

# EVOLUTION OF PHENOLIC COMPOSITION AND SENSORY PROPERTIES IN RED WINE AGED IN CONTACT WITH PORTUGUESE AND FRENCH OAK WOOD CHIPS

## ÉVOLUTION DE LA COMPOSITION PHÉNOLIQUE ET DES PROPRIÉTÉS SENSORIELLES DE VINS ROUGES EN CONTACT AVEC DES COPEAUX DE CHÊNES FRANÇAIS ET PORTUGAIS

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**Summary:** A red wine was matured in contact with 4 g/L of oak wood chips from Portuguese (*Quercus pyrenaica* Willd.), French (*Quercus petraea* L.) and a mixture (50:50) of this two oak wood species, during 13 weeks, in order to evaluate the effects of these different oak wood chip species (specially Portuguese oak wood) on the phenolic composition evolution of the wine and in their sensory properties.

In general, for the phenolic compounds studied, it wasn't possible to detect remarkable differences between the control wine (aged without oak wood chips) and the wines aged in contact with the two oak wood chips species used. However, for non-flavonoid phenols, the presence of oak wood chips contributed to an increase of these compounds in red wines. The influence of oak wood chips in anthocyanins evolution were similar for all wines, except for malvidin-3-glucoside, which decrease was more evident for the wine aged in contact with oak wood chips. The oak wood chips species and the chips concentration used in this study, didn't affect the proanthocyanidin contents in the wines during the time considered.

Sensory results showed that, the wines aged in contact with Portuguese and French oak wood chips and the mixture of this two oak wood species, differed significantly from the control wine in several sensorial characteristics. The wines aged in contact with wood chips showed a higher punctuation values for intensity, toasted, wood and vanillin aroma, taste intensity and global appreciation. This positive effect was more evident for wines aged with Portuguese oak wood chips. Probably this results, suggest that the Portuguese oak wood samples species (*Quercus pyrenaica* Willd.) used could be considered suitable for barrel production because it has a positively effect in sensorial red wine attributes. Thus with this study we tried to contribute for understand the Portuguese oak wood role in red wine characteristics.

**Résumé:** Un vin rouge a été élevé pendant 13 semaines en présence de 4 g/L de copeaux de bois de chênes portugais (*Quercus pyrenaica* Willd.) et français (*Quercus petraea* L.) ainsi qu'un mélange des deux espèces, dans le but d'évaluer les effets de ces deux types de bois de chêne (en particulier le portugais), sur la composition phénolique du vin, ainsi que sur ses propriétés sensorielles.

En général, pour les composés phénoliques étudiés, il n'a pas été possible de détecter de différences remarquables entre le vin témoin, élevé sans copeaux de bois de chêne, et les vins élevés, avec les deux espèces de copeaux de chêne. Toutefois, la présence de copeaux de bois de chêne a contribué pour l'enrichissement des vins en composés phénoliques non-flavanoïdes. L'influence des copeaux de chêne sur l'évolution des anthocyanines a été très semblable dans tous les vins, sauf pour la malvidin 3-glucoside, pour laquelle, la baisse de concentration a été plus évidente pour le vin élevé en présence de copeaux. La concentration en proanthocyanidines dans les vins n'a pas été affectée par les différentes espèces de copeaux de bois de chêne.

Du point de vue sensoriel, les vins élevés en présence de copeaux de chênes portugais et français ou du mélange des deux bois, présentent des différences significatives par rapport au vin témoin. Les vins élevés avec les copeaux de chêne ont été bien évalués pour les caractères boisé, vanillé, grillé, l'intensité du goût et l'appréciation globale. Cet effet positif a été plus évident dans le cas des vins rouges élevés en présence de copeaux de chêne portugais. Ces résultats suggèrent que l'espèce de bois de chêne portugais (*Quercus pyrenaica* Willd.) pourrait être utilisée en tonnellerie, car elle a montré des effets assez positifs, en particulier lors de l'analyse sensorielle.

**Mots clés :** vin rouges, copeaux de bois de chêne, composés phénoliques, analyse sensorielle

**Key words:** red wines, oak wood chips, phenolics compounds, sensory analysis

## INTRODUCTION

The production of barrique wines is a current way of further processing of natural red and white wines. The maturing of the wines in toasted oak-barrels changes them and emphasises some of their characteristics. The phenolic and sensory characteristics of red wine ageing in oak wood barrels depends on the quality of the basic wine (LASZLAVIK *et al.*, 1995), the species and origin of oak wood (JINDRA and GALLENDER, 1987; CHATONNET *et al.*, 1997; GONZALEZ-MENDOZA and POMAR, 2001; GONZÁLEZ-SAN JOSÉ and REVILLA, 2001), the level of toasting and charring of the barrel wood (REAZIN, 1981; CHATONNET *et al.*, 1989), the dissolved oxygen in wines and barrel volume (MOUTOUNET *et al.*, 1998; PEREZ-PRIETO *et al.*, 2002) and finally the ageing time (KADIM and MANNHEIM, 1999).

Many constituents can be extracted from staves during ageing in barrels: ellagitannins (PENG *et al.*, 1991; VIRIOT *et al.*, 1993); and tannins, gallic, ferulic, vanillic and ellagic acids, vanillin, coumarins and a great number of volatile compounds (MOUTOUNET *et al.*, 1989; CHATONNET *et al.*, 1990; CHATONNET *et al.*, 1997; PÉREZ-COELLO *et al.*, 2000; ESCALONA *et al.*, 2002). Derivatives of benzoic and cinnamic acids are the main phenolic compounds that give the typical character to the wines ageing in barrels. Further compounds found in the wines are 2-furaldehyde and its derivatives that are products of sugars degradation during toasting of the wood (CHATONNET *et al.*, 1989; CUTZACH *et al.*, 1997; GOLDBERG *et al.*, 1999). On the other hand, the presences of derivatives of 2-furaldehyde and vanillin have a significant effect in wine aroma too (SINGLETON, 1995; MARTINEZ *et al.*, 1996).

Phenolic compounds contribute directly or indirectly to colour, astringency, bitterness and aroma of barrique wines. Determination of this group of compounds is important since they can characterise variations in wine type's styles, in winemaking and in maturation processes. On the other hand, more studies has been made about these compounds, because their antioxidant properties (SHAHIDI and NACZK, 1995; SHRIKHANDE, 2000; HOLLMAN, 2001).

During the maturation of red wine in oaks casks or using oak wood chips, and mainly in toasted barrels or chips, several changes in red wine phenolic compounds occur. An increase in the content of phenols extracted from the wood was already reported. The amount of phenolics extracted into wine depends on ageing time, oak type, size and the possibility of the barrel having been used (ROUS and ALDERSON, 1983; JINDRA and GALLENDER, 1987; LASZLAVIK *et al.*, 1995; PISARNITSKII, 1995; GONZÁLEZ-SAN JOSÉ and

REVILLA, 2001; CHATONNET *et al.*, 1997; HO *et al.*, 2001).

The chemical differences between most oak wood species used for ageing wines (*Quercus robur*, *Petraea* and *alba*) and their influence on wine quality were put in evidence in previously studies. Meanwhile, the influence of Portuguese oak wood (*Quercus pyrenaica* Willd.) in phenolic and sensorial properties evolution of red wine is not usual in the research works. Several studies (BELCHIOR *et al.*, 1998; CANAS *et al.*, 2000 a; JORDÃO *et al.*, 2005) demonstrated that this species contains large quantities of extractable substances (volatile compounds and ellagitannins) and for some of them the content is similar to quantified in French oak species. *Quercus pyrenaica* species, occupies a part of the north Portuguese forests, but usually is not used expressively in wine maturation. In Portugal, this oak wood species is traditionally used for brandies ageing (CANAS *et al.*, 2000 b).

Thus, the aim of our study was evaluate the time-dependent changes in the phenolic compounds and sensory properties in one red wine matured in contact with Portuguese (*Quercus pyrenaica* Willd.), French (*Quercus petraea* L.) and a mixture containing this two oak wood chip species (50:50). On the other hand, all parameters studied in wines aged with the different oak wood chips were compared with the values quantified in the standard wine (aged without oak wood chips).

## MATERIAL AND METHODS

### I - WINE

The wine used in this study was a 2002 vintage mixture of 5 different red grape varieties (Trincadeira Preta, Castelão, Cabernet Sauvignon, Touriga Nacional and Syrah) each in 20 %, from a vineyard in Instituto Superior de Agronomia, Lisbon. The wine was made by a classical wine making technology, with a maceration time of 10 days. The wine main characteristics were the following: ethanol 10.5 % (v/v), pH 3.43, total SO<sub>2</sub> 68 mg/L, titratable acidity 4.95 g/L and volatile acidity 0.68 g/L.

### II - OAK WOOD CHIP SAMPLES

In Portugal several forest regions with oak trees from *Quercus pyreniaca* Willd. species with a potential use by cooperage companies it is possible to be found. Nevertheless, in this study, we used only oak wood chip samples from Gerês forest (one of the most important Portuguese forest where *Quercus pyrenaica* Willd. is present). Thus, Portuguese oak wood samples from Gerês region and French (*Quercus petraea* L.) from Allier region were used in this work. All oak wood chips samples used (particles with a size less than 2 mm) presented a medium

toasting (20 min at temperature on the wood surface 160-170 °C) and a grain of 3.0 to 3.5 mm.

### III - EXPERIMENTAL CONDITIONS

A total of four assays (in duplicate) were made in this study, using 10 L of red wine in each assay: red wine + 4 g/L of oak wood chips from *Q. pyrenaica*; red wine + 4 g/L of oak wood chips from *Q. petraea*; red wine + mixture of *Q. pyrenaica* and *Q. petraea* (50:50) and control wine (without oak wood chips). Thus, after the malolactic fermentation, the wines were aged in contact with oak wood chips, in the dark for 13 weeks at a temperature ranged from 16 to 18 °C.

### IV - CHEMICAL ANALYSIS

The wine used in our study was analysed for pH, titratable and volatile acidity, alcohol level, SO<sub>2</sub> level, total phenols index and total anthocyanins, colour density and hue, using the analytical methods recommended by the OIV (1990). During red wine ageing, total phenols and flavonoid phenols determinations, were analysed as described by SOMERS and EVANS (1977). For non-flavonoid phenols, a method described by SINGLETON *et al.* (1971) was used.

For fractionation of wine proanthocyanidins according to their polymerisation degree: catechins (monomers), oligomeric and polymeric fraction, a method described by SUN *et al.* (1998 a) was used. For each fraction obtained previously, the quantification of flavanols was performed by the modified vanillin assay presented by SUN *et al.* (1998 b). All determinations were done in duplicate.

### V - CHROMATOGRAPHIC ANALYSIS

For analysis of individual anthocyanins from wine the apparatus used was a Perkin Elmer system (Norwalk, USA), equipped with a 410-LC pump, a solvent programmer Model 420, and a manual injector (Rheodyne 7125-A) fitted with a 20 °L loop. The column (250 x 4.6 mm, particle size 5 µm) was a C18 LiChrospher® 100 (Merck, Darmsdadt, Germany) protected by a guard column of the same material. The solvents were (A) 40 % formic acid, (B) pure acetonitrile and (C) bidistilled water. The individual anthocyanins were analyzed by HPLC using the method described by DALLAS and LAUREANO, (1994). Thus, initial conditions were 25 % A, 10 % B, and 65 % C, followed by a linear gradient from 10 to 30% B, and 65 to 45 % C for 40 min, with a flow rate of 1 mL/min. The detection was made at 520 nm using a Konik detector coupled to a Konichrom data treatment station (Konik Instruments, Barcelone, Spain). All analyses were made in duplicate.

### VI - SENSORY EVALUATION

After 13 weeks, two replicates of each wine sample were reunified and stored 24 hours at room temperature before analysis. The sensory analysis was performed at 20-22 °C in a sensorial analyses room with individual cabins for each expert. In the session about 30 mL samples were presented in coded and a 150 mL sensorial wine glass were used. Eleven expert judges of the Ferreira Lapa Laboratory evaluated the several wines. The wines were evaluated using different descriptors for colour (intensity and hue), aroma (intensity, fruited, vanillin, wood, and toasted), taste (intensity, body, astringency, equilibrium and persistence) and global appreciation. The judges were asked to evaluate the samples on a 1 - 7 point's quality scale (1 less and 7 more intense) for each characteristic, according to their sensory knowledge, training and experience.

### VII - STATISTICAL ANALYSES

In order to study the influence of the two oak wood chips species and the mixture of the oak species studied in sensorial evaluation of the wines, an analysis of variance and comparison of treatment means (ANOVA, one-way) was performed using SPSS software program version 11.0 (SPSS Inc. Headquarters, Chicago, Illinois, USA).

## RESULTS AND DISCUSSION

### I - EFFECT OF OAK WOOD CHIPS IN WINE PHENOLIC COMPOUNDS EVOLUTION

The results of phenolic composition obtained in the wine used in this study are shown in table I. One of the important phenolic family present in wines, are the proanthocyanidins (condensed tannins). This compounds,

**Table I - General phenolic composition of the standard red wine using in the study.**

**Composition phénolique du vin rouge utilisé dans cette étude**

Phenolic parameters	Values
Total phenols index (280 nm, abs. units)	39.8
Total phenols (mg/L, gallic acid equiv.)	3383.2
Non-flavonoid phenols (mg/L, gallic acid equiv.)	305.3
Flavonoid phenols (mg/L, gallic acid equiv.)	3078.5
Total anthocyanins (mg/L, malvidin-3-glucoside equiv.)	275.7
Colour density (abs. units)	6.84
Colour hue (abs. units)	0.64
Degree of ionisation of anthocyanins (%)	19.1
Total pigments (abs. units)	15.66
Polymeric pigments (abs. units)	1.66
Degree of polymerisation (%)	10.6

contribute for the astringency, bitterness and colour of the wines too.

The monomeric forms ((+)-catechin and (-)-epicatechin) and the principal dimeric and trimeric proanthocyanidins quantified in the wine used, are presented in table II.

Table III shows the total phenols, flavonoid phenols and non-flavonoid phenols evolution during red wine

**Table II - Monomeric and some oligomeric proanthocyanidins (flavanols, medium values expressed in mg/L) of the standard red wine using in the study**  
**Monomères et proanthocyanidines oligomériques du vin témoin utilisé dans cette étude (flavanols, valeurs moyennes exprimées en mg/L)**

Flavanols	Values
<b>Monomers</b>	
(+)-Catechin	10.5
(-)-Epicatechin	19.9
<b>Dimers</b>	
Proanthocyanidin B1	25.8
Proanthocyanidin B2	25.3
Proanthocyanidin B3	1.8
Proanthocyanidin B4	3.9
<b>Trimers</b>	
Proanthocyanidin Trimer 2	3.9
Proanthocyanidin Trimer C1	1.8
<b>Dimer Gallates</b>	
Proanthocyanidin B1-3-0-gallate	1.4
Proanthocyanidin B2-3-0-gallate	0.4
Proanthocyanidin B2-3'-0-gallate	3.3

ageing in contact with different oak wood chips species and the control wine. In general, for all wines, the total and flavonoid phenols content remained nearly constant during the 13 storage weeks studied. This evolution could be explained due to a complex equilibrium between phenolic substances extracted from wood and colouring material precipitated in red wine during the storage time. As well as possible adsorption on wood walls phenolic wines compounds. However, in standard wine same variation in phenols values was observed. Similar evolution was reported by GÓMEZ and GONZALEZ (1995). However, other studies showed an increase in total phenols during the ageing period in contact with oak wood as a consequence of phenols transfer from wood to the wine (JINDRA and GALLENDER, 1987).

The effect on the wine of oak wood chips contact and the different oak wood species used on total and flavonoid phenols were not evident. Similar observation was reported by POMAR and GONZALEZ-MENDOZA (2001). Probably the storage time or the oak wood chip concentration used was not sufficient for us to see remarkable difference between the wines aged in contact of different oak chip species used in this study.

Quantitative changes in non-flavonoid phenols compounds in all wine were followed over the same period (table III). It is seen that non-flavonoid phenols compounds remained nearly constant during the first 6 storage weeks followed by a slightly increase of the values. The values found were higher in the wines aged in contact with oak wood chips than in standard wine. However among the wines aged with Portuguese, French and mixture of this two oak wood species, no differences in the concentrations of non-flavonoid phenols content occurred. Meanwhile at the end of ageing, the values were 319.5; 402.0; 344.9 and 469.9 mg/l for standard wine (Sw), wines in contact with French oak chips (Fw),

**Table III - Evolution of total phenols, flavonoid phenols and non-flavonoid phenols (mean values expressed in mg/L of gallic acid equivalents) in red wine aged in contact with different oak wood chips species (4 g/L) and the standard wine**  
**Évolution des phénols totaux, des phénols flavanoïdes et non flavanoïdes (moyennes des valeurs exprimées en mg/l d'équivalents d'acide gallique) dans le vin rouge élevé avec différents copeaux (4 g/l) et le vin témoin**

Wines	0 week			3 weeks			6 weeks			10 weeks			13 weeks		
	Total Phenols	Flavonoid Phenols	Non Flavonoid Phenols	Total Phenols	Flavonoid Phenols	Non Flavonoid Phenols	Total Phenols	Flavonoid Phenols	Non Flavonoid Phenols	Total Phenols	Flavonoid Phenols	Non Flavonoid Phenols	Total Phenols	Flavonoid Phenols	Non Flavonoid Phenols
Sw	3383.2	3078.5	305.3	3163.4	2895.6	267.8	3055.6	2833.1	222.5	3402.1	3019.0	383.1	3265.1	2945.6	319.5
Fw	3383.6	3078.6	305.0	3173.3	2842.3	331.0	3042.4	2666.7	375.7	3427.9	2742.5	685.4	3080.3	2678.3	402.0
Pw	3383.1	3078.5	305.6	3241.8	2840.7	401.1	3420.1	3063.1	357.0	3395.6	2872.1	523.5	3530.7	3185.8	344.9
PFw	3383.0	3078.8	304.2	3247.1	2910.1	337.0	3197.7	2919.4	278.4	3382.3	2866.6	515.7	3195.0	2725.1	469.9

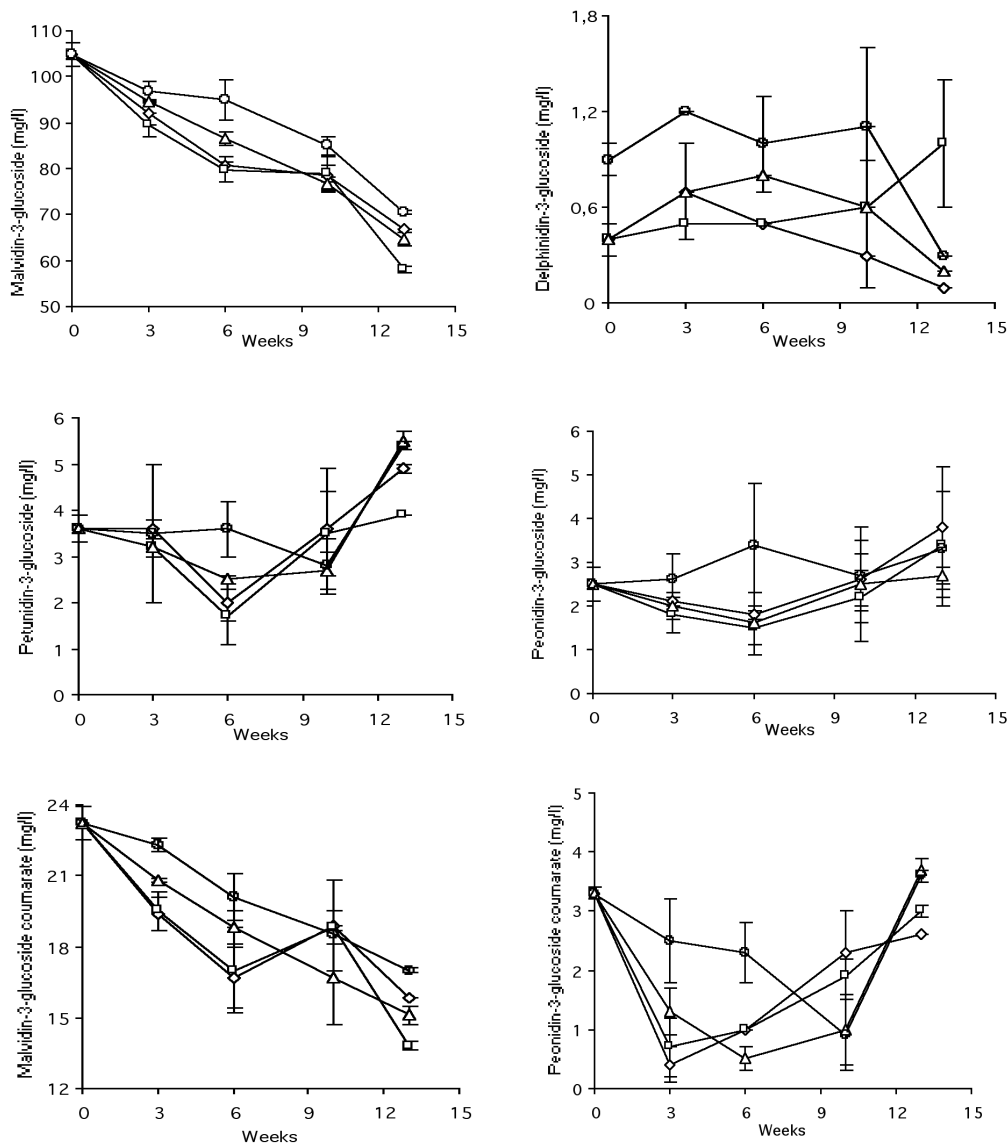
Sw: Standard wine; Fw: Wine aged in contact with French oak wood chips; Pw: Wine aged in contact with Portuguese oak wood chips; PFw: Wine aged in contact with a mixture (50:50) of Portuguese and French oak chips

Portuguese oak chips (Pw) and the mixture of these two oak wood chips species (PFw), respectively. These results are similar to those found by other authors using different oak wood species in wine ageing (ROUS and ALDERSON, 1983; JINDRA and GALLENDER, 1987; YOKOTSUKA *et al.*, 1994; MATEJÍCEK *et al.*, 2005).

The high values of non-flavonoids quantified in wines aged in contact with oak wood chips probably, corresponded to an extraction of some phenols compounds, namely, gallic acid, protocatechic, vanillic, caffeic, syringic and p-coumaric acids from oak wood chips to the wine. On the other hand, ellagitannins and ellagic acid will also

contribute for this non-flavonoid increase. These results emphasise the importance of several phenolic compounds in the quantitative non-flavonoid compounds extraction from the oak wood. Ellagitannins and ellagic acid, for example, have been shown to account for 10 % of the total phenolic content of wines or model wine treated with oak wood chips (QUINN and SINGLETON, 1985; FEUILLAT *et al.*, 1997).

The individual monomeric anthocyanins evolution studied during wine ageing are shown in figure 1. In general, an evident decrease of anthocyanin content was exhibited during the wine ageing (including in the standard



**Figure 1 - Changes in concentrations of different anthocyanins during red wine aged in contact with different oak wood chip species**

○ Standard wine; ◇ Wine aged in contact with French oak wood chips; □ Wine aged in contact with Portuguese oak wood chips; △ Wine aged in contact with a mixture (50:50) of Portuguese and French oak wood chips

**Changements dans la concentration des différentes anthocyanines durant l'élevage du vin rouge avec différents copeaux**

○ Vin témoin ; ◇ Vin élevé avec des copeaux de chêne français ; □ Vin élevé avec des copeaux de chêne portugais ; △ Vin élevé avec un mélange (50 :50) de copeaux de chênes portugais et français

wine). This decrease in anthocyanin content was probably due to anthocyanin condensation and polymerization reactions that occurred during wine ageing. On the other hand, a precipitation of these compounds will be another reason for the decrease observed. This result is consistent with previous works (NAGEL and WULF 1979; POMAR and MENDOZA 2001). PÉREZ-PRIETO *et al.* (2003), suggested that the decrease of these pigments in wines matured in oak wood is probably due to anthocyanin degradation reactions different from the condensation reactions that lead to wine colour stabilization. Meanwhile, other studies indicate that the anthocyanin potential decrease can be attributed to enzymatic or chemical degradation during wine aged in contact with oak wood (CASTELLARI *et al.*, 2001; MATEJÍČEK *et al.*, 2005).

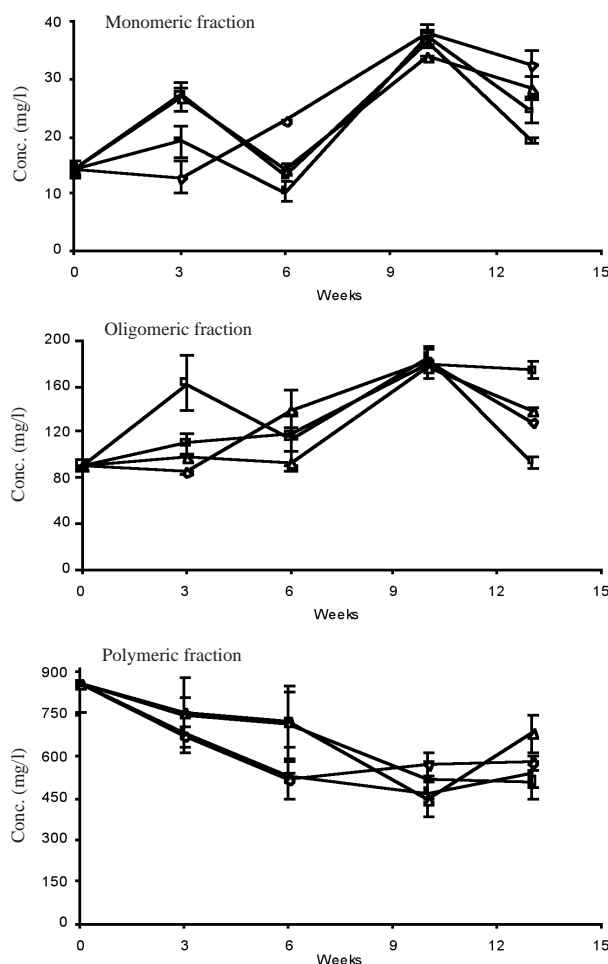
The decreases of anthocyanin values were more evident for malvidin-3-glucoside and malvidin-3-glucoside coumarate. However, for petunidin-3-glucoside and peonidin-3-glucoside a slightly increase of values occurred after 6 weeks of ageing time in all wines. After 13 ageing weeks the wines matured in contact with oak wood chips exhibited lower contents of individual anthocyanins (namely for malvidin-3-glucoside and malvidin-3-glucoside coumarate) than the standard wine (without contact with oak wood chips). Thus, after 13 weeks, for malvidin-3-glucoside, the values quantified were 58.2 and 70.4 mg/L, for wine aged in contact with Portuguese oak wood chips (Pw) and standard wine (Sw), respectively.

It's possible that some reactions between malvidin-3-glucoside and the phenol components extracted from oak wood chips are only visible for this individual anthocyanin, because in wine this pigment is always quantified in higher levels than the other individual anthocyanin forms. Thus, maybe only for high levels of the components involved in this kind of reactions that it will be possible to detect some variations in their values. Nevertheless, future studies should be necessary in order to understand the probably reactions that occur between malvidin-3-glucoside (and other individual anthocyanins) and oak wood components, specially using model wine solutions.

The results in figure 2 show the monomeric, oligomeric and polymeric fraction of proanthocyanidins evolution in all red wines studied. In general, for monomeric and oligomeric proanthocyanidins fraction, small fluctuation variations of the values were noted in all wines, but for polymeric fraction, a slightly decrease of the values occurred during the ageing period. In last storage weeks analysed a maximum at 10 weeks followed by a decrease of the values were been noted for these two proanthocyanidins fraction. On the other hand, a tendency for an

increase of the values in the last weeks has been noted, for the polymeric proanthocyanidins.

Between the several wines studied, there were no appreciable quantitative differences in proanthocyanidins fractions studied. Thus, it appears that the oak wood chips concentration (4 g/l) and the oak wood species used in our study, didn't affect significantly the proanthocyanidin fraction content and their evolution in the wines during the storage time considered.



**Figure 2 - Changes in concentrations of monomeric, oligomeric and polymeric fraction**

**of proanthocyanidins during red wine aged in contact with different oak wood chips species**

O Standard wine; ◇ Wine aged in contact with French oak wood chips; □ Wine aged in contact with Portuguese oak wood chips; △ Wine aged in contact with a mixture (50:50) of Portuguese and French oak wood chip. Conc.: concentration values.

**Changement de la concentration**

**des fractions monomérique, oligomérique et polymérique durant l'élevage du vin rouge avec différents copeaux**

O Vin témoin ; ◇ Vin élevé avec des copeaux de chêne français ; □ Vin élevé avec des copeaux de chêne portugais ; △ Vin élevé avec un mélange (50 :50) de copeaux de chênes portugais et français. Conc. : concentration

This work confirms the results found by other authors (CHATONNET *et al.*, 1997). For these authors, it wasn't possible to detect significant differences in oligomeric proanthocyanidins content in wines matured in different oak wood barrels species (*Quercus petraea* and *Quercus alba*). Other studies, using different oak wood barrels species, reported high values of polymerized phenolics, indicating a more rapid polymerization of monomeric phenols in wines matured in oak wood barrels than the wines matured in tanks (CASTELLARI *et al.*, 2001). For KADIM and MANNHEIM (1999), the favourable rate of surface/volume of the barrels may be considered the origin of the easier polymerisation of phenolics, because the contact between wood and wine and the oxygen dissolution in wine may be improved.

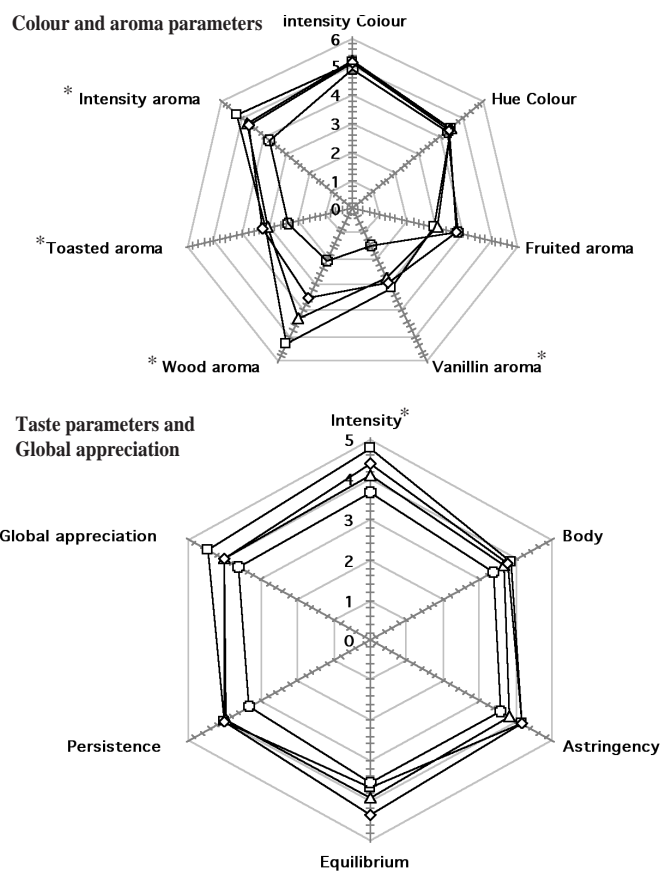
## II - EFFECT OF OAK WOOD CHIPS SPECIES ON WINE SENSORY CHARACTERISTICS

Although the chemical analysis (specially the phenolic composition) allowed us to reach some conclusions concerning the evolution of wines in contact in different oak wood chips species, we were very interested in the result of the sensory analysis. Previous studies showed significant difference in sensorial characteristics between wines matured in American oak versus French oak wood (JINDRA and GALLENDER, 1987; POMAR and GONZALEZ-MENDONZA, 2001; PÉREZ-PRIETO *et al.*, 2002; 2003). Thus, after 13 weeks of different oak wood chips species contact, a sensory analysis was made using several experts taster. For this analyse we used different descriptors (see material and methods), which we considered more appropriated to this kind of study.

The results of the sensory analysis for the wines ageing in contact with different oak wood chips species and the standard wine (without contact with oak wood chips) are shown in figure 3. This figure is a spider diagram of the mean scores of wine's attributes. In these diagrams, the centre of the figure represents low intensity of each attribute increasing to an intensity of seven at the perimeter. Our results showed that the different oak wood chip species did not cause significant differences in intensity and hue colour and fruited aroma between all wines (including standard wine). Results, for colour parameters are according to the values obtained in phenolic composition of the different wines. In general, for all phenolic parameters studied (figure 1, 2 and table III) it wasn't possible to detect great differences between the wines aged in contact with different oak wood chips species and the standard wine. On the other hand, comparing, intensity, toasted and vanillin aroma character, significant differences were found between the wines ageing in contact with oak wood chips and the standard wine. In that case the judges preferred the wines ageing in contact with oak

wood chips independently the oak wood chip species used.

For aroma descriptors, only the wood aroma descriptor produced significant differences in the judge's punctuation between the wines aged in contact with oak wood chip species. Thus, for wood aroma, the wine aged with Portuguese oak wood chips had the highest punctuation in the quality scale (5.3 mean points), followed by the wine aged with a mixture of Portuguese and French oak wood chips (4.3 mean points) and the wine aged with French oak wood chip (3.5 mean points). These differences probably could be explain as a result of more *trans* and *cis*- $\beta$ -methyl- $\gamma$ -octalactone present in wine aged in



**Figure 3 - Sensorial analysis results for the red wines aged in contact with different oak wood chip species and analysis of variance and comparison of treatment means (ANOVA)**

An asterisk means values with significant differences ( $p < 0.05$ ).  
 O Standard wine; ◇ Wine aged in contact with French oak wood chips; □ Wine aged in contact with Portuguese oak wood chips; △ Wine aged in contact with a mixture (50:50) of Portuguese and French oak wood chips

### Résultats de l'analyse sensorielle des vins rouges élevés avec différents copeaux de chêne et analyse de variance et comparaison des moyennes (ANOVA)

L'astérisque montre des valeurs avec des différences significatives ( $p < 0,05$ ) ;  
 O Vin témoin ; ◇ Vin élevé avec des copeaux de chêne français ; □ Vin élevé avec des copeaux de chêne portugais ; △ Vin élevé avec un mélange (50 :50) de copeaux de chênes portugais et français

contact with Portuguese oak wood chips than the values in wine aged with French oak wood chips. Previously studies (PÉREZ-PRIETO *et al.*, 2002) showed, that the concentration of oak lactones, and specially *cis*- $\beta$ -methyl- $\gamma$ -octalactone, had an important role in woody character of the wines.

Concerning the taste descriptors of the different wines after 13 ageing weeks, our results indicated (figure 3) that the two oak wood species used and the mixture of these species did not have influence for all taste descriptors (intensity, body, astringency, equilibrium and persistence) for the wines aged in contact with oak wood chips. However, between the wines aged in contact with oak wood chips and the standard wine some differences were found. Thus, taste intensity was more significant pronounced in oak wines (4.7 and 4.4 mean points for wine aged in contact with Portuguese and French oak wood chip species, respectively) than standard wine (3.6 mean points).

For global appreciation, the results show a slight preference for wines aged with oak wood chips (especially for Portuguese matured wines). However, no significant statistical difference in judges punctuation have been noted for wine global appreciation descriptor between the wines aged in contact with oak wood chip species and standard wine.

Finally, these results showed that, in general, the use of Portuguese oak chips had a more positive effect in wine sensory properties than the effect obtained for wines aged in contact with French oak wood species.

## CONCLUSIONS

In our work, we obtain more information about Portuguese oak wood species impact in oak wood wine maturation and consequently more information for the oenologists, when is necessary selecting oak wood species to produce matured wines. However, the economic factors must not be forgotten in oak wood species choice.

Meanwhile, although the data presented here contributed for understand the oak wood role in red wine characteristics, it must be remembered that the results showed are not definitive. Several factors remain unknown, particularly with regard to the relation between the chemical variation and sensory response, the influence of difference oak wood chips concentration, the aged time and the interaction between phenolic compounds from wine and the compounds extractable from oak wood.

The results obtained for the oak wood species (Portuguese and French) and chip concentration (4 g/L) used, in wine phenolic compounds evolution was negligible, except for non-flavonoid phenolic and for some

individual anthocyanins content (especially for malvidin-3-glucoside), independently of oak wood species used. At the same time, this result showed that the wood species did not lead to significant differences in wine proanthocyanidin content evolution.

In respect of the sensory evaluation, our results indicated that the wine aged in contact with oak wood chips (independently of oak wood species used) obtained higher scores for intensity, toasted, wood and vanillin aroma, taste intensity and global appreciation than the standard wine. Meanwhile between the wines aged in contact with different oak wood chip species, only for woody character was possible to detect significant differences, with a higher punctuation for the wine aged in contact with Portuguese oak chips.

Finally, in our study we used Portuguese oak wood samples only from one Portuguese region (Gerês region) and not from all Portuguese forest regions. Thus, in order to have a more global knowledge about the importance of this oak wood species, it will be necessary to use a great number of oak wood samples from different Portuguese forest regions and not only single samples from one forest region.

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## BIBLIOGRAPHY

- BELCHIOR A.P., CALDEIRA I., TRALHÃO G., COSTA S., LOPES C., CARVALHO E., 1998. Incidência da origem e queima da madeira de carvalho (*Q. pyrenaica*, *Q. robur*, *Q. sessiflora*, *Q. alba/stellata* + *Q. lyrata/bicolor*) e de castanho (*C. sativa*) em características físico-químicas e organolépticas de aguardentes Lourinhã em envelhecimento. *Ciência Tec. Vitiv.*, **13**, 97-108.
- CANAS S., CONCEIÇÃO M.L., SPRANGER M.I. and BELCHIOR A.P., 2000a. Influence of botanical species and geographical origin on the content of low molecular weight phenolic compounds of woods used in Portuguese cooperage. *Holzforchung*, **54**, 255-261.
- CANAS S., GRAZINA N., BELCHIOR A.P., SPRANGER M.I. and BRUNO DE SOUSA R., 2000b. Modelisation of heat treatment of Portuguese oak wood (*Quercus pyrenaica* L.). Analysis of the behaviour of low molecular weight phenolic compounds. *Ciência Tec. Vitiv.*, **15**, 75 - 94.
- CASTELLARI M., PIERMATTI B., ARFELLI G. and AMATI A., 2001. Influence of aging conditions on the quality of red sangiovese wine. *J. Agric. Food Chem.*, **49**, 3672-3676.
- CHATONNET P., BOIDRON J.N. et PONS M., 1989. Incidence du traitement thermique du bois de chêne sur sa composition chimique. 2<sup>e</sup> partie: évolution de certains composés en



- fonction de l'intensité de brûlage. *J. Int. Sci. Vigne Vin*, **23**, 223-250.
- CHATONNET P., BOIDRON J.N. and PONS M., 1990. Maturation of red wines in oak barrels: evolution of some volatile compounds and their aromatic impact. *Sci. Aliments*, **10**, 565-587.
- CHATONNET P., RICARDO-DA-SILVA J.M. et DUBOURDIEU D., 1997. Influence de l'utilisation de barriques en chêne sessile européen (*Quercus petraea*) ou en chêne blanc américain (*Quercus alba*) sur la composition et la qualité des vins rouges. *Rev. Fr. Oenol.*, **165**, 44-48.
- DALLAS C. and LAUREANO O., 1994. Effects of pH, sulphur dioxide, alcohol content, temperature and storage time on colour composition of a young Portuguese red table wine. *J. Sci. Food Agric.*, **65**, 477-485.
- ESCALONA H., BIRKMYRE L., PIGGOTT J.R. and PATERSON A., 2002. Effect of maturation in small oak casks on the volatility of red aroma compounds. *Anal. Chim. Acta*, **458**, 45-54.
- FEUILLAT F., MOIO L., GUICHARD E., MARINOV M., FOURNIER N. and PUECH J.L., 1997. Variation in the concentration of ellagitannins and *cis*- and *trans*- $\beta$ -methyl- $\gamma$ -octalactone extracted from oak wood (*Quercus robur* L., *Quercus petraea* Liebl.) under model wine cask conditions. *Am. J. Enol. Vitic.*, **48**, 509-515.
- GÓMEZ C.C. and GONZÁLEZ-SAN JOSÉ M.L., 1995. Interpretation of colour variables during the aging of red wines: relationship with families of phenolic compounds. *J. Agric. Food Chem.*, **43**, 557-561.
- GONZÁLEZ-SAN JOSÉ M.L. and REVILLA I., 2001. Effect of different oak woods on aged wine color and anthocyanin composition. *Eur. Food Res. Technol.*, **213**, 281-285.
- GONZALEZ-MENDOZA L.A. and POMAR M., 2001. Changes in composition and sensory quality of red wine aged in American and French oak barrels. *J. Int. Sci. Vigne Vin*, **35**, 41-48.
- HOP P., SILVA M.C.M. and HOGG T.A., 2001. Changes in colour and phenolic composition during the early stages of maturation of porto in wood, stainless steel and glass. *J. Sci. Food Agric.*, **81**, 1269-1280.
- HOLLMAN P.C.H., 2001. Evidence for health benefits of plant phenolics: local or systemic effects ? *J. Sci. Food Agric.*, **81**, 842-849.
- JINDRA J.A. and GALLENDER J.F., 1987. Effect of American and French oak barrels on the phenolic composition and sensory quality of Seyval blanc wine. *Am. J. Enol. Vitic.*, **38**, 133-138.
- JORDÃO A.M., RICARDO-DA-SILVA J.M. and LAUREANO O., 2005. Comparison of volatile composition of cooperage oak wood of different origins (*Quercus pyrenaica* vs. *Quercus alba* and *Quercus petraea*). *Mitt. Klosterneuburg*, **55**, 31-40.
- KADIM D. and MANNHEIM C.H., 1999. Kinetics of phenolics extraction during ageing of model wine solutions and white wine. *Am. J. Enol. Vitic.*, **50**, 33-39.
- LASZLAVIK M., GÁL L., MISIK S. and ERDEI L., 1995. Phenolic compounds in two Hungarian red wines matured in *Quercus robur* and *Quercus petraea* barrels: HPLC analysis and diode array detection. *Am. J. Enol. Vitic.*, **46**, 67-74.
- MATEJÍČEK D., MIKES O., KLEJDUS B., STERBOVA D. and KUBÁN V., 2005. Changes in contents of phenolic compounds during maturing of barrique red wines. *Food Chem.*, **90**, 791-800.
- MARTINEZ R.G., GARCIA DE LA SERRANA, H.L., MIR M.V., GRANADOS J.Q. and MARTINEZ C.L., 1996. Influence of wood heat treatment, temperature and maceration time on vanillin, syringaldehyde, and gallic acid contents in oak wood and wine spirit mixtures. *Am. J. Enol. Vitic.*, **47**, 441-446.
- MOUTOUNET M., RABIER P.H., PUECH J.L., VERETTE E., BARILLERE J.M., 1989. Analysis by HPLC of extractable substances in oak wood. Application to a Chardonnay wine. *Sci. Aliments*, **9**, 35-51.
- NAGEL C.W. and WULF L.W., 1979. Changes in the anthocyanins, flavonoids and hydroxycinnamic acid esters during fermentation and aging of Merlot. *Am. J. Enol. Vitic.*, **30**, 111-116.
- OIV, 1990. Recueil des méthodes internationales d'analyse des vins et des moûts. *Office International de la Vigne et du Vin*, Paris.
- PENG S., SCALBERT A. and MONTIES B., 1991. Insoluble ellagitannins in *Castanea sativa* and *Quercus petraea* woods. *Phytochemistry*, **30**, 775-778.
- PÉREZ-PRIETO L.J., LÓPEZ-ROCA J.M., MARTÍNEZ-CUTILLAS A., PARDO M.F. and GÓMEZ-PLAZA E., 2002. Maturing wines in oak barrels. Effects of oak origin, volume and age of the barrel on the wine volatile composition. *J. Agric. Food Chem.*, **50**, 3272-3676.
- PÉREZ-PRIETO L. J., HERA-ORTS M.L., LÓPEZ-ROCA J.M., FERNÁNDEZ-FERNÁNDEZ J.I. and GÓMEZ-PLAZA, E., 2003. Oak-matured wines: influence of the characteristics of the barrel on wine colour and sensory characteristics. *J. Sci. Food Agric.*, **83**, 1445-1450.
- PISARNITSKII A.F., 1995. Aldehydes as a factor of lignin degradation in the seasoning of spirits. *Applied Bioch. Microb.*, **31**, 126-128.
- POMAR M. and GONZALEZ-MENDOZA L. A., 2001. Changes in composition and sensory quality of red wine aged in American and French oak barrels. *J. Int. Sci. Vigne Vin*, **35**, 41-48.
- QUINN M.K. and SINGLETON V. L., 1985. Isolation and identification of ellagitannins from white oak and an estimation of their roles in wine. *Am. J. Enol. Vitic.*, **36**, 429-434.
- REAZIN G.H., 1981. Chemical mechanisms of whiskey maturation. *Am. J. Enol. Vitic.*, **32**, 283-289.
- ROUS C. and ALDERSON B., 1983. Phenolic extraction curves for white wine aged in French and American oak barrels. *Am. J. Enol. Vitic.*, **34**, 211-215.
- SHAHIDI F. and NACZK M., 1995. *Food Phenolics*. Technomic Publishing, basel, Switzerland. 94-97.
- SHRIKHANDE A.J., 2000. Wine by-products with health benefits. *Food Res. Int.*, **33**, 467-475.

- SINGLETON V.L., SULLIVAN A.R. and KRAMER C., 1971. An analysis of wine to indicate aging in wood or treatment with wood chips or tannic acid. *Am. J. Enol. Vitic.*, **22**, 161-166.
- SINGLETON V.L., 1995. Maturation of wines and spirits: Comparisons, facts, and hypotheses. *Am. J. Enol. Vitic.*, **46**, 98-115.
- SOMERS T.C. and EVANS M.E., 1977. Spectral evaluation of young red wines: anthocyanins equilibria, total phenolics, free and molecular SO<sub>2</sub>, «chemical age». *J. Sci. Food Agric.*, **28**, 279-287.
- SUN B.S., LEANDRO C., RICARDO-DA-SILVA J.M. and SPRANGER I., 1998a. Separation of grape and wine proanthocyanidins according to their degree of polymerization. *J. Agric. Food Chem.*, **46**, 1390-1396.
- SUN B.S., RICARDO-DA-SILVA J.M. and SPRANGER I., 1998b. Critical factors of vanillin assay for catechins and proanthocyanidins. *J. Agric. Food Chem.*, **46**, 4267-4274.
- VIRIOT C., SCALBERT A., LAPIERRE C.L. and MOUTONNET M., 1993. Ellagitannins and lignins in aging of spirits in oak barrels. *J. Agric. Food Chem.*, **41**, 1872-1879.
- YOKOTSUKA K., MATSUNAGA M. and SINGLETON V.L., 1994. Comparison of composition of Kosku white wines fermented in oak barrels and plastic tanks. *Am. J. Enol. Vitic.*, **45**, 11-16.

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