ISSN 1330-9862
(FTB-2736)

original scientific paper

Dependence of Oak-Related Volatile Compounds on the Physicochemical Characteristics of Barrel-Aged Wines

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Received: February 14, 2011
Accepted: June 20, 2011

Summary

This paper focuses on the effect of some of the physicochemical characteristics of wines such as volatile acidity, titratable acidity, pH, free SO_2 and alcohol content on the accumulation of oak-related volatile compounds in barrel-aged wines, in order to give more light on the contradictory results found by other authors in this respect. For this, three different single variety wines were aged for twelve months in barrels with the same characteristics (same cooperage, wood origin, toasting level and volume), repeating the experiment in two consecutive years. Our results show that the percentage of wine alcohol and its titratable acidity positively correlated with the final concentration of vanillin and guaiacyl compounds in the oak-matured wines and negatively with the *cis*- and *trans*-β-methyl-γ-octalactone concentration. Therefore, when studying the effect of oak barrel variables (oak origin and seasoning, size of the barrel, number of uses, *etc.*) on the concentration of oak-related volatile compounds in wine, the effect of the physicochemical variables of the wine, especially titratable acidity and alcohol content, should also be taken into account since the final wine aroma composition will also depend on these characteristics.

Key words: oak-related volatile compounds, wine, ethanol, acidity

Introduction

The maturation of wine in oak barrels, a common practice in red wine making, is a complex process in which several phenomena combine to produce organoleptic changes in wines that generally improve its flavour and taste (*1*). The wine acquires a greater aromatic complexity as a result of the extraction of volatile compounds from oak barrels.

The main volatile compounds extracted from oak wood (*cis*- and *trans*-β-methyl-γ-octalactones, the volatile phenolic aldehydes and alcohols vanillin, guaiacol, 4-methylguaiacol and eugenol, and furfural, 5-methylfurfural and 5-hidroxymethylfurfural) impart a pleasant aroma to oak-aged wines. The accumulation of these compounds in wine mainly depends on the quantity of compounds that are potentially extractable and on the contact time between wine and wood (*2–6*).

Accordingly, it might be expected that the levels of oak-related volatile compounds in wines stored in identical barrels will be very similar, even taking into account barrel-to-barrel variations but this fact is not always true. It has been stated that the characteristics of the wine itself may also be very important during the ageing process, since pH, ethanol and acidity may have a direct influence on ethanolysis of wood components, whereas SO_2 may have a solvent effect on some of them (*3,7*). This topic has been less investigated and, moreover, contradictory results have been found. Garde-Cerdán *et al.* (*4,8*) stated that the extraction of furanic aldehydes, oak lactones, phenolic aldehydes and alcohols was influenced by the wine alcohol content, the wine pH being less significant, whereas Ortega-Heras *et al.* (*3*) did not find significant correlations between wine ethanol content and the levels of any of the volatile compounds studied. In a recent study, Garde-Cerdán *et al.* (*9*) stated that

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*Corresponding author; Phone: ++34 868 887 323; Fax: ++34 868 884 147; E-mail: encarnag@um.es they did not find any significant correlation between the physicochemical properties of 510 oak-aged red wines and the concentration of their oak-related volatile compounds. Most of these studies were conducted during just one year and, in some cases, using the physicochemical properties of the aged wines and not the initial wine properties (before the oak-ageing period). This latter fact could partly explain some of the contradictory results that can be found in the literature since wine physicochemical properties change during the ageing period (both oak and bottle ageing) (*10,11*) and the physicochemical composition after the ageing period could be rather different than the initial properties, the ones that will play an important role in the accumulation of the oak-related compounds in wines.

Our previous results of soaking oak chips into model solutions with different physicochemical characteristics showed that the extraction of some compounds was clearly affected by the characteristics of the model solution (*12*). In this current study, we have tried to shed more light on this research, determining the correlations, if any, between the extraction and accumulation of oak-related volatile compounds in wines and their physicochemical characteristics at the moment of filling the oak barrels, using wines from different varieties but elaborated in the same winery, aged in oak barrels with the same characteristics and repeating the study in two consecutive years.

Material and Methods

Wines

Monastrell, Petit Verdot and Cabernet Sauvignon red wines from D.O. Jumilla (Spain) were produced on an industrial scale in Finca Omblancas winery in 2006 and 2007 under identical winemaking conditions. These wines were aged each year in new 300-litre medium toasted French oak (*Quercus petraea*) barrels supplied by Seguin Moreau cooperage (Cognac, France). For the toasting of the barrels, and according to the cooperage firm, the process was divided into two steps. In the first step, whose objective was bending of the wood, the wood was exposed to a temperature of 100–120 °C for 10–15 min. In the second step, the wood was exposed to a temperature of 150–190 °C for 10–15 min, increasing to 200–220 °C at the end of toasting.

Three barrels were filled with each single-variety wine and samples from each barrel were taken after 3, 6 and 12 months. Classic oenological parameters of these wines were determined before they were transferred to the barrels and during the ageing process, according to the OIV methods (*13*).

The cellar conditions were established at 17 °C and 80 % of relative humidity. The barrels were refilled every ten days to prevent losses due to the evaporation of water and ethanol. No rackings were performed during ageing and corrections of SO_2 were done when the free SO_2 levels were below 10 mg/L.

GC-MS volatile analyses

Volatile compounds were extracted from the wines and analysed following a modification of the method described by Pérez-Prieto *et al.* (*14*), where commercial internal standards were used instead of deuterated standards. The internal standards used for quantification were 4,5-dimethylfurfural (Sigma-Aldrich, Steinheim, Germany) for furfuryl compound quantification, γ-hexalactone (Sigma-Aldrich, St. Louis, MO, USA) for lactone quantification, and 3,4-dimethylphenol (Sigma-Aldrich, St. Louis, MO, USA) for guaiacol, 4-methylguaiacol and vanillin quantification. Analyses were performed using an Agilent 6890N gas chromatograph (Santa Clara, CA, USA) equipped with an Agilent 5973 mass spectrometric detector. Samples were injected in splitless mode (purge time 2 min), and volatile compounds were separated using an HP-INNOWax polyethylene glycol column (30 m×0.25 mm i.d., and 0.50 mm nominal film thickness), supplied by Agilent (Madrid, Spain). The oven temperature was programmed from 70 to 240 °C (10 °C/min). Helium was used as the carrier gas at an average linear velocity of 1.1 mL/min. The injector was maintained at 250 °C and the line of transfer was set at 280 °C. For the quantification of the compounds, mass spectra were recorded in the selective ion monitoring (SIM) mode due to its higher sensitivity. From the mass spectrum of the compounds of interest, we selected the ions that would be monitored during the analysis. These ions monitored in the SIM runs were (boldface ions are those used for quantification): (1) *m/z*=67, 95 and **96** for furfural; (2) *m/z*=81, 109 and **110** for 5-methylfurfural; (3) *m/z*=81, 109, **124** and 127 for guaiacol; (4) *m/z*=95, 123, **138**, 139 and 141 for 4-methylguaiacol; (5) *m/z*=**99**, 101, 114, 118, 132, 138, 156 and 160 for *trans*-oak lactones; (6) *m/z*=**99**, 101, 114, 118, 132, 156 and 160 for *cis*-oak lactones; *m/z*=151, **152**, 154, 155 and 156 for vanillin; (7) *m/z*=81, 109, 123, **124** and 127 for 4,5-dimethylfurfural (internal standard for furfural quantifications); (8) *m/z*=70, **85**, 86 and 114 for γ-hexalactone (used for lactone quantifications) and (9) *m/z*=107, 111, **122** and 126 for 3,4-dimethylphenol (for guaiacol, 4-methylguaiacol and vanillin quantification). The concentration of each substance was measured by comparison with the calibrations made with the pure reference compounds analysed under the same conditions. The corresponding calibration was made for each compound, and linear regression coefficients between 0.996 and 0.999 were obtained.

Statistical analyses

Significant differences among the samples were assessed by an analysis of variance. Correlation analysis was also performed to study the effects of the physicochemical properties of the wines and the accumulation of volatile compounds after 12 months of oak ageing. These statistical analyses were performed using Statgraphics Plus v. 5.1 (StatPoint Technologies, Inc., Warrenton, VA, USA).

Results and Discussion

The physicochemical properties of wine before and after the ageing process are shown in Table 1. In both years the Monastrell red wines before ageing showed higher acidity and higher alcoholic content and lower values of pH and free SO_2 than the Cabernet Sauvignon and Petit Verdot wines. The lowest titratable acidity and

Table 1. Physicochemical characteristics of Monastrell, Cabernet Sauvignon and Petit Verdot wines before and after ageing in barrels during 12 months

Ageing time		2006			2007		
		Monastrell	CS	PV	Monastrell	CS	PV
t_0	γ(VA)/(g/L)	0.50b	0.42a	0.49b	0.53b	0.55c	0.27a
	pH	3.78a	3.99c	3.84b	3.51a	3.71c	3.66b
	γ(free SO_2)/(mg/L)	31.67a	38.67b	37.67b	15.67a	42.00b	63.33c
	γ(TA)/(g/L)	5.48c	4.43a	4.80b	6.60c	5.37a	6.00b
	φ(alcohol)/%	15.33c	13.67a	13.93b	16.00c	15.50b	13.10a
t_{12}	γ(VA)/(g/L)	0.66b	0.66b	0.51a	0.72b	0.71b	0.40a
	pH	3.70b	3.75c	3.66a	3.47a	3.58b	3.70c
	γ(free SO_2)/(mg/L)	25.00a	28.00b	28.00b	12.80a	19.20b	26.33c
	γ(TA)/(g/L)	5.65c	4.65a	5.40b	6.15c	5.43b	4.65a
	φ(alcohol)/%	15.40c	13.70a	13.90b	16.00c	15.50b	13.10a

CS: Cabernet Sauvignon wines, PV: Petit Verdot wines, VA: volatile acidity (expressed as g of acetic acid per L), TA: titratable acidity (g of tartaric acid per L), free SO_2 (expressed as mg per L). Different letters within the same row and for each year indicate significant differences ($p<0.05$) according to the LSD test

highest pH were found in Cabernet Sauvignon wines in both years. After the ageing period, the Monastrell red wines still showed higher acidity and alcoholic content than the other wines, and higher values of volatile acidity than Petit Verdot wines.

The accumulation of oak-related volatile compounds during the oak maturation period is shown in Figs. 1 to 6, and to check whether the physicochemical characteristics of the wines influenced the concentrations of these compounds, a matrix was built with the linear correlations between the final levels of the oak-related volatile compounds and the initial physicochemical characteristics of the wines.

Furfuryl derivates

The evolution of the concentrations of furfural and 5-methylfurfural during ageing (Figs. 1 and 2) was very similar although furfural reached a higher final concentration. Both compounds were extracted rapidly, reaching a maximum level after which a decrease was observed. In 2006, the maximum was observed at 3 months of ageing for Cabernet Sauvignon wine and at 6 months for Monastrell and Petit Verdot wines, while the maximum was reached after 3 months in all the wines in 2007. These results are in agreement with those of Ferreira *et al.* (*15*), who stated that the evolution of furfural represents the profile of compounds suffering strong degradations during the ageing process. The maximum values reached for the different wines were quite similar in both years, and the final concentrations were also quite similar, especially in 2007. The decrease in furfuryl compounds has been associated with the biochemical reduction of both compounds in wines to the corresponding alcohols, furfuryl alcohol and 5-methylfurfuryl alcohol (*16–19*). This reduction could be due to the action of furfural reductases (*20,21*) or to NADH and NADPH-alcohol dehydrogenases (*22–25*), the presence of which may be due to yeast lysis or to alcoholic fermentation, and they would remain in wines which have not been filtered (*26*). The relationship between furfuryl derivate degradation and the existence of these enzymatic activities in wines was also observed by Rodríguez-Rodríguez and Gómez-Plaza (*12*), who found that when soaking oak chips in model solutions, the decrease of furfuryl compounds was not observed, whereas it was clearly observed in wine samples. The correlation matrix (Table 2) showed that no significant correlations were found between furfural or 5-methylfur-

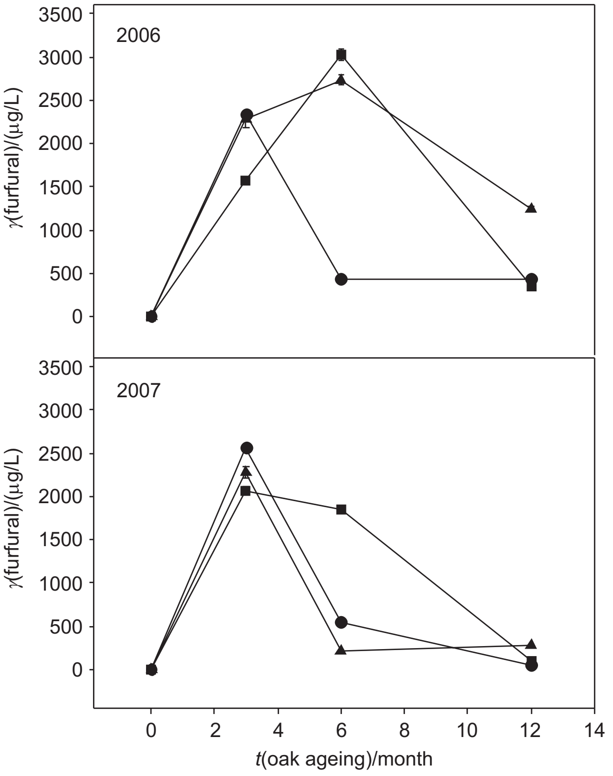

Fig. 1. Evolution of the concentration of furfural during barrel ageing in 2006 and 2007 for Monastrell (▲), Cabernet Sauvignon (●) and Petit Verdot (■) wines. Error bars represent the standard deviation of the mean value (*N*=3)

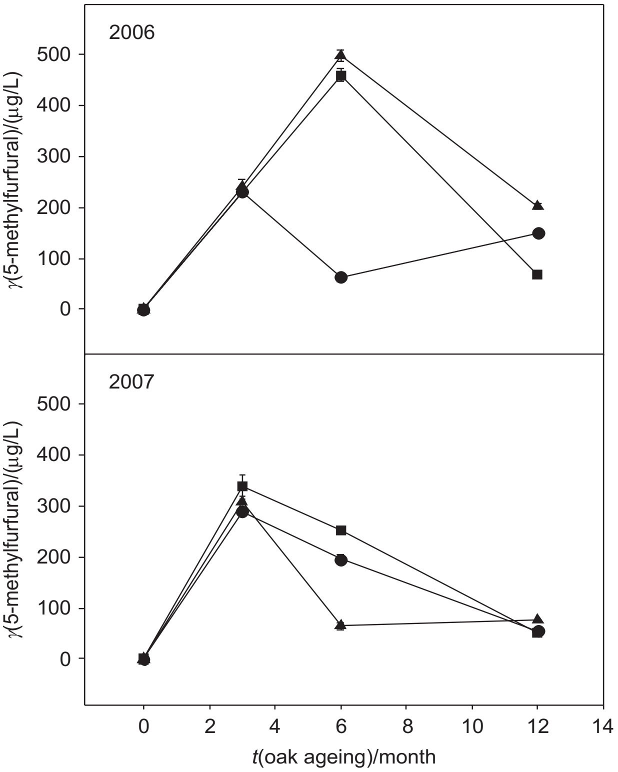

Fig. 2. Evolution of the concentration of 5-methylfurfural during barrel ageing in 2006 and 2007 for Monastrell (▲), Cabernet Sauvignon (●) and Petit Verdot (■) wines. Error bars represent the standard deviation of the mean value (N=3)

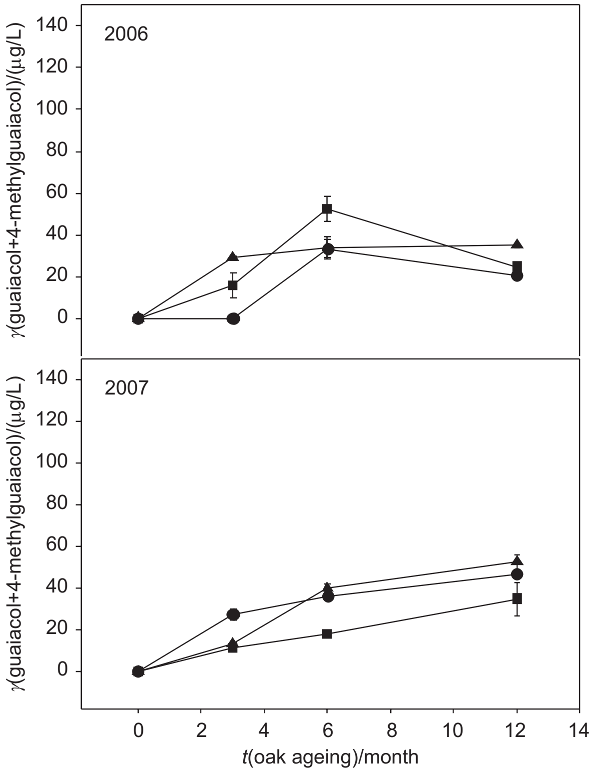

Fig. 3. Evolution of the concentration of the sum of guaiacol and 4-methylguaiacol during barrel ageing in 2006 and 2007 for Monastrell (▲), Cabernet Sauvignon (●) and Petit Verdot (■) wines. Error bars represent the standard deviation of the mean value (N=3)

Table 2. Correlation matrix for the initial physicochemical properties and oak-related volatile compounds in red wines

	TA	VA	pH	SO_2	Alcohol
F	ns	ns	ns	ns	ns
MF	ns	ns	ns	ns	ns
TL	–0.48*	–0.73***	0.55*	0.51*	–0.93***
CL	–0.64**	–0.47*	0.72***	ns	–0.67**
V	0.75***	ns	–0.83***	–0.43*	0.67**
G+MG	0.58*	ns	–0.72***	ns	ns

*p<0.05, **p<0.01, ***p<0.001, ns=not significant

Abbreviations: F: furfural, MF: 5-methylfurfural, G: guaiacol, MG: 4-methylguaiacol, TL: *trans*-lactone, CL: *cis*-lactone, V: vanillin, TA: titratable acidity (g of tartaric acid per L), VA: volatile acidity (expressed as g of acetic acid per L), SO_2: free sulphur dioxide

fural and any of the physicochemical parameters evaluated, which is coincident with the findings of Ortega-Heras *et al.* (*3*) and Garde-Cerdán *et al.* (*9*).

Guaiacol and 4-methylguaiacol

Guaiacol and 4-methylguaiacol have smoky aromas and are indicators of the relative toast level of barrels (*27,28*), since they are formed almost exclusively by the degradation of lignin during the toasting process.

In our study, very low quantities of these compounds were detected (Fig. 3) and very similar levels were reached in all three wines, although Monastrell wine showed slightly higher values, perhaps due to the higher acidity and alcohol content of that wine. Indeed, the correlation matrix showed that the effect of alcohol content and titratable acidity was positive and statistically significant for guaiacyl compounds. A high alcohol content and acidity could favour the alcoholysis of lignin and a higher amount of guaiacyl compounds could then be released, as suggested by many authors (*4,29–32*).

Cis- and trans-β-methyl-γ-octalactones

As can be seen in Figs. 4 and 5, *cis*-oak lactone was detected at a higher concentration than *trans*-oak lactone, with a *cis*/*trans* ratio of around 2, similar to the findings of Gómez-Plaza *et al.* (*33*). The *cis*-oak lactone content was lower in Monastrell wine than in the Petit Verdot and Cabernet Sauvignon wines, the latter two having a *cis*-oak lactone concentration three times higher than Monastrell wine, difference that will certainly affect the sensory characteristics of the wines. Similar behaviour was observed for *trans*-oak lactone, especially in 2006. This could be associated with the higher percentage of alcohol and titratable acidity observed in both years in Monastrell wines (Table 1). A higher percentage of alcohol could favour the open form of the lactones as was described by Waterhouse and Towey (*34*), and this phenomenon could explain the lower amount of lactones in Monastrell wine. Table 2 shows that the correlation between the alcohol content and the lactone concentration

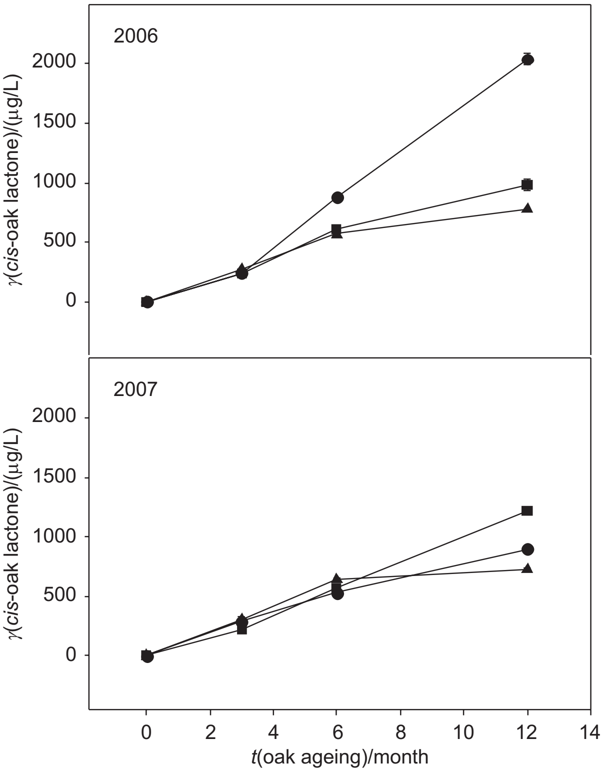

Fig. 4. Evolution of the concentration of *cis*-lactone during barrel ageing in 2006 and 2007 for Monastrell (▲), Cabernet Sauvignon (●) and Petit Verdot (■) wines. Error bars represent the standard deviation of the mean value (*N*=3)

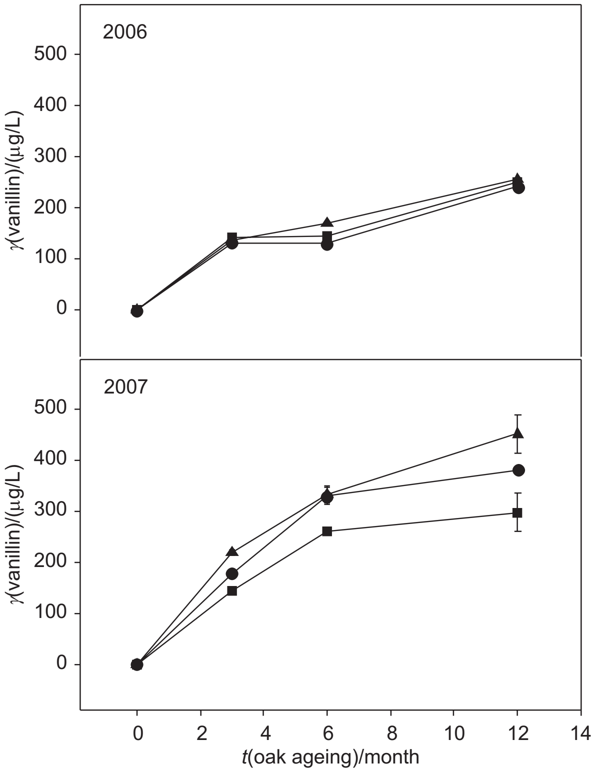

Fig. 6. Evolution of the concentration of vanillin during barrel ageing in 2006 and 2007 for Monastrell (▲), Cabernet Sauvignon (●) and Petit Verdot (■) wines. Error bars represent the standard deviation of the mean value (*N*=3)

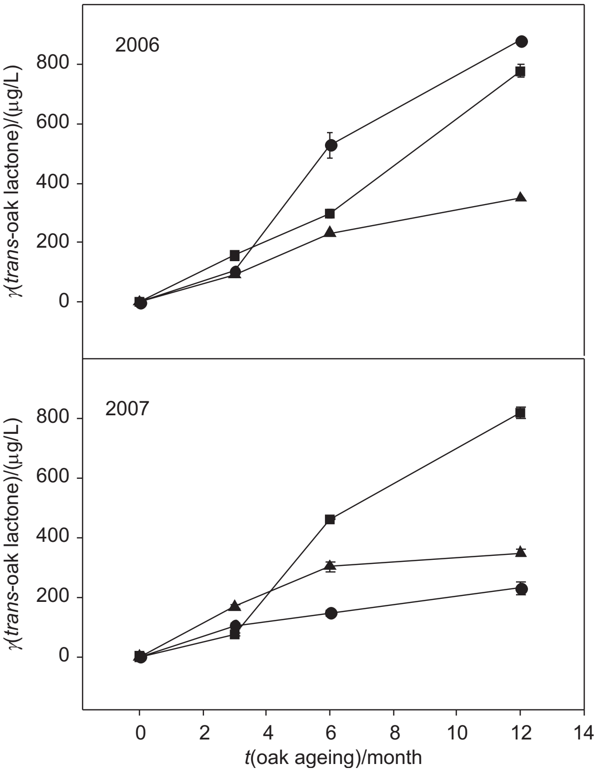

Fig. 5. Evolution of the concentration of *trans*-lactone during barrel ageing in 2006 and 2007 for Monastrell (▲), Cabernet Sauvignon (●) and Petit Verdot (■) wines. Error bars represent the standard deviation of the mean value (*N*=3)

was negative and highly significant (–0.93 for *trans*-oak lactone and –0.67 for *cis*-oak lactone). Also, since the presence of the open form of lactones could be reinforced by high acidity values, this could explain the significant negative correlation of the lactone concentration with titratable acidity (–0.49 and –0.64 for *trans*- and *cis*-oak lactone, respectively) and volatile acidity (–0.73 and –0.47 for *trans*- and *cis*-oak lactone, respectively). The importance of the alcohol content and acidity of the wines in lactone concentration was also observed in model solutions with oak chips (*12*). Cabernet Sauvignon, with the lowest alcohol content in 2006, and Petit Verdot wine in 2007 showed the highest concentrations of *cis*- and *trans*-oak lactones in these years.

Vanillin

In 2006, the level of vanillin was very similar in all wines, although slightly higher in Monastrell one, the differences being more pronounced in 2007. The correlation matrix indicated that the effect of alcohol content and titratable acidity was significant and positive for vanillin (0.67 and 0.75, respectively), probably because both parameters favoured alcoholysis of the lignin, as stated in previous studies (*4*,*12*,*35*). We also found a negative correlation between vanillin and the free SO_2 in the wines, perhaps because the carbonyl group of the phenolic aldehydes combines with the SO_2 (*36*). This finding also agrees with those of other authors (*3*,*7*).

Conclusions

The wines studied in this research were made from three different grape varieties and were aged in barrels with the same specifications and under the same ageing conditions (same cellar and oenological practices during ageing). The study was performed in two consecutive years. Our results showed high and significant correlations between some of the wine physicochemical parameters and the accumulation of oak-related volatile compounds, the level of significance of our results being higher than those reported by other authors. These results reinforced the fact that, when studying the factors affecting the accumulation of oak-related volatile compounds in aged wines, not only the oak barrel variables (oak origin and seasoning, size of the barrel, number of uses, *etc.*) influence the concentration of these compounds in the aged wine, but the effects of the physicochemical variables of the wine itself, especially titratable acidity and alcohol content, are also a very important issue that should be taken into account. Due to the correlations that our results have confirmed for several oak volatile compounds, a winemaker could predict, to a certain level, the impact of wood on the wine aroma, not only by choosing the appropriate size and toast level of the barrel, origin of the wood, and the length of ageing but also knowing the initial physicochemical properties of the wine.

Acknowledgements

The authors would like to acknowledge Finca Omblancas S.L. for their collaboration in the study.

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