 4 g/L of oak chips			
	French oak				French oak				French oak			
	American oak	Vosges	central France	Allier	American oak	Vosges	central France	Allier	American oak	Vosges	central France	Allier
guaiacol	3.51	3.93	2.71	3.18	1.64	1.31	0.86	1.50	0.89	0.76	0.84	0.82
eugenol	10.79	9.81	25.10	9.68	10.82	4.95	12.19	5.94	3.74	3.12	10.27	4.21
cis-oak lactone	338.11	341.32	228.24	197.40	154.34	130.27	78.93	88.48	68.85	59.35	67.28	47.76
trans-oak lactone	26.37	85.18	165.65	28.87	15.95	37.52	71.58	17.14	8.38	18.24	63.71	9.80
cis/trans lactone	12.80	4.00	1.30	6.80	9.60	3.40	1.10	5.16	8.20	3.20	1.05	4.80
furfural	129.09	68.11	61.76	66.39	18.84	14.67	9.13	11.54	15.69	8.18	9.47	5.93
5-OH-methylfurfural	0.48	0.53	0.68	0.39	2.40	2.29	2.29	2.57	2.69	2.41	3.42	2.33
furfuryl alcohol	0.79	1.42	0.92	0.95	8.28	5.60	5.63	4.91	6.33	3.54	5.11	2.21
vanillin	37.67	22.22	48.07	7.37	5.22	0.64	0.49	0.73	0.49	0.25	0.33	1.24



**Table 3. Mean Scores for Each of the Selected Attributes Earned by the Wines Fermented with 4 g/L and 7 g/L of American or French Oak Chips**

attribute	control wine	wines fermented with American oak chips		wines fermented with French oak chips					
		4 g/L	7 g/L	Vosges		central France		Allier	
				4 g/L	7 g/L	4 g/L	7 g/L	4 g/L	7 g/L
fresh aroma <sup>a</sup>	5.0	1.0	0.4	1.7	1.2	2.5	1.4	3.2	1.8
unripe fruit aroma <sup>a</sup>	2.8	0.7	0.2	0.3	0.2	1.2	0.3	2.5	0.7
unripe fruit flavor <sup>a</sup>	4.1	1.7	0.8	2.6	1.2	4.9	4.3	5.0	4.2
oak aroma <sup>a</sup>	0	2.4	4.4	1.3	3.9	0.8	2.3	1.3	1.7
oak flavor <sup>a</sup>	0	1.3	3.7	1.3	3.6	0.8	2.2	1.2	2.4
acidity <sup>a</sup>	5.4	3.8	4.0	3.7	4.2	5.0	4.3	4.0	4.5
aftertaste <sup>a</sup>	4.8	4.7	5.4	6.2	5.4	5.5	5.1	7.4	6.5
overall impression <sup>a</sup>	5.1	6.8	7.0	6.2	6.7	7.8	6.3	7.4	8.0

<sup>a</sup> Attributes that presented significant differences between the control wines and the wines fermented with oak chips according to the ANOVA Test

fermented in the presence of the oak chips than in the control wines; these attributes are characteristic of young white wines. This effect was more pronounced when wines were fermented with the higher concentration of oak chips (7 g/L) and when American oak and Vosges French oak were used. All the attributes present significant differences between the control wines and the wines fermented with oak chips according to the ANOVA test.

Other researchers have reported losses in fresh or unripe aromas in barrel-aged wines (Aiken and Nobel, 1984a,b; Wilker and Gallander, 1988). In the present experiment, it is likely that such attributes were masked by the oak aromas, since chemical analysis showed that the wines fermented with oak chips had higher concentrations of esters and acetates than the control wines.

On the other hand, the attributes oak aroma and oak flavor were perceived in the wines fermented in the presence of the oak chips at both concentrations tested. These wood-induced attributes were more pronounced in the batches fermented with wood chips of American oak and Vosges oak, which is consistent with the higher content of the cis-oak lactone, the substance chiefly responsible for oak aroma, revealed by the results of the chemical analysis of the samples.

Francis et al. (1992) found that model solutions of oak from the Vosges region were more aromatic, including particularly the attributes coconut and vanilla aroma.

The scores for overall impression showed that the wines fermented in the presence of the oak chips (4 g/L or 7 g/L) were preferred to the control wines; it seems that small amounts of wood components allow the wines to retain part of their fruity attributes and contribute to an improvement on overall flavor. The best scores were assigned to wines fermented using oak from the Allier region of France and from central France.

## CONCLUSIONS

White wines fermented in the presence of oak chips exhibit both higher fermentation yields and a higher production of volatile components during fermentation.

In addition, such wines also contain wood components, such as the oak lactones, eugenol, and vanillin, increasing the complexity of their aroma. American oak yielded the largest amounts of cis-oak lactone. Furfural and vanillin were metabolized by the yeasts in the wines fermented in the presence of the oak chips.

On the other hand, the use of oak chips allows the fermentation to be carried out in stainless steel tanks,

avoiding the loss of fruity aromas and color changes that could negatively affect the quality of wines.

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