



Chemical and chromatic characteristics of Tempranillo, Cabernet Sauvignon and Merlot wines from DO Navarra aged in Spanish and French oak barrels

Estrella Cadahía^a, Brígida Fernández de Simón^{a,*}, Miriam Sanz^a, Pilar Poveda^a, Javier Colio^b

^a Centro de Investigación Forestal (CIFOR-INIA), Apdo 8111, 28080 Madrid, Spain

^b Bodega Señorío de Otazu, Señorío de Otazu, 31174 Echauri, Navarra, Spain

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ABSTRACT

The oenological quality of Spanish oak wood from Navarra (*Quercus petraea* Liebl.) in relation to French wood of recognised quality in cooperage has been evaluated. We have studied the effects of the Spanish wood on wine evolution during ageing, in regards to chromatic characteristics, polyphenols, and volatile compounds related to oak wood, in three different mono-varietal wines (cv. Tempranillo, cv. Cabernet Sauvignon and cv. Merlot) from the Origin Denomination Navarra. The results were compared with those obtained for the same wines aged in French oak barrels from central regions of France, obtained from five different French and Spanish cooperages.

The wines aged in Spanish oak evolved in the same way as those aged in French. In general, at the end of the ageing process the differences in their chromatic and chemical characteristics were very small, although each wood type added special characteristics, the exact properties of which also depended on the grape variety. The Spanish oak wood (*Q. petraea* Liebl.) from Navarra has an oenological quality similar to French oak wood of the same species from Central region.

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1. Introduction

The widespread use of new oak barrels (*Quercus* spp.) in the ageing of wines in many winemaking regions of the world, including Spain, has generated an increase in demand, which has resulted in a necessity for new sources of quality wood for cooperage. Consequently, woods from different European countries (Hungary, Poland, Russia, Ukraine, Romania, Portugal, etc.) have entered the market, as alternatives to French (*Quercus petraea* and *Quercus robur*) and American (*Quercus alba*) oak woods. This has led to looking into the possibility of utilising Spanish oak as yet another alternative; interest has increased as the direct result of studies carried out on its oenological quality. According to the last Spanish forest inventory (III-IFE, 2002), different oak species found in Spain which may be used for oenological purposes include: *Q. petraea* Liebl. and *Q. robur* L., which are similar to the French species traditionally used in cooperage (Cadahía, Fernández de Simón, & Jalocha, 2003; Cadahía, Muñoz, Fernández de Simón, & García-Vallejo, 2001; Cadahía, Varea, Muñoz, Fernández de Simón, & García-Vallejo, 2001; Fernández de Simón, Cadahía, Conde, & García-Vallejo, 1996, 1999), and *Quercus pyrenaica* wild, which is native to the Iberian peninsula (Fernández de Simón, Sanz, Cadahía, Poveda, & Broto, 2006). *Q. petraea* predominates in the north and northeast of the peninsula, showing similar wood

qualities to that of other oak woods commonly used in cooperage. The oak wood from Navarra (*Q. petraea*) has very fine or fine grain, and similar chemical composition (polyphenols, tannins and volatile compounds) to that of the best quality French wood, and some specific characteristics valued for use in oenology such as high concentrations of *cis*- β -methyl- γ -octalactone. Its behaviour during the barrel making process, when considering the evolution in chemical composition during natural wood seasoning and toasting, was also similar to that shown by French oak woods (Cadahía, Fernández de Simón, Vallejo, Sanz, & Broto, 2007). Nevertheless, further studies on its behaviour during wood–wine interaction must be carried out.

The structural characteristics of wood (grain, porosity and permeability) and chemical composition (polyphenols, tannins and volatile compounds) can influence the complex physical, chemical and biochemical processes that take place during the oxidative ageing of wine in barrels, affecting their composition and organoleptic properties, and contributing to their stability. The simple extraction of aromatic compounds (volatiles and polyphenols), and tannins from wood can add a richness and complexity to the aroma and taste of wines (Díaz-Plaza, Reyero, Pardo, Alonso, & Salinas, 2002; Fernández de Simón, Cadahía, & Jalocha, 2003; Fernández de Simón, Hernández, Cadahía, Dueñas, & Estrella, 2003; Pérez-Prieto, López-Roca, Martínez-Cutillas, Pardo-Mínguez, & Gómez-Plaza, 2002). In fact, a great variety of aromatic compounds belonging to very different chemical families, like phenolic aldehydes, phenolic ketones and their isomers, volatile phenols, lactones, furanic compounds,

* Corresponding author. Tel.: +34 913476789; fax: +34 913476767.

E-mail address: fdesimon@inia.es (B. Fernández de Simón).

and pyranones, among others, have been identified in oak wood (Cadahía et al., 2003; Cutzach, Chatonnet, Henry, & Dubourdieu, 1997; Fernández de Simón et al., 2006).

In addition, polyphenolic compounds extracted from wood can interact with polyphenolic components in wine, contributing directly or indirectly to colour stability and other sensory and chemical changes. They take part in the oxidation process that happens during wine ageing along with anthocyanins and wine flavonoids. The relationship between colour evolution and stability, and anthocyanin and flavanol compounds of wines, as well as the effects of oak wood on the levels of the anthocyanin derivatives formed during ageing, are well-known (Pérez-Magariño & González-Sanjosé, 2004; Revilla & González-Sanjosé, 2001). Diverse condensation products are thought to result from the reactions between wine flavanols and anthocyanidins, in which different oak wood phenolic and furanic aldehydes serve as intermediates, or even, from direct reaction of oak wood phenolic aldehydes with the flavanols and anthocyanidins (Eas-Safi, Cheyner, & Moutounet, 2000; Nonier Bourden et al., 2008; Nonier, Vivas, Vivas de Gauejac, Pianet, & Fouquet, 2007; Sousa, Mateus, Perez-Alonso, Santos-Buelga, & De Freitas, 2005; Sousa et al., 2007), contributing to wine colour and astringency evolution. The ellagitannins from oak wood also play an important role in these wine attributes. They behave as antioxidants, due to their ability to consume high quantities of oxygen, regulating the oxidation reactions. They can undergo numerous chemical transformations, e.g., intervening in condensation processes of wine anthocyanins and tannins with acetaldehyde (Vivas & Glories, 1996) or reacting with flavanols (Saucier, Jourdes, Glories, & Quideau, 2006).

The wood physical and chemical characteristics are, therefore, important quality factors in the wine ageing process, since they affect the wood–wine interaction phenomena, such as oxygen-diffusion, compound extraction from wood, and oxidation processes in wines (Cutzach, Chatonnet, & Dubourdieu, 2000; Fernández de Simón, Hernández, et al., 2003; Hernández, Estrella, Dueñas, Fernández de Simón, & Cadahía, 2007; Matejíček, Mikes, Klejdus, Sterbová, & Kuban, 2005). On the other hand, however, all these processes have an important varietal influence. The polyphenolic composition in wine depends on grape variety, edaphoclimatic and cultural conditions, and the winemaking process (Fernández de Simón et al., 2008; Monagas, Gómez-Cordovés, Bartolomé, Laureano, & Ricardo da Silva, 2003; Pérez-Magariño & González-Sanjosé, 2004; Revilla, López, & Ryan, 2005; Sun & Spranger, 2005). So, different mono-varietal wines in identical conditions of ageing can leech polyphenols and volatiles from wood to varying degrees and at different paces.

The main goal of this research is to evaluate the oenological quality of Spanish oak wood from Navarra (*Q. petraea*), in relation to that of the French of comparable quality in cooperage. We studied the effects of Spanish wood on the evolution, during ageing, of chromatic characteristics as well as polyphenols and oak-related volatile compounds, in three different mono-varietal wines from the Origin Denomination Navarra. The results were compared with those obtained for the same wines aged in French oak barrels from different French and Spanish cooperages.

2. Materials and methods

2.1. Barrels

The Spanish oak barrels (12 barrels) were made following a traditional process. Oak heartwood from *Q. petraea* grown in Navarra (Garaioa) was harvested in January 2002 and placed in a cooperage in Álava province, where it was split and the staves naturally seasoned in the open air for two years. During this process, staves

were subjected to environmental conditions and watering at the cooperage. The barrels were toasted at medium intensity level in the traditional way over a wood fire. Barrels of French oak from the Central region with very fine grain and medium intensity toasting were provided by different French and Spanish cooperages (12 barrels × 5 cooperages = 60 barrels). All barrels were new, and with a capacity of 225 L.

2.2. Wines

Three red wines from DO Navarra (Spain) were produced on an industrial scale by Otazu wine cellar in 2003, from cv. Tempranillo (100%), cv. Cabernet Sauvignon (100%), and cv. Merlot (100%) grapes, according to traditional methods. The habitual oenological parameters of these wines (Table 1) were determined at wine cellars before they were transferred to the barrels, according to the OIV methods (1990). They were put into the barrels in February 2004 to February 2005, and finally bottled. During this storage time, wine samples from each barrel were taken, after ageing 3, 6, 9 and 12 months. A control sample of each wine was also taken before the wine was put into the barrels and was analysed in the same way. All samples were analysed in duplicate.

2.3. Colour parameters and total polyphenol determinations

A direct measurement of wine absorbance at 420, 520 and 620 nm was carried out in a Du 70 Beckman spectrophotometer with a quartz cell of 1 mm path length. From the absorbance values the following variables were then calculated according to Glories procedure (1984), colour intensity, colour tonality, proportion of red, blue, and yellow. A variable responsible for the proportion of red produced by the flavilium cation of the free and bound anthocyanidins (pure red) (dA%) was also determined. Total polyphenols (TP) were evaluated directly in wine by reaction with Folin–Ciocalteu reagent (Singleton & Rossi, 1965).

2.4. HPLC polyphenol analyses

The wine samples from each barrel were analysed separately, following the previously described method by Fernández de Simón, Hernández, et al. (2003), with some modifications. Samples, concentrated to 25% of their initial volume, were extracted with diethyl ether and ethyl acetate. The organic fractions were combined and evaporated to dryness, and the residue re-dissolved in MeOH–H₂O (1:1) to be analysed by RP-HPLC-DAD. The liquid chromatography system used was a HP1200, equipped with a diode array detector and a C18 Hypersil ODS (5 µm) column (25 cm × 4 mm id). The elution conditions were as follows: flow rate, 1 mL/min; temperature, 30 °C; solvent A = water/acetic acid

Table 1
Oenological parameters of red single-variety wines.^a

	Tempranillo	Merlot	Cabernet Sauvignon
Alcohol level (% v/v)	14.0	14.1	14.3
Dry extract (g/L)	32.3	28.4	32.0
pH	4.12	3.66	3.77
Total acidity ^b (g/L)	4.9	5.4	4.9
Malic acid (g/L)	0.2	0.2	0.2
Sugar (g/L)	1.2	1.5	1.4
Volatile acidity ^c (g/L)	0.52	0.35	0.35
Free SO ₂ (mg/L)	26	23	ND
Total SO ₂ (mg/L)	35	40	54

ND, not determined.

^a Measured according to OIV methods (1990).

^b Expressed as tartaric acid.

^c Expressed as acetic acid.

(98:2 v:v), solvent B = water/acetonitrile/acetic acid (78/20/2 v:v:v); gradient profile, 0–55 min, 100–20% A, 55–70 min, 20–10% A, 70–80 min, 10–5% A, 80–90 min, 5–0% A, 90–100 min, 0% A. Detection was performed by scanning from 190 to 400 nm. The quantification of compounds was carried out by the external standard method, using detection at 280, 310 and 340 nm depending on their UV spectra.

2.5. GC–MS volatile analyses

Volatile compounds were extracted from wines and analysed following the method described by Fernández de Simón, Cadahía, et al. (2003). Analyses were performed using a Hewlett–Packard 6890N gas chromatograph (Palo Alto, CA) equipped with a mass spectrophotometric detector model HP 5975B. Samples were injected in split mode (30:1, 0.5 min), and volatiles were separated using a fused silica capillary column (SUPELLOWAX-10) (30 m × 0.25 mm id, and 0.25 µm film thickness), supplied by Supelco (Madrid, Spain), and under the working conditions described by Fernández de Simón, Cadahía, et al. (2003). Quantitative determinations were carried out by the internal standard method, using peak areas obtained from selected ion monitoring (SIM). The selected ions for each one of the evaluated compounds were: 95/**96** furfural, 109/**110** 5-methylfurfural, **97**/126 5-hydroxymethylfurfural, 97/**98** (furfuryl alcohol), **99** (*cis*- and *trans*-β-methyl-γ-octalactone), **126** (maltol), 109/**124** (guaiacol), 123/**138** (4-methylguaiacol), **94** (phenol), **107**/108 (*o*-cresol, *p*-cresol, and *m*-cresol), **164** (eugenol and isoeugenol), 139/**154** (syringol), **168** (4-methylsyringol), **194** (4-allylsyringol), 151/**152** (vanillin), 151/**166** (acetovanillone), **151**/180 (propiovanillone), **151**/194 (butirovanillone), **137**/168 (methyl vanillyl ether), **137**/182 (ethyl vanillyl ether), 181/**182** (syringaldehyde), **181**/196 (acetosyringone), and **181**/210 (propiosyringone). The *m/z* ion in boldface letter was used for quantification. The concentrations of each substance were measured by comparison with calibrations made with pure reference compounds analysed under the same conditions. The corresponding calibration was made for each compound, and linear regression coefficients between 0.98 and 0.999 were obtained.

2.6. Statistical analyses

Univariate analyses were performed using ANOVA, applying the Student Newman–Keuls multiple range test. Multivariate canonical discriminant analyses were also carried out with the evaluated compounds, using the SAS statistical programme (version 6, SAS Institute, Cary, NC).

3. Results and discussion

3.1. Colour characteristics and total polyphenols

Table 2 shows the mean values of colour parameters, and total phenol index in wines after 12 months of ageing in the different types of woods. Comparing the final wines (Na and Fr) of each variety (Tempranillo, Merlot and Cabernet Sauvignon), only insignificant differences between the wines aged in Spanish oak from Navarra and French oak from the Central region were observed, considering the colour parameters (intensity, tonality, % yellow, % red, % blue, and pure red) and the total phenol index. In general, the differences in wine chromatic characteristics at the end of ageing have been related to differences in the wood structural and chemical characteristics (Fernández de Simón, Hernández, et al., 2003; Revilla & González-Sanjosé, 2001). Our results indicate similar behaviour by both woods during ageing, which can be explained, at least in part, by the similarity of their grain and

ellagitannin contents, both of which influence the oxidation processes, and by the polymerisation and condensation reactions of the anthocyanins. Moreover, the colour evolution, during ageing, was as expected, similar in the three mono-varietal wines. The majority of the parameters changed in the final wines (aged 12 months) relative to the initial ones. An increase in the tonality and yellow percentage, and a decrease in the red percentage and pure red (dA%) were observed, while no significant variations were detected in the colour intensity and total phenol index. These results are in agreement with data previously published (Gómez-Codovés & González-Sanjosé, 1995; Pérez-Magariño & González-Sanjosé, 2004).

3.2. Nonanthocyanin polyphenols

The HPLC analysis of diethyl ether/ethyl acetate extract from Tempranillo, Merlot and Cabernet Sauvignon wines allowed the identification of a wide variety of nonanthocyanin phenolic compounds, including nonflavonoid phenolic compounds (hydroxybenzoic and hydroxycinnamic acids and their derivatives, stilbenes, and phenolic alcohols) and flavonoid compounds (flavanols and flavonols). Most of them have been previously described in Tempranillo (Fernández de Simón, Hernández, et al., 2003), Merlot and Cabernet Sauvignon wines (Monagas et al., 2003) and other varieties of *Vitis vinifera* L. (Castillo-Muñoz, Gómez, García, & Hermosín, 2007), but others were previously described as coming from the wood (Fernández de Simón, Hernández, et al., 2003; Hernández et al., 2007; Matejíček et al., 2005). From among them, we have quantified only 27 compounds, selected on the basis of their concentration in wine and their sensory potential. Benzoic and cinnamic aldehydes would have been included in this compound group, but once again, they were not detected in diethyl ether/ethyl acetate extracts from wines, so they were analysed by GC–MS with the volatile compounds. Their absence has been attributed to the tendency to react with wine glycols forming acetal derivatives during the extraction process with organic solvents, and successive concentrations carried out previously by HPLC analysis (Spillman, Pollnitz, Liacopoulos, Skouroumounis, & Sefton, 1997). The HPLC quantitative evaluations of the 27 polyphenolic compounds obtained in the three initial wines and in the same ones after 12 months of ageing in Spanish (*Q. petraea* from Navarra) and French (*Q. petraea* from Central region) oak woods, are grouped by chemical families shown in Fig. 1.

3.2.1. Non-flavonoid compounds

Looking at the initial wines, the hydroxybenzoic acids and their derivatives showed similar levels in the three varieties, although the concentration of ethyl gallate was slightly higher in Merlot wines, in accordance with other authors (Monagas, Suárez, Gómez-Codovés, & Bartolomé, 2005). However, greater differences were found in hydroxycinnamic acid levels, highlighting the high concentration of caffeic and *p*-coumaric acids in Tempranillo wine, and of caftaric acid in Cabernet Sauvignon wine. Monagas et al. (2005) also found a high concentration of caftaric acid in the Cabernet Sauvignon variety. Among the alcohols and stilbenes, only the tryptophol showed differences, its concentration being smaller in Cabernet Sauvignon than in Tempranillo and Merlot wines.

During ageing, changes in the concentrations of the majority of phenolic compounds of the wines were observed (Fig. 1). These depended on the grape variety, oak wood kind, and permanence time of the wine in the barrels. In general, the concentration of the hydroxybenzoic acids and their derivatives increased slowly in wines aged for 12 months relative to the initial wines. Specifically, the gallic, *p*-hydroxybenzoic, and ellagic acids, and ethyl gallate showed concentrations significantly higher in the aged wines than

Table 2
Global determinations and colour percentage intensity in red single-variety wines, after 12 months of ageing in Spanish and French oak wood barrels. dA% pure red, TA total anthocyanidins (mg/L), TP total polyphenols (g/L).

	Tempranillo			Merlot			Cabernet Sauvignon		
	W	Na	Fr	W	Na	Fr	W	Na	Fr
Intensity	11.7ab	11.4 b	12.3ab	12.0ab	12.2ab	12.7ab	11.7ab	13.6a	13.0ab
Tonality	0.780b	0.856a	0.855a	0.670e	0.787b	0.788b	0.730d	0.759c	0.760c
% Yellow	38.2c	40.9a	40.7a	36.6e	39.9b	39.8b	37.6d	38.7c	38.7c
% Red	49.1d	47.7e	47.6e	54.8a	50.7c	50.5c	51.7b	50.9c	50.9c
% Blue	12.6a	11.4b	11.7b	8.5e	9.3d	9.7d	10.7c	10.4c	10.3c
dA%	48.3d	45.3e	44.9e	58.8a	51.4c	51.0c	53.3b	51.8c	51.8c
TP	3.84a	3.74a	3.60a	2.92a	3.03a	2.98a	3.61a	3.43a	3.45a

Different letters in the same row denote a statistical difference with 95% confidence level (Student–Newman–Keuls multiple range test) that was carried out excluding the initial wine. W, initial wines; Na = wines aged in *Quercus petraea* Spanish oak from Navarra and Fr, wines aged in *Q. petraea* French oak from central region.

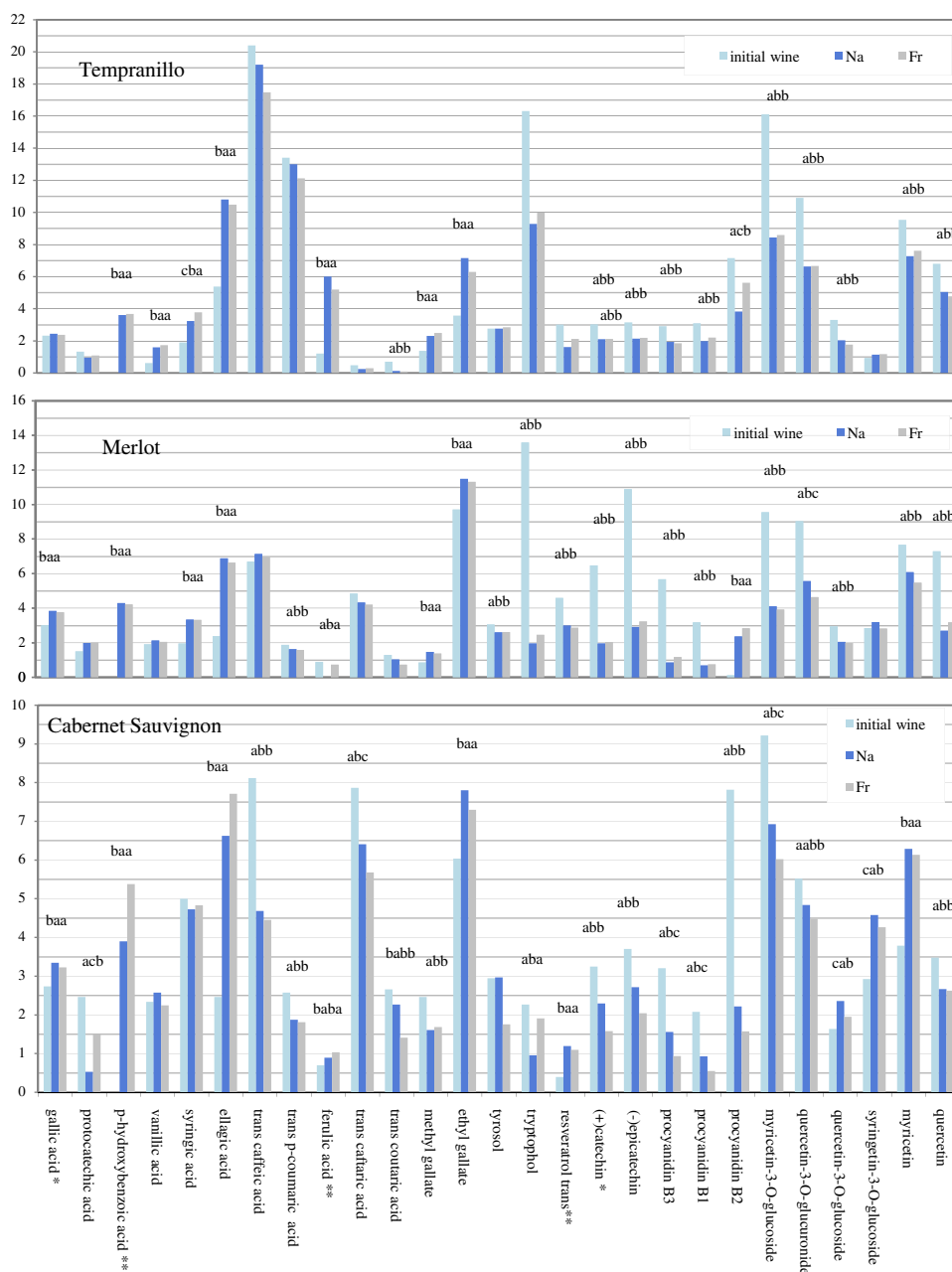


Fig. 1. HPLC quantitative evaluation of low molecular weight phenolic compounds (mg/L) in the red single-variety wines after 12 months of ageing in Na: Spanish (*Q. petraea* from Navarra) and Fr: French (*Q. petraea* from central region) oak woods. (*) Concentrations were divided by 10. (**) Concentrations were multiplied by 10. Different letters in each compound denote a significant difference with 95% confidence level in the Student–Newman–Keuls multiple range. Compounds without letters denote no significant differences.

in the initial ones. Only insignificant differences were obtained between wines from Spanish (Na, Fig. 1) and French (Fr) oak wood, even for gallic and ellagic acids, whose levels in wine have been related to elagitannin contents in each type of wood (Fernández de Simón, Hernández, et al., 2003). Hydroxycinnamic acid concentrations, on the other hand, decreased in the three wines during ageing, except in the case of ferulic acid. These decreases were major for *trans*-caffeic acid in Cabernet Sauvignon wines and again, only insignificant differences were obtained when comparing wines from Spanish and French oak woods. Similar and contrary behaviours for the benzoic and cinnamic acids have been described in previous works, carried out with the same varieties of wines but from different origins (Fernández de Simón, Hernández, et al., 2003; Martínez, 2004; Matejíček et al., 2005). The evolution of phenolic compounds in wine during ageing is complex because of the wide variety of factors involved. The lignin and ellagitannin degradation from oak wood and subsequent release of these products into the wine can explain the increase of the hydroxybenzoic acids during ageing. Phenolic compounds, however, are subjected to degradation reactions, and also to sorption processes into new polymerised compounds in wine. The prevalence of different mechanisms depending on the conditions could explain the contrary results reported by some authors. The decrease in cinnamic acids obtained in this study could be due to their involvement in oxidative condensation and copigmentation processes during ageing (Monagas et al., 2005; Rentzsch, Schwarz, Winterhalter, & Hermosín-Gutiérrez, 2007; Yokotsuka & Singleton, 2001).

Other non-flavonoid compounds (Fig. 1), like alcohols (mainly tryptophol) and stilbenes (*trans*-resveratrol) also showed changed levels in wines, decreasing or increasing during ageing, depending on the variety. However, only insignificant differences between wines from Spanish and French woods were observed, in accordance with Fernández de Simón, Hernández, et al. (2003) and Martínez (2004). The *trans*-resveratrol concentration was especially low in Cabernet Sauvignon wines (0.4 mg/L, Fig. 1) in relation to the other varieties studied, and similar to those previously described by Monagas et al. (2005) in the same variety. These small differences could be attributed to variations in the proportion of resveratrol in its free form and glycoside derivatives, since for example, the glucose forms are supposed to be cleaved to their aglycon forms by hydrolysis (Mattivi, Reniero, & Korhammer, 1995), and the stilbene profile of wines depends on multiple factors such as grape variety, fungal infection, winemaking procedure, and climatological conditions (Moreno-Labanda et al., 2004).

3.2.2. Flavonoid compounds

Flavanols were the principal flavonoid compounds in the three wines, especially due to the high concentrations of (+)-catechin (Fig. 1). The initial Merlot wine showed the highest levels of catechin, epicatechin, and procyanidins B3 and B1, but also the greatest decrease in these compounds during ageing. Tempranillo and Cabernet Sauvignon wines exhibit similar concentrations to each other in initial wines, slightly decreasing during ageing, with Cabernet Sauvignon decreasing slightly more than Tempranillo. The levels of all flavanols, except of procyanidin B2, were significantly different in aged wines when compared to initial wines. However, only insignificant differences were obtained when comparing wines from Spanish and French oak woods, except for the levels of procyanidin B2 in Tempranillo, and of procyanidin B3 and B1 in Cabernet Sauvignon wines. Previous studies have described similar decreases in catechin and epicatechin, and dimeric derivatives, which depend on the characteristics of oak wood barrels (Fernández de Simón, Hernández, et al., 2003; Martínez, 2004), in addition to other parameters like grape variety (Monagas et al., 2005). This decrease in free flavanols takes place in conjunction with an increase in the levels of polymerised derivatives (like trimers and tetra-

mers) and of anthocyanin derivatives or “new pigments”, that contribute to the maintenance of colour intensity and the addition of blue-violet hues in aged wines (Pérez-Magariño & González-Sanz, 2004). In these and other polymerisation and copigmentation processes the wood plays an important role, since physical characteristics like grain, porosity, or permeability, and polyphenolic composition, influence the amount of available oxygen which ends up in the wine. In addition, many wood polyphenols can take part in these processes. For example, phenolic and furanic aldehydes from oak wood serve as intermediates in the reactions between flavanols and anthocyanins, while diverse condensation products have been described as resulting from the direct reaction of oak wood aldehydes with the flavanols or anthocyanins of wine (Eas-Safi et al., 2000; Nonier Bourden et al., 2008; Nonier et al., 2007; Sousa et al., 2005, 2007), contributing to wine colour and astringency evolution during ageing.

In relation to the flavonols, the glycosidic derivative concentrations decreased in the three wines (Fig. 1) during ageing, except for quercetin-3-*O*-glucoside in Cabernet Sauvignon wine, and syringetin-3-*O*-glucoside in all cases. This decrease could go together with the increase in the corresponding aglycones, as a result of the glycosidic compound hydrolysis (Zafrilla et al., 2003). However, this has only been shown for myricetin in Cabernet Sauvignon wine. Other oxidation and condensation reactions involving aglycones and glycosidic derivatives may explain the myricetin and quercetin decreases (Gutiérrez, Sánchez, & Vicario, 2005). The influence of oak wood on the flavonol profiles of final wines was small, and no significant differences were observed between wines from Spanish and French woods.

In order to compare the behaviour of wines aged in barrels made of the studied oak woods, different canonical discriminant analyses for each variety were carried out, using data of overall phenolic compounds at the end of ageing (data in Fig. 1), and also at three and nine months (data not published). Fig. 2 (graphics I, II, and III) illustrates the graphical representations on the plane defined by the two principal canonical axes, and Table 4 the total canonical structure coefficients, obtained for the canonical functions. Another canonical discriminant analysis comparing the three varieties at the end of ageing was also made (Fig. 2, graphic IV). The phenolic composition of wines evolved during the ageing process, showing different profiles as a function of stay time in barrel and grape variety, and not of the kind of oak wood. The canonical analyses showed patterns of distribution in which four well-defined groups could be distinguished: initial wines (T), wines aged for three months (A, B), for nine months (C, D), and for 12 months (E, F), even though the ones aged for 9 and 12 months were very close (graphics I, II, and III, in Fig. 2). Each variety demonstrated different responses to identical ageing conditions, according to the distribution patterns for each mono-varietal wine, and the contribution of the variables in the principal canonical functions (Fig. 2, Table 4). In general, wines aged in Spanish oak wood barrels behaved similar to those from barrels of French wood, which shows that both wood types exhibit a similar influence on the oxidation process during ageing. Although after short periods of ageing it was possible to see little differences among them (for example in the Tempranillo variety), in the end these differences disappeared. The canonical discriminant analysis (graphic IV, in Fig. 2) that compares the three mono-varietal wines at the end of ageing illustrates the predominance of variety over the effect of the wood.

3.3. Volatile compounds

Among the wide variety of volatile compounds identified in oak woods which are able to give higher aromatic complexity to wines aged in barrels (Cadahía et al., 2003; Cutzach et al., 1997;

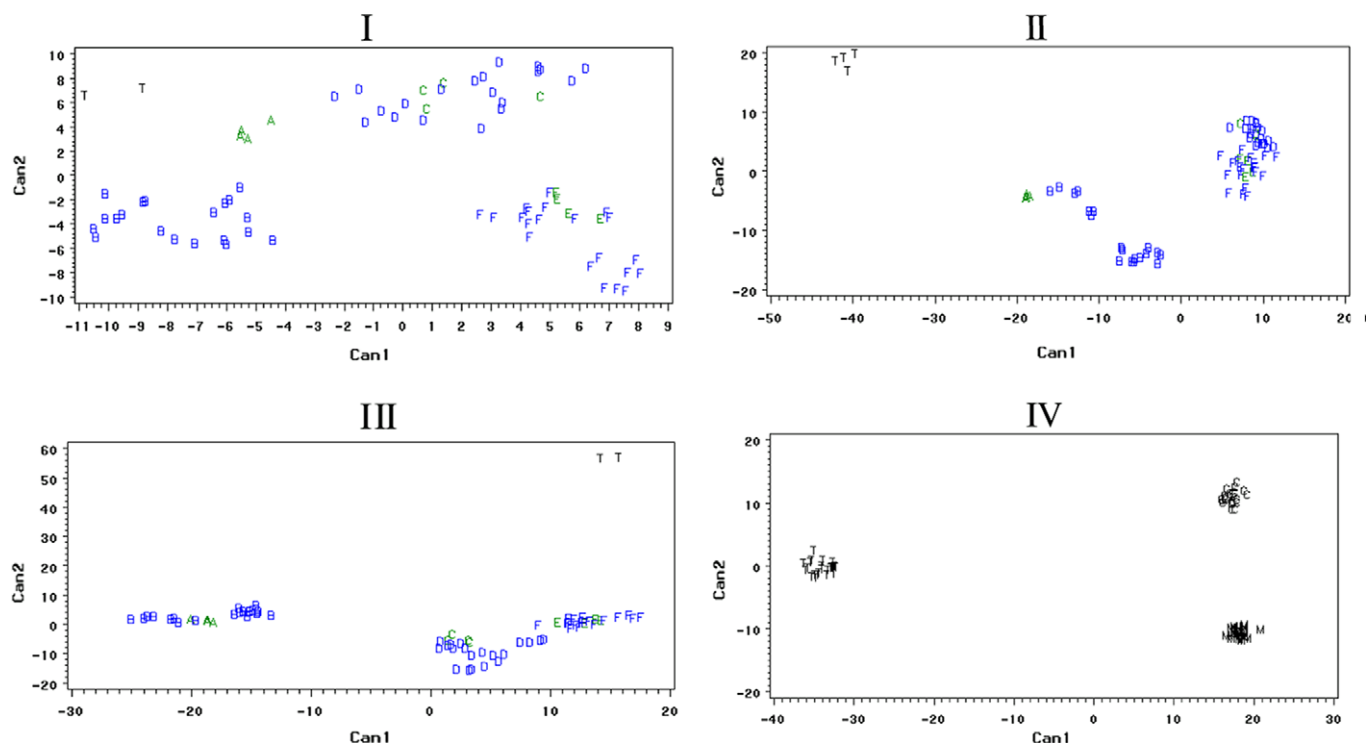


Fig. 2. Canonical discriminant analysis of the evolution of low molecular weight phenolic compounds in the red single-variety wines, cv. Tempranillo (I), Merlot (II) and Cabernet Sauvignon (III), during ageing in Spanish (*Q. petraea* from Navarra) and French (*Q. petraea* from Central Region) oak woods. T = initial wines, three months (A = Navarra, B = France), nine months (C = Navarra, D = France) and 12 months of ageing (E = Navarra, F = France). Graphic IV: canonical discriminant analysis for the three wines, cv. Tempranillo (T), Merlot (M) and Cabernet Sauvignon (C) at 12 months of ageing in the different oak woods. I (total variance: 30% and 26%, canonical correlation: 0.989 and 0.987 and eigenvalues: 43.4 and 37.2, for can1 and can2, respectively). II (total variance: 51% and 21%, canonical correlation: 0.997 and 0.994 and eigenvalues: 197.5 and 82.1, for can1 and can2, respectively). III (total variance: 55% and 18%, canonical correlation: 0.988 and 0.983 and eigenvalues: 223.5 and 72.2, for can1 and can2, respectively). IV (total variance: 89% and 11%, canonical correlation: 0.999 and 0.994 and eigenvalues: 632.3 and 79.5 for can1 and can2, respectively). The total canonical structure coefficients of can1 and can2 for each graphic are included in Table 4.

Table 3
GC quantitative evaluation in $\mu\text{g/L}$ of volatile compounds (furanic compounds, lactones, pyranones, volatile phenols, phenolic aldehydes and related compounds) in red single-variety wines, after 12 months of ageing in Spanish and French oak wood barrels.

	Tempranillo			Merlot			Cabernet Sauvignon		
	W	Na	Fr	W	Na	Fr	W	Na	Fr
Furfural	0.09	165b	155b	11.1	54.9b	73.9b	5.50	851a	836a
5-Methylfurfural	0.02	74.6b	46.7b	0.66	119b	109b	–	276a	289a
5-hydroxymethylfurfural	0.61	77.9ab	44.6b	22.8	51.7b	43.4b	31.5	100ab	114a
Furfuryl alcohol	2.62	24700a	21039a	57.6	12100a	13500a	23.5	726a	629a
<i>trans</i> - β -Methyl- γ -octalactone	–	44.6a	25.0a	–	36.3a	33.8a	–	24.0a	49.5a
<i>cis</i> - β -Methyl- γ -octalactone	–	253b	154c	–	322a	152c	–	214bc	142c
<i>cis/trans</i> Ratio	–	5.7	6.2	–	8.9	4.5	–	8.9	2.9
Maltol	1.77	150a	150a	51.0	105a	132a	50.7	92.4a	111a
Guaiacol	0.24	34.4a	28.1ab	3.26	14.5c	28.9ab	3.32	11.2c	19.6bc
4-Methylguaiacol	0.02	11.8ab	9.80ab	1.31	2.69b	15.9a	1.28	2.66b	7.22ab
Phenol	0.16	12.3bc	15.7a	4.11	10.9bc	10.1c	5.75	13.7b	12.4bc
<i>o</i> -Cresol	0.02	2.45a	2.87a	0.81	1.34bc	1.54b	1.31	0.74c	1.35bc
<i>p</i> -Cresol	0.07	3.61ab	4.16a	3.09	3.27b	3.64ab	3.77	2.90b	3.21b
<i>m</i> -Cresol	0.04	0.94ab	1.10a	0.78	0.75b	0.93ab	1.03	0.65b	0.68b
Eugenol	0.18	25.0ab	32.7a	0.90	20.5ab	25.2ab	0.94	17.15b	20.5ab
Isoeugenol	0.09	18.9a	11.5ab	3.61	1.54b	13.6ab	3.47	4.00b	7.78ab
Syringol	1.35	81.7a	72.5a	20.6	37.6b	74.3a	28.1	34.1b	73.5a
4-Methylsyringol	0.08	9.91a	9.37a	5.74	4.02a	12.6a	5.58	2.89a	7.66a
4-Allylsyringol	0.37	24.9a	31.1a	1.69	7.17bc	15.1b	1.01	5.05c	8.68bc
Vanillin	0.10	42.8a	48.4a	3.62	43.4a	42.6a	1.78	45.7a	62.0a
Acetovanillone	1.92	61.7ab	83.4a	49.1	44.7bc	61.0ab	19.6	22.0c	24.4c
Propiovanillone	0.24	47.6a	47.7a	11.8	28.5bc	36.2ab	5.29	18.2c	19.3c
Butirovanillone	1.61	197a	218a	16.6	90.9b	123b	20.9	107b	108b
Methyl vanillyl ether	16.6	448a	503a	243	176c	246b	95.1	82.1d	80.6d
Ethyl vanillyl ether	3.51	198ab	225a	34.6	123bc	175abc	29.2	100c	119bc
Syringaldehyde	0.08	12.2a	9.02a	4.12	3.38a	23.0a	2.74	5.40a	23.3a
Acetosyringone	0.36	14.3a	22.3a	15.2	11.4a	19.6a	4.37	7.26a	11.4a
Propiosyringone	0.48	36.9ab	44.1a	12.7	24.7bc	33.7ab	5.99	17.0c	23.5bc

Different letters in the same row denote a statistical difference with 95% confidence level (Student–Newman–Keuls multiple range test) that was carried out excluding the initial wine. W, initial wines; Na = wines aged in *Quercus petraea* Spanish oak from Navarra and Fr, wines aged in *Q. petraea* French oak from central region.

Table 4

Total canonical structure coefficients of the main discriminant variables in can1 and can2, obtained for phenolic compounds (Fig. 2). The non-showed coefficients showed values smaller than 0.500 in can1 and 0.400 in can2.

Graphic	I		II		III		IV	
	Can1	Can2	Can1	Can2	Can1	Can2	Can1	Can2
Gallic acid		0.565	0.756	0.496	0.896		0.886	
Protocatechic acid				0.475		0.517		
<i>p</i> -Hydroxybenzoic acid			0.719		0.876			
Vanillic acid	0.500	−0.455			0.669		0.591	
Syringic acid	0.592		0.688		0.730			0.865
Elagic acid	0.923		0.600	0.654	0.776		−0.712	
Caffeic acid						0.772	−0.947	
<i>trans</i> - <i>p</i> -Coumaric acid		0.453			0.603	0.480	−0.967	
Ferulic acid					0.577		−0.949	
Caftaric acid		0.481			−0.549		0.945	
<i>trans</i> -Coutaric acid			−0.535				0.674	0.414
Tyrosol								−0.431
Tryptofol	−0.684		−0.686		−0.748		−0.963	
Methyl gallate	0.589		0.698		0.784	0.498	−0.928	
Ethyl gallate	0.725		0.537	0.664	0.871		0.617	−0.685
(+)-Catechin	−0.568		−0.509	0.677	−0.681			
(−)-Epicatechin			−0.514	0.701	−0.799			−0.554
Procyanidin B3			−0.585	0.563	−0.833		−0.623	
Procyanidin B1	−0.571	−0.566		0.657	−0.549		−0.939	
Procyanidin B2		−0.677					−0.826	
Myricetin-3- <i>O</i> -glucoside	−0.753		−0.753				−0.863	0.452
Hyperoside			−0.573	0.553			−0.831	
Isoquercitrin				0.671				
Myricetin				0.929		−0.512	−0.836	
Flavonol F6				0.611		−0.636	0.857	0.405
Quercetin				0.857		−0.424	−0.864	
Resveratrol				0.464	−0.810			−0.813

Fernández de Simón et al., 2006), in the wines studied we have analysed the 27 most important, considering their concentrations in the oak wood, and their aromatic potential. They are grouped in chemical families with quite varied structures, such as furanic compounds, lactones, phenols, pyranones, phenolic aldehydes, phenolic ketones, and others. Table 3 shows the concentrations obtained by GC–MS analysis in both initial wines and after 12 months of ageing in barrels made from the Spanish and French woods. A variance analysis was carried out considering two factors, the type of wood used for ageing and the grape variety, excluding the initial wines, in which the majority of compounds showed negligible concentrations when compared to the aged ones.

Concerning furanic compounds (Table 3), at the end of the ageing process only insignificant differences were observed between wines of the same variety from Spanish and French woods. However, not all wines had the same extraction capacity when it came to the furanic aldehydes from barrel wood; Cabernet Sauvignon wines reached higher levels of furfural, 5-methylfurfural and 5-hydroxymethylfurfural than Tempranillo and Merlot ones. This effect has previously been seen with regards to wine characteristics (alcohol level, pH) and geographical origin (Garde-Cerdán et al., 2008). Also, the furanic aldehydes can take part in many common reactions during wine ageing, for instance they are responsible for the formation of furfuryl alcohol (Spillman, Pollnitz, Liacopoulos, Pardon, & Sefton, 1998), of 2-furanmethanethiol (Tominaga, Blanchard, Darriet, & Dubourdieu, 2000) or of brown adducts with (+)-catechin (Nonier Bourden et al., 2008), with each wine evolving in a different way. Unlike furanic aldehydes, the furfuryl alcohol concentration was a lot smaller in Cabernet Sauvignon wines (726–629 µg/L) than in the other varieties (>12,000 µg/L). It is not extracted from wood, since its concentration in non-toasted and toasted oak woods is of only a few µg/L (Cadahía et al., 2003). Its origin in wine is the biological reduction of furfural, even though alcoholic and malolactic fermentation are completed prior to wine maturation in oak barrels (Ferreira, Jarauta, & Cacho, 2006; Spillman et al., 1998), so its formation will depend on factors

affecting enzymatic activity, such as oenological parameters or microbiological residual activity, which seem to be correlated with varietal and cultural grape factors (Aznar, López, Cacho, & Ferreira, 2003; Fernández de Simón et al., 2008; Ortega-Heras, González-Sanjosé, & González-Huerta 2007). Our results point to the theory that varietal factors or varying conditions during the production of Tempranillo and Merlot wines were more favourable to furfuryl alcohol formation than those of Cabernet Sauvignon. Most oenological parameters (Table 1) were similar in the three initial wines except the total SO₂ levels, so the low levels of furfuryl alcohol in Cabernet Sauvignon wines could be explained the lower levels of microbiological residual activity, possibly related to the SO₂ levels in this wine in comparison with the other varieties.

The concentrations of the furanic compounds in the three wines studied never reached their threshold levels (20–45 mg/L, Chatonnet, Dubourdieu, & Boidron, 1992), so their direct sensory contribution to the wine aroma may be null. However, they have been shown to reinforce the aromatic properties of other aromatic compounds such as methyl- γ -octalactones (Díaz-Plaza et al., 2002).

The *cis*- and *trans*-isomers of β -methyl- γ -octalactone were the only compounds that were not detected in the initial wines (Table 3). Their presence in wines is the result of their extraction from oak wood, obtained at the end of ageing levels as a function of wood type (Fernández de Simón, Cadahía, et al., 2003; Spillman, Sefton, & Gawel, 2004). The most important differential characteristic among the wines studied was the high levels of the *cis*-isomer shown in wines from Navarra oak (214–322 µg/L) in relation to those from French oak (142–154 µg/L), with both cases being over the threshold levels (54 µg/L in red wine, Brown, Sefton, Taylor, & Elsey, 2006). The *Q. petraea* oak wood from Navarra was especially rich in *cis*- β -methyl- γ -octalactone, showing levels around 55.9 ± 36.1 µg/g, after seasoning for two years (Cadahía et al., 2007), which are even comparable to those of American oak wood. The concentration of the *trans*-isomer was always smaller than that of the *cis*-isomer, under its threshold level (370 µg/L, Brown et al., 2006), and without significant differences among wine

varieties. On the other hand, a different extraction capacity of the *cis*- β -methyl- γ -octalactone from the same wood among the varieties was observed, which agreed with previous results (Fernández de Simón et al., 2008; Garde-Cerdán, Torrea-Goñi, & Ancín-Azpilicueta, 2004; Ortega-Heras et al., 2007). In the wines aged in Spanish oak wood from Navarra, the Merlot variety showed levels of *cis*-isomer significantly higher than Tempranillo and Cabernet Sauvignon (Table 3). These differences could be generated by differences in the characteristics of the wines (Table 1) which affect their extraction capacities, and also their evolution mechanisms during the ageing involving these compounds (Garde-Cerdán et al., 2008). The *cis*-lactone can be generated both during ageing in barrel as bottled and from precursors that are also extracted from the oak wood (Pollnitz, Pardon, & Sefton, 2000). Several natural oak lactone precursors, that can release oak lactone, have been identified in American and French oak wood extracts, consisting of ring-opened *cis*-oak lactone gallate, *cis*- and *trans*-oak lactone glucosides, and *cis*-oak lactone galloylglucoside (Hayasaka, Wilkinson, Else, Raunkjaer, & Sefton, 2007; Masson, Baumes, Guernevé, & Puech, 2000).

The ratio of *cis/trans*-isomers has been used by many authors as a marker to distinguish wines aged in different woods, showing values around two for French wood and higher than five for American wood (Chatonnet, & Dubordieu, 1998; Díaz-Plaza et al., 2002; Fernández de Simón, Cadahía, et al., 2003; Waterhouse & Towey, 1994). The wines aged in Spanish and French oaks (Table 3)

showed values higher than five, given in general, for American woods. The exceptions were Merlot and Cabernet Sauvignon wines aged in French oak. The results we acquired in these cases agreed with those of other authors that indicated difficulty in establishing a critical value in order to discriminate wines from different kinds of wood, due to the high variability shown even in wines aged in the same wood type. Also, the length of contact between wine and wood induces important changes in this ratio and the wines show different extraction capacities with regards to each isomer (Ortega-Heras et al., 2007). On the other hand, a clear difference between the *cis*- and *trans*-oak lactone formation rates from the corresponding precursors, ring-opened analogues, in wines has been shown, and they are strongly influenced by the pH of the medium (Wilkinson, Else, Prager, Pollnitz, & Sefton, 2004). The high values of the *cis/trans*-isomer ratio shown for the *Q. petraea* wood from Navarra (8.95 ± 7.29 , after seasoning for two years, Cadahía et al., 2007) were reflected in the Merlot and Cabernet Sauvignon wines, with *cis/trans* ratios of 8.9, higher than the values 4.5 and 2.9 shown by the same wines aged in French oak wood (Table 3). However, the values obtained in Tempranillo wines from Spanish and French oak wood showed similar results (*cis/trans* ratios 5.7 and 6.2, respectively). It is evident that wine characteristics (Table 1) related to the grape variety and cultivation conditions play an important role, influencing the extraction capacity of *cis*- and *trans*-lactones and their corresponding precursors, as well as their mechanism of formation and degradation in wine. Also, the

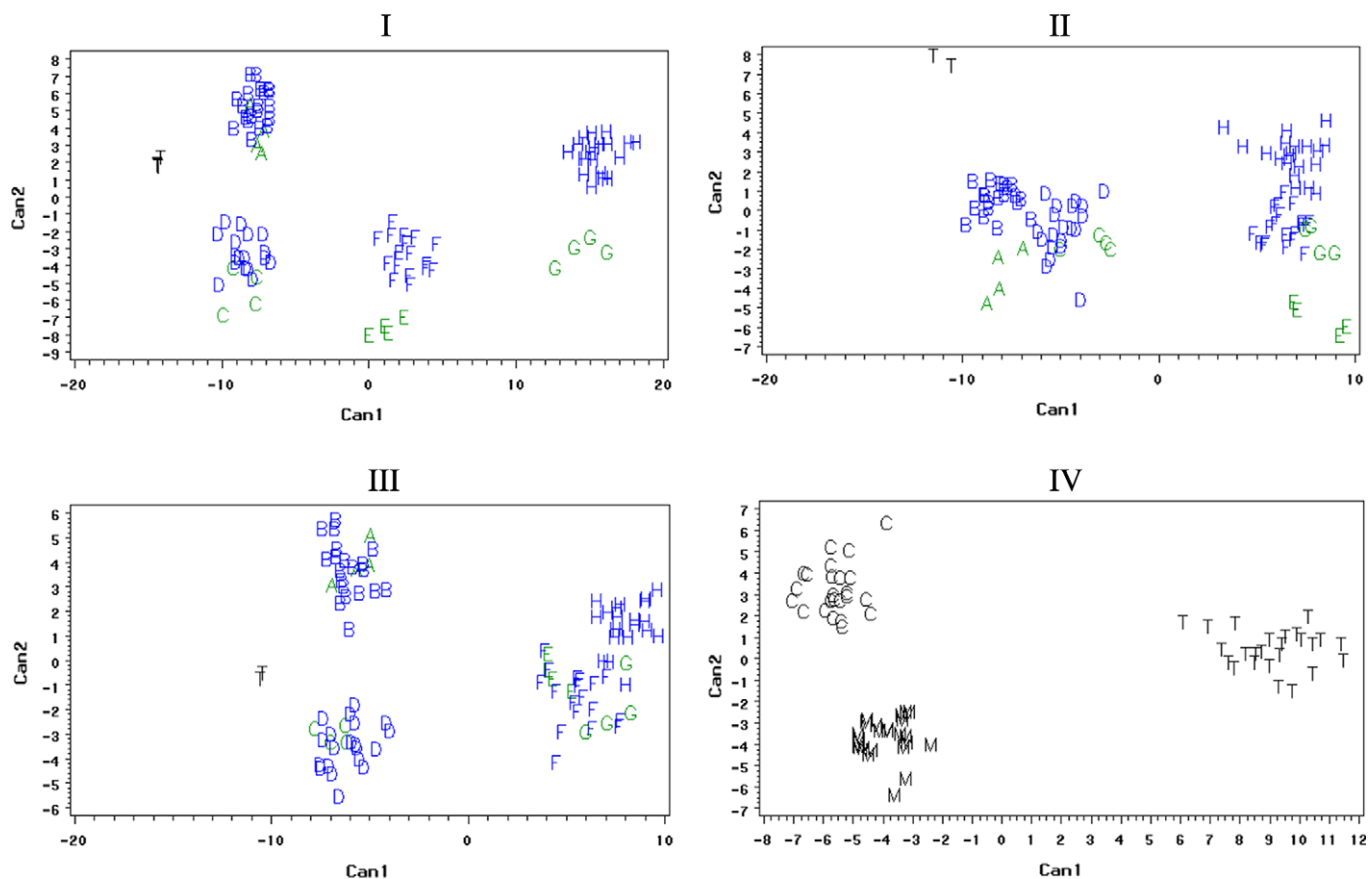


Fig. 3. Canonical discriminant analysis of the evolution of volatile compounds in the red single-variety wines, cv. Tempranillo (I), Merlot (II) and Cabernet Sauvignon (III), during ageing in Spanish (*Q. petraea* from Navarra) and French (*Q. petraea* from central region) oak woods. T = initial wines, three months (A = Navarra, B = France), six months (C = Navarra, D = France) nine months (E = Navarra, F = France) and 12 months of ageing (G = Navarra, H = France). Graphic IV: canonical discriminant analysis for the three wines, cv. Tempranillo (T), Merlot (M) and Cabernet Sauvignon (C) at 12 months of ageing in the different oak woods. I (total variance: 74% and 11%, canonical correlation: 0.996 and 0.972 and eigenvalues: 113.7 and 17.2, for can1 and can2, respectively). II (total variance: 75% and 8%, canonical correlation: 0.991 and 0.922 and eigenvalues: 52.2 and 5.6, for can1 and can2, respectively). III (total variance: 74% and 13%, canonical correlation: 0.990 and 0.944 and eigenvalues: 48.0 and 8.2, for can1 and can2, respectively). IV (total variance: 85% and 16%, canonical correlation: 0.989 and 0.945 and eigenvalues: 45.7 and 8.4, for can1 and can2, respectively). The total canonical structure coefficients of can1 and can2 for each graphic are included in Table 5.

wood processing in cooperage (seasoning and toasting) can influence the levels and the ratio of the two isomers (Cadahía et al., 2003). In fact, wines from other Spanish Origin Denominations aged in the same oak wood from Navarra, but seasoned and toasted in different conditions (different cooperage), showed levels and ratios of *cis*- and *trans*-lactones different to those obtained in this work, even though they were also more similar to those given by the American woods (Fernández de Simón et al., 2008).

Once again, the levels of volatile phenols such as guaiacol, eugenol, *t*-isoeugenol; syringol and derivatives showed a direct relationship with the wood and wine kind, although in this case the effect of wood was higher than that of grape variety (Table 3). In general, the wines aged in French oak wood were richer than those aged in Spanish wood from Navarra, with some exceptions in Tempranillo wines, which reached levels of guaiacol, 4-methylguaiacol, isoeugenol, syringol and 4-methylsyringol higher in those from Navarra wood. The concentration of volatile phenols in Spanish toasted oak wood from Navarra (1.54 ± 0.70 µg/g, data not published) was in general lower than that showed by the French woods (0–4 µg/g, Cadahía et al., 2003; Chatonnet, Cutzach, Pons, & Dubordieu, 1999), which is in agreement with the levels obtained in the studied wines. Although the concentration in wines of eugenol and guaiacol, which are the most important phenols due to their aromatic properties, were always smaller than their sensory thresholds (500 µg/L for eugenol and 75 µg/L for guaiacol, Boidron, Chatonnet, & Pons, 1988), additive and synergistic effects can take place in the wine, inducing sensory effects. Similar differences in the concentrations of eugenol and guaiacol were described in studies made with other Spanish single-variety wines aged in American wood (Garde-Cerdán et al., 2008; Ortega-Heras et al., 2007) and American, French and Spanish oak woods (Fernández de Simón et al., 2008). Other phenols like phenol, and *o*-, *p*- and *m*-cresol, did not show outstanding differences in the wines with relation to the wood kind of barrels.

The same comment can be made about maltol (Table 3), whose concentrations did not show any statistically significant differences between wines from Spanish and French oak woods. In addition,

this compound has limited sensory importance due to its high threshold in wine (5000 µg/L, Cutzach, Chatonnet, Henry, Pons, & Dubordieu, 1998) which in any case has been passed.

Referring to the group of phenolic aldehydes (vanillin and syringaldehyde) and related compounds (acetovanillone, propiovanillone, butirovanillone, acetosyringone, and propiosyringone), if we compare their levels in each wine variety, no differences have been obtained among wines from different woods, with the exception of methyl vanillyl ether in Merlot wines (Table 3). Some compounds showed concentrations higher in wine from French oak wood, but the high variability obtained for each kind of wood resulted in these differences being insignificant. Looking at vanillin, their levels in wines were similar to the concentrations described for French and Spanish toasted oak woods (Cadahía et al., 2003). The low levels obtained when compared with other studies (Fernández de Simón et al., 2008) could be due to differences in the compound concentration in the barrel toasted wood because of the toasting done at each cooperage, or to wine characteristics such as grape variety, alcoholic degree, pH, redox potential and turbidity that can determine a wine's capacity for extraction from wood and its evolution during wine ageing. In fact, the vanillin can be transformed into vanillic acid and its ethyl ester or, by reduction, to vanillin alcohol (Chatonnet et al., 1992; Spillman et al., 1997). The levels of syringaldehyde were always smaller in wines from Navarra oak wood than ones from French wood, especially in Merlot (3.38 and 23.0 µg/L, respectively) and Cabernet Sauvignon (5.4 and 23.3 µg/L). None of the wines showed concentrations above their sensory thresholds, 320 µg/L for vanillin (Boidron et al., 1988) and 50 mg/L for syringaldehyde (Singleton, 1995) in red wine, so possibly they have little influence in the sensory characteristics of obtained wines.

Taking into account the quantitative evaluation of overall volatiles in wines at the end of ageing (Table 3) and also at three, six and nine months (data not published), we have carried out a canonical discriminant analysis for each single-variety wine. The graphic representations and total canonical structure coefficients for the discriminant variables are shown in Fig. 3 (graphics I, II

Table 5

Total canonical structure coefficients of the main discriminant variables in can1 and can2, obtained for volatile compounds (Fig. 3). The non-shown coefficients showed values smaller than 0.500 in can1 and 0.400 in can2.

Graphic	I		II		III		IV	
	Can1	Can2	Can1	Can2	Can1	Can2	Can1	Can2
Furfural			-0.697		-0.503			0.698
5-Methylfurfural							-0.506	0.434
5-Hydroxymethylfurfural			-0.518					0.557
Furfuryl alcohol					0.563	-0.457		
<i>trans</i> -β-Methyl-γ-octalactone		-0.527						
<i>cis</i> -β-Methyl-γ-Octalactone	0.624		0.587		0.530			
Maltol	0.646							
Guaiacol	0.895		0.596		0.552			
4-Methylguaiacol	0.635			0.451	0.521			
Phenol	0.851		0.913				0.598	0.426
<i>o</i> -Cresol	0.740			0.593		0.487	0.787	
<i>p</i> -Cresol	0.895			0.449			0.532	
<i>m</i> -Cresol							0.578	
Eugenol	0.837		0.660		0.601			
Isoeugenol	0.759			0.425				
Syringol	0.715					0.409		
4-Methylsyringol				0.447				
4-Allylsyringol	0.892		0.551		0.514		0.811	
Acetovanillone	0.867			0.460			0.638	-0.400
Propiovanillone	0.953		0.508		0.589		0.665	
Butirovanillone	0.946		0.661		0.793		0.758	
Methyl vanillyl ether	0.837			0.427		0.619	0.918	
Ethyl vanillyl ether	0.882		0.731		0.859		0.582	
Syringaldehyde		0.596						
Acetosyringone	0.632				0.507			
Propiosyringone	0.925		0.613		0.748		0.605	

and III), and Table 5, respectively. Other canonical analysis was carried out, comparing the composition of the three single-variety wines at the end of ageing (graphics IV, in Fig. 3). Similarly to that observed for the polyphenolic composition, the volatile compound evolution of wines (graphics I, II and III in Fig. 3) corresponded more directly with the stay time in barrel and grape variety than with wood kind. The three wines showed similar behaviour, and only small differences between ones from Spanish and French oaks were observed, in Tempranillo (I) and Merlot (II) varieties, which remained during the entire ageing process. The patterns of distribution for the wines in the canonical plane (can1–can2) show groups according to the stay time in barrel, initial wines (T), wines aged for three months (A, B), for six months (C, D), for nine months (E, F), and for 12 months (G, H). As well as this, within each group we can distinguish the wines according to the oak wood kind of barrel. For example, Tempranillo wines aged for 9 and 12 months in Spanish oak (E and G, graphic I in Fig. 3) can be differentiated from the same wines aged in French oak (G and H), although the statistical variations are very small. The coefficients of total canonical structure (Table 5) show that the most volatile compounds contribute as discriminant variables to the canonical function one which account for 75% of the total variance. Many discriminant variables are compounds with small sensory incidence in wines because of their high sensory threshold, but others such as *cis*- β -methyl- γ -octalactone have a high aromatic potential, which can increase their contribution to the sensory differentiation of wines, although they show canonical coefficients similar to or smaller than the first ones (Table 5).

The statistical analysis, which looks at the volatile composition of the three wines at the end of ageing (IV, in Fig. 3) proves different extraction capacities for each variety for the volatile compounds obtained from wood. We have collected different wines based mainly on the grape variety, although the statistical differences among them were smaller than in the statistical analysis of the phenolic compounds. This confirms the results of other studies in which the volatile and polyphenolic composition extracted from wood by mono-varietal wines aged in identical conditions were different (Fernández de Simón et al., 2008; Monagas et al., 2005; Pérez-Magariño & González-Sanjosé, 2004; Revilla et al., 2005; Sun & Spranger, 2005).

4. Conclusions

In light of this study, we can conclude that wines aged in Spanish oak from Navarra evolved in the same way as those aged in French oak from the Central region obtained from five French and Spanish cooperages of recognised quality. In general, at the end of the ageing process the differences in their chemical and chromatic characteristics were very small, although each wood kind added special characteristics to the wine that depended on the grape variety. Looking at special characteristics of wines aged in Spanish oak wood, we can underline the higher levels of *cis*- β -methyl- γ -octalactone in comparison with the same wines aged in French oak wood. So, the Spanish oak wood (*Q. petraea*) from Navarra has an oenological quality similar to the French oak wood (*Q. petraea*) from the Central region commonly marketed by French and Spanish cooperages.

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