Received: 15 Juillet 2020 | Accepted: 10 November 2020 | Published: 7 December 2020
DOI:10.20870/oeno-one.2020.54.4.4026

# Impact of oak (*Q. pyrenaica* and *Q. pubescens*) and cherry (*P. avium*) wood chip contact on phenolic composition and sensory profile evolution of red wines during bottle storage

Manuela Costa[1], Luisa Fontes[1], Ana C. Correia[1], Uroš Miljić[2] and António M. Jordão[1,3*]

[1] Polytechnic Institute of Viseu, Agrarian Higher School, Department of Food Industries, 3500-606 Viseu, Portugal
[2] University of Novi Sad, Faculty of Technology, Novi Sad, Serbia
[3] Chemistry Research Centre of Vila Real (CQ-VR), 5001-801 Vila Real, Portugal

*corresponding author: antoniojordao@esav.ipv.pt

## ABSTRACT

The majority of published work has studied the impact of wood chips on red wine composition by conducting analyses during wood chip contact or immediately after the removal of chips from wine. Less attention has been directed at the potential influence of prior chip-wine contact on the further phenolic and sensory evolution of red wines during bottle storage. Therefore, this work focuses on the evolution over a period of 18 months of several phenolic parameters and sensory characteristics of bottled Touriga Nacional red wines that had previously been in contact with toasted wood chips from cherry (*Prunus avium*) and two oak species (*Quercus pyrenaica* and *Quercus pubescens*) during 30 days of pre-bottling storage. Various global phenolic parameters, colour properties, individual anthocyanin content and sensory profile of the wines were studied at 6, 12 and 18 months of bottle storage. The results showed less decrease in the phenolic composition and red colour of wines which had prior contact with oak chips, as well as a less developed brown colour during bottle storage, compared to the wine previously in contact with cherry chips and the control wine. In addition, wine previously in contact with cherry chips always showed an evolution similar to the control wine. From a sensory point of view, the wines previously in contact with oak wood chips showed a tendency for higher aroma scores for "vanilla" and "coconut" descriptors and lower scores for "brown colour" during bottle storage than wines previously in contact with cherry chips and the control wine. The outcomes of this research could be of practical interest to winemakers since they could improve the knowledge of the impact of prior contact with wood chips in the future evolution of the red wines during bottle storage.

## KEYWORDS

bottle storage, red wine, cherry, oak, phenolic, sensory characteristics, wood chips

 © 2020 International Viticulture and Enology Society - IVES 

## INTRODUCTION

The possibility of the use of wood fragments in wine production, including during the aging process, has emerged. However, this option only became possible, for example, in Europe after the publication of EEC regulation in 2006, more recently modified by the EEC Regulation N° 2019/934 of 12 March 2019 (Appendix 7), which changed previous rules about the use of pieces of oak for winemaking and the description and presentation of wine undergoing this treatment. It states that the pieces of oak wood used for winemaking and aging must exclusively come from the *Quercus* genus and be used at different toasting levels or without toasting.

Traditionally, there are three main species of wood used in oenology: *Q. petraea* Liebl. and *Q. robur* L., the most common oak species in French forests, and *Q. alba* L, the American oak. Therefore, scientific literature contains a large amount of data related to the impact of the different oak wood species of French and American origin when using oak barrels (Fernández de Simón *et al*., 2003a; Chira and Teissedre, 2015) and oak fragments (De Coninck *et al*., 2006; Tavares *et al*., 2017) on red wine chemical and sensory characteristics. However, in terms of the use of other oak wood species, the literature is scarcer. A few studies have reported the potential use in oenology of other oak wood species, namely *Quercus pyrenaica* (De Coninck *et al*., 2006; Fernández de Simón *et al*., 2008; Gonçalves and Jordão, 2009; Gallego *et al*., 2012; Tavares *et al*., 2017), *Quercus faginea* (Fernández de Simón *et al.*, 2003a; Fernández de Simón *et al.*, 2003b), *Quercus frainetto* (Vivas, 2005), *Quercus oocarpa* (Vivas *et al*., 1996), and *Quercus humboldtii* from Colombia (Martínez-Gil *et al.*, 2017; Martínez-Gil *et al.*, 2018a). No studies exist on the potential use in wine aging of *Quercus pubescens* (usually referred to as Pubescent oak), one of the oak wood species used in this study. According to Bordács *et al*. (2019), this is one of the most abundant tree species in central and south eastern European forests, as well as in the Anatolia region.

Other wood species, namely acacia (*Robinia pseudoacacia*), cherry (*Prunus avium* L.) and chestnut (*Castanea sativa* Mill), have been widely used for oenological purposes in the Mediterranean area in the past, due to their widespread availability and low cost. In recent years, study and interest in them has been renewed. The increased demand for these non-oak wood species results from the fact that the increased demand for traditional oak wood species for wine production has caused a remarkable limited availability of materials and environmental costs (Martínez-Gil *et al*., 2018b).

In general, cherry wood is usually characterised by an abundance of condensed tannins (procyanidin type) and appreciable values of (+)-catechin, but very low levels of hydrolysable tannins. In addition, it is possible to use some phenolic acids and their esterification products (benzoic acid, *p*-hydroxybenzoic acid, 3,4,5-trimethylphenol, *p*-coumaric acid, methylsyrin-gate and methyl-vanillate), and flavonoids (such as naringenin, aromadendrin, isosakuranetin and taxifolin) on cherry wood as phenolic markers for authentication purposes (De Rosso *et al*., 2009a; Springmann *et al*., 2011; Sanz *et al*., 2012a; Jordão *et al*., 2016). When compared to oak, this wood also has high levels of some volatile compounds, such as methyl syringate and benzoic acid, but low levels of phenyl aldehydes and phenyl ketones, except vanillin and syringaldehyde (Sanz *et al*., 2010a; Sanz *et al*., 2012b).

Some studies have been recently published regarding the use of cherry wood for white (Délia *et al*., 2017; Del Galdo *et al*., 2019), rosé (Santos *et al*., 2019; Nunes *et al*., 2020) and red (Chinnici *et al*., 2015; Fernández de Simón *et al*., 2014a; Tavares *et al*., 2017) wine aging. Some of these studies, particularly those on red wines, demonstrated that wines aged in cherry barrels or in contact with cherry chips showed intermediate quality (i.e., in terms of phenolic content and sensory evaluation) compared to red wines aged in contact with oak woods. However, according to Chinnici *et al*. (2011), the use of cherry barrels results in the faster evolution of wine pigments with a fast increase in the formation of derived and polymeric compounds. Furthermore, cherry barrels were shown to provide a favourable environment for oxidative reactions, thus making them less suitable for longer aging periods (De Rosso *et al*., 2009b). Tavares *et al*. (2017) reported that red wines aged in contact with cherry chips during a period of 90 storage days received similar sensory parameter scores for visual and taste descriptors to the red wines aged in contact with oak chips.

Despite the above-mentioned work demonstrating the value of different woods in oenology, in general published work has studied the impact

 © 2020 International Viticulture and Enology Society - IVES 

of oak and cherry wood chips on red wine composition by conducting analyses immediately after removal of wood chips from wine or during the contact of wine with wood chips. Therefore, less attention has been paid to the potential influence of previous wine-chip contact on the further evolution of wine characteristics during wine bottle storage. Moreover, several studies have been previously conducted on the evolution of the phenolic composition and sensory properties of red wines during bottle storage (Monagas *et al*., 2006; Dobrei *et al*., 2010; Gómez-Gallego *et al*., 2013; Avizcuri *et al*., 2016; Guiffrida de Esteban *et al*., 2019). In general, these published studies have reported the strong influence of storage time, oxygen levels present in the wines, grape variety used, initial wine phenolic composition, storage temperature and closure type on the phenolic and sensory changes which occur during bottle storage. According to Monagas *et al*. (2003), changes in phenolic content during aging in bottles (i.e., a decrease in anthocyanins due to the disappearance of monomeric anthocyanins by precipitation and condensation reactions) are expected to affect the red wine colour in different ways, including a decrease in colour intensity**.** In addition, it is well known that during wine maturation and aging in bottles, phenolic compounds participate in numerous chemical reactions. Condensed pigments are thus formed between free anthocyanins and the colourless phenols present in grapes during wine storage, including during wine storage in bottles. In addition, anthocyanins are progressively transformed into more stable oligomeric and polymeric pigments, thereby inducing significant changes to the colour and astringency of wines (Gómez-Plaza *et al*., 2002).

In this context, the present study aims to evaluate several global phenolic parameters, individual anthocyanins and the sensory profile evolution of red wines stored in bottles for 18 months after having previously been in contact with oak and cherry wood chips for 30 days. In addition, this work will contribute to the better understanding of the potential use of oak wood chips from *Quercus pubescens* species in oenology.

## MATERIAL AND METHODS

### 1. Red wine and wood chips

The red wine used in this experiment was a varietal wine made from Portuguese *Vitis vinifera* cv red grape variety Touriga Nacional, harvested at the technological stage of ripeness (1250 kg with 24 °Brix) in September 2017 in the Dão region (northwest Portugal), following standard red winemaking technology with a maceration time of 7 days at 24 °C ±2. The sulfitation of the grapes (30 mg/L of $SO_2$) was followed by alcoholic fermentation, which was carried out in a closed stainless-steel tank (1000 L) using a standard *Saccharomyces cerevisiae* yeast strain (Fermol Plus by AEB Group, Brescia, Italy) and inoculated at 20 g/hL. After alcoholic and malolactic fermentation, the wine was kept in the stainless-steel tank under controlled conditions (temperature 20 °C) for 3 months and analysed for the free $SO_2$ level regularly. At the beginning of wine storage in contact with wood chips, the main wine physicochemical characteristics were as follows: alcohol content = 13.0 % (v/v); pH = 3.58; total acidity = 5.59 g/L (expressed as tartaric acid); volatile acidity = 0.42 g/L (expressed as acetic acid); sugar content = 1.5 g/L and free $SO_2$ = 35 mg/L.

The wood chips used in this research were cherry (*Prunus avium*) purchased from AEB Bioquímica (Viseu, Portugal), Iberian oak (*Quercus pyrenaica*) from J.M. Gonçalves Cooperage (Palaçoulo, Portugal) and Pubescent oak (*Quercus pubescens*) from Djordjevic Cooperage (Vranje, Serbia). According to information provided by the cooperages, all wood chips used underwent medium toasting (15 min at 165 °C on the wood surface for cherry chips, 20 min at 170 °C on the wood surface for Iberian oak chips and 20 min at 180 °C on the wood surface for Pubescent oak chips) and had a medium particle size of 8 mm.

### 2. Experimental conditions

The experimental work was performed in duplicate at laboratory scale (20 litres for each assay), according to the procedure summarised in Figure 1. The treatment consisted of the wines being kept in contact with the different wood chip species (2 g/L) in closed stainless-steel vats for 30 days prior to bottling. The characteristics (level of toast and particle size) and concentration of the wood chips used in this experimental work, particularly for the oak species, were based on previous work carried out on red wines of the Touriga Nacional variety (Tavares *et al*., 2017) also produced in the Dão region.


© 2020 International Viticulture and Enology Society - IVES

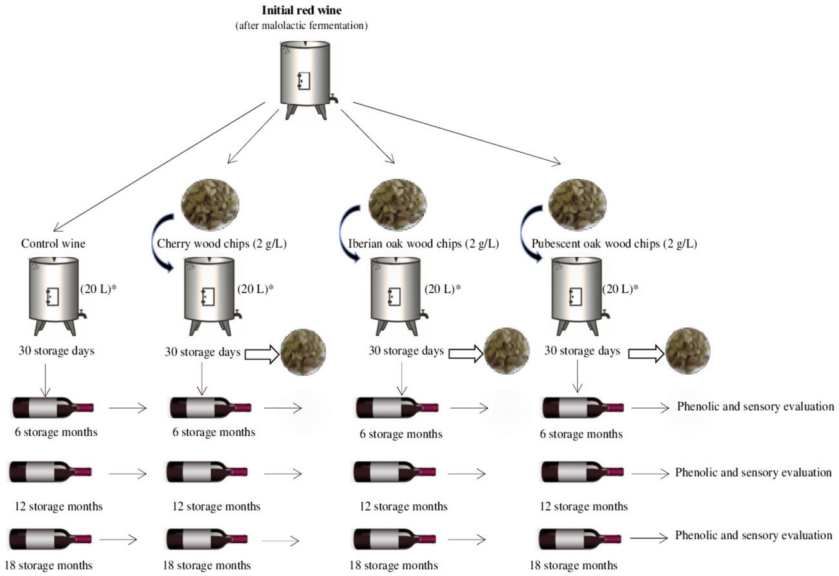



**FIGURE 1.** Experimental procedure developed in the work.
* experimental work performed in duplicate.

Each wood chip species was placed in a sanitised polyethylene bag in the central part of each vat, without agitating the contents during the contact time. The vats were only opened after 30 days. After this time, the wood chips were removed and the wines analysed. Subsequently, all the wines were bottled in 0.75 L green bottles with cork closures (Neutrocork®, Amorim cork company, Santa Maria de Lamas, Portugal), and characterised every 6 months, corresponding to 6, 12 and 18 storage months. Immediately after manual filling, and before applying the cork closure, nitrogen was added to remove oxygen from the bottle headspace (5 mL). For each assay and storage time considered, 5 bottles were selected for chemical characterisation (3 bottles) and sensory evaluation (2 bottles). Before wine bottling, the values for free $SO_2$ were adjusted to 35 mg/L. All wines during this experiment were stored at cellar temperature (between 16-17 ºC) and kept in the dark. The wine samples were filtered (pore diameters of 13 µm) before laboratory analysis.

## 3. General wine physicochemical characterisation

The general red wine physicochemical characterisation (pH, total and volatile acidity, alcohol strength, total and free sulfur dioxide) was performed following the analytical methods recommended by International Organization of Vine and Wine (OIV, 2014). All analyses were done in triplicate.

## 4. Global phenolic parameters

During the red wine bottle storage, several global phenolic parameters were analysed. Total polyphenolic content was determined according to Ribéreau-Gayon *et al.* (2006) methodology, while non-flavonoid and flavonoid phenols were determined using the improved method described by Kramling and Singleton (1969). Briefly, the quantification of non-flavonoid phenols was based on the determination of the phenolic content before and after the precipitation of flavonoids through reaction with formaldehyde under certain conditions (low pH, room temperature and darkness). After 24 hours, a dilution with distilled water (1:10) was carried out and the absorbance was read at 280 nm on a UV-Vis spectrophotometer (model UV-1900i, Shimatzu, Duisburg, Germany). Flavonoid phenols come from subtracting non-flavonoid phenols from total phenols. The results obtained were expressed as gallic acid equivalents by means of calibration curves with standard gallic acid (Extrasynthese, Genay, France).

Total pigments, total and coloured anthocyanins, and polymeric pigments were quantified according to Somers and Evans (1977). For total and coloured anthocyanins, the results were expressed as malvidin-3-monoglucoside equivalents by means of calibration curves with the standard of this individual monomeric anthocyanin (Extrasynthese, Genay, France). Colour intensity at 420, 520 and 620 nm and

colour hue were evaluated following the methodology described by the OIV (2014).

Tanning power was quantified following the methodology developed by De Freitas and Mateus (2001). This method included a 1:50 dilution with a hydroalcoholic solution (12 % v/v and pH 3.2 at 20 ºC), followed by a reading (d0) on a turbidimeter (model HI93703 Hanna Instruments, Limena, Italy). Then, 8 mL of the previous dilution and 300 µL of BSA (bovine serum albumin) were put in a tube and, after agitation and 45 min in the darkness, a second reading was carried out on the turbidimeter (d1). The final value (NTU/mL) was calculated as Tanning power = (d1 - d0)/0.08. All laboratory measurements were performed in triplicate.

**5. Individual monomeric anthocyanins analysis**

For the analysis of individual monomeric anthocyanins, the apparatus used was a high-performance liquid chromatography (HPLC) Dionex Ultimate 3000 Chromatographic System (Sunnyvale, CA, USA) equipped with a quaternary pump Model LPG-3400 A, an auto sampler Model ACC-3000, a thermostatted column compartment (adjusted to 25 °C) and a multiple Wavelength Detector MWD-300. The column (250 x 4.6 mm, particle size 5 µm) was a $C_{18}$ Acclaim® 120 (Dionex) protected by a guard column of the same material. The solvents were (A) 40 % (v/v) formic acid, (B) pure acetonitrile and (C) bi-distilled water. The individual anthocyanins were analysed by HPLC using the method described by Dallas and Laureano (1994); 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 0.7 mL/min. The injection volume was 40 µL. Detection was carried out at 520 nm, for which a Chromeleon software program version 6.8 (Dionex, Sunnyvale, CA, USA) was used. Individual anthocyanins were quantified using a calibration curve obtained with standard solutions containing different concentrations (covering a range of 0-800 mg/L) of malvidin-3-monoglucoside chloride (> 9 % purity, Extra-synthese, Genay, France). The chromatographic peaks of anthocyanins were identified according to reference data previously described by Dallas and Laureano (1994). All analyses were done in triplicate.

**6. Sensory evaluation**

After 6, 12 and 18 months of bottle storage, the red wine samples were evaluated by seven expert judges. Furthermore, at the beginning (i.e., after 30 days of wood chip contact), the wines were tasted immediately before bottling. Each red wine sample was stored for 24 hours at room temperature before sensory analysis, which was performed at 20-22 ºC in a sensory analysis room with individual booths for each expert and according to standardised procedures (ISO 3591, 2016). Samples of 30 mL from each wine sample were presented to the panel in tasting glasses marked with three-digit numbers. In addition, wine samples were presented to each expert judge in a different randomised order. All data were collected using specific sensory evaluation sheets.

All evaluations were conducted in the afternoon (between 16:00 to 17:00 ) and always by the same seven expert judges (4 men and 3 women aged between 44 and 60 years old and with over 15 years of wine tasting experience) from Dão Wine Institute (the official organisation that certifies wines produced in the Dão region of northeast Portugal). All expert judges had been previously selected and trained for 6 months to assess the sensorial attributes of wines produced in the Dão wine region (including red wines from the Touriga Nacional grape variety, one of the most common red grape varieties from this region, which was used to produce the wines used in this study). During this training period and under supervision of the panel leader, several sessions were carried out in order to train judges about the meaning of each attribute and to achieve reliable intensity ratings. The sensorial attributes used were grouped in the following way: colour ("red" and "brown"), aroma ("fruity", "floral", "vanilla", "boisé", "coconut", "sawdust" and "balance"), taste ("body", "bitterness", "astringency", "persistence" and "balance") and overall appreciation. The experts scored each sensory attribute on a scale of 1 to 5 (1 = "absence"; 2 = "little intensity"; 3 = "moderate intensity"; 4 = "intense"; 5 = "high intensity") according to their sensory knowledge, training and experience. Overall appreciation was also scored on a scale of 1 to 5 (1 = "bad"; 2 = "pleasant"; 3 = "good"; 4 = "very good"; 5 = "excellent").


© 2020 International Viticulture and Enology Society - IVES

**TABLE 1.** Evolution of general physicochemical characteristics of the red wines during the experimental work.

| Experimental storage conditions (wine codes) | General physicochemical parameters | After 30 storage days with wood chip contact (30D) | Bottle storage months | | |
|---|---|---|---|---|---|
| | | | 6M | 12M | 18M |
| Control wine (CW) | pH | $3.60^{a(*)} \pm 0.01$ | $3.63^{a} \pm 0.01$ | $3.67^{a} \pm 0.02$ | $3.72^{a} \pm 0.01$ |
| | Total acidity (g/L tartaric acid) | $5.55^{a} \pm 0.03$ | $5.50^{a} \pm 0.02$ | $5.48^{a} \pm 0.01$ | $5.20^{a} \pm 0.03$ |
| | Volatile acidity (g/L acetic acid) | $0.43^{a} \pm 0.03$ | $0.45^{a} \pm 0.01$ | $0.47^{a} \pm 0.02$ | $0.49^{a} \pm 0.01$ |
| | Free sulfur dioxide (mg/L) [(a)] | $33^{a} \pm 1$ | $31^{a} \pm 2$ | $29^{a} \pm 1$ | $26^{a} \pm 3$ |
| Wine + Cherry wood chips (WCH) | pH | $3.60^{a} \pm 0.01$ | $3.62^{a} \pm 0.01$ | $3.65^{a} \pm 0.02$ | $3.74^{a} \pm 0.01$ |
| | Total acidity (g/L tartaric acid) | $5.56^{a} \pm 0.01$ | $5.51^{a} \pm 0.01$ | $5.50^{b} \pm 0.01$ | $5.15^{a} \pm 0.02$ |
| | Volatile acidity (g/L acetic acid) | $0.45^{a} \pm 0.01$ | $0.46^{a} \pm 0.01$ | $0.48^{a} \pm 0.01$ | $0.51^{a} \pm 0.02$ |
| | Free sulfur dioxide (mg/L) [(a)] | $30^{a} \pm 2$ | $31^{a} \pm 2$ | $28^{a} \pm 1$ | $23^{a} \pm 2$ |
| Wine + Iberian oak wood chips (WIO) | pH | $3.59^{a} \pm 0.01$ | $3.61^{a} \pm 0.01$ | $3.66^{a} \pm 0.02$ | $3.76^{a} \pm 0.01$ |
| | Total acidity (g/L tartaric acid) | $5.54^{a} \pm 0.01$ | $5.53^{a,b} \pm 0.01$ | $5.49^{a,b} \pm 0.02$ | $5.07^{b} \pm 0.02$ |
| | Volatile acidity (g/L acetic acid) | $0.44^{a} \pm 0.02$ | $0.45^{a} \pm 0.01$ | $0.46^{a} \pm 0.01$ | $0.47^{a} \pm 0.01$ |
| | Free sulfur dioxide (mg/L) [(a)] | $31^{a} \pm 2$ | $34^{a,b} \pm 1$ | $33^{b} \pm 2$ | $30^{a,b} \pm 2$ |
| Wine + Pubescent oak wood chips (WPO) | pH | $3.59^{a} \pm 0.01$ | $3.61^{a} \pm 0.01$ | $3.65^{a} \pm 0.02$ | $3.73^{a} \pm 0.02$ |
| | Total acidity (g/L tartaric acid) | $5.55^{a} \pm 0.01$ | $5.50^{a} \pm 0.01$ | $5.49^{a,b} \pm 0.01$ | $5.17^{a} \pm 0.01$ |
| | Volatile acidity (g/L acetic acid) | $0.45^{a} \pm 0.03$ | $0.47^{a,b} \pm 0.01$ | $0.49^{a} \pm 0.03$ | $0.53^{a,b} \pm 0.01$ |
| | Free sulfur dioxide (mg/L) [(a)] | $33^{a} \pm 1$ | $32^{a} \pm 2$ | $29^{a} \pm 2$ | $24^{a} \pm 2$ |

D - after 30 storage days in contact with wood chips; 6M - after 6 months of bottle storage; 12M - after 12 months of bottle storage; 18M - after 18 months of bottle storage; average values of three replicates ± standard deviation; [(*)] Average values for the same storage time (in column) and analytical parameter showing the same letter are not significantly different ($p < 0.05$); [(a)] before wine bottling the values for free sulfur dioxide were adjusted to 35 mg/L.

### 7. Statistical analysis

The data are presented as mean ± standard deviation. Phenolic and sensory parameter data were statistically tested by analysis of variance (ANOVA, one-way). Tukey test ($p < 0.05$) was applied to the data to determine significant differences between red wines. Principal component analysis (PCA) was used to analyse the data and to study the relationships between the red wines with previous wood chip contact and their phenolic composition and sensory characteristics during the bottle storage time. Since variables with different scales were used, the PCA analysis was performed with a previous standardisation of the initial variables and conducted using a correlation matrix.

In addition, a hierarchical cluster analysis (HCA) with the Ward criteria was also applied to the global phenolic and sensory parameters of all red wines. All analyses were performed using SPSS software version 26.0 (SPSS Inc., Chicago, IL, US).

## RESULTS AND DISCUSSION

### 1. Evolution of general physicochemical composition

Table 1, shows the general physicochemical characteristics of the different red wines after 30 storage days in contact with the different wood chip species, as well as after 6, 12 and 18 months of bottle storage.

During bottle storage, a slight decrease in total acidity was observed for all red wines. This decrease ranged from 0.35 to 0.47 g/L of tartaric acid. The total acidity decrease was probably due to a slight precipitation of tartaric acid in the form of potassium bitartrate that may have occurred in red wines over time, and which also contributed to the increase in pH values. Therefore, during bottle storage, the pH values increased between 0.09 and 0.17 pH units.

Slight changes in volatile acidity also occurred during the 18 months of bottle storage, with an increase in acetic acid which varied between 0.03 and 0.08 g/L. However, these changes were not appreciable and can only be considered acceptable when there is no microbiological spoilage during storage. The slight increase in volatile acidity observed may be associated with an oxidative chemical process, as suggested by other authors (Clarke and Bakker, 2004). A very slight and progressive decrease in free sulphur dioxide values of between 4 and 8 mg/L during bottle storage was detected. In addition, it is important to note that before wine bottling, the values for free sulphur dioxide were adjusted to 35 mg/L. Finally, for each storage period, the wines kept in contact with the different wood chip species generally showed similar values for the different general oenological parameters studied.

### 2. Global phenolic parameters

#### 2.1. Total, non flavonoid and flavonoid phenols

Figure 2 shows the total, non-flavonoid and flavonoid phenols content of red wines after 30 storage days in contact with different wood chip species and their changes during 18 months of bottle storage. After 30 storage days in contact with different wood chips, the red wines in contact with the two oak wood chip species under study had significantly higher values of total phenols (1980 and 1990 mg/L gallic acid equivalent respectively for the red wine in contact with Iberian and Pubescent oak wood chips), while the wine kept in contact with cherry chips and the control wine showed significantly lower values (1860 and 1849 mg/L gallic acid equivalent respectively). This increase in total phenol content is a clear consequence of phenol transfer from oak wood to wine. In fact, the majority of published work has also reported a higher phenolic composition in wines aged in contact with wood, in particular in contact with oak wood species (De Coninck *et al*., 2006; Nunes *et al*., 2017; Tavares *et al*., 2017; Santos *et al*., 2019; Nunes *et al*., 2020). In addition, the red wine kept in contact with cherry wood chips showed significantly lower values compared with the red wines in contact oak wood chips. These results can be explained by the low phenolic content usually quantified for cherry wood, particularly for ellagitannins, with respect to the values quantified for oak woods (Sanz *et al*., 2012a; Jordão *et al*., 2016).

Regarding the values obtained for the red wines kept in contact with the two oak wood chip species under study (Iberian and Pubescent oaks), no significant differentiation for total phenols among the red wines was found. A similar trend was also observed for flavonoid and non-flavonoid phenols, for which no significant differences were detected between the wines after 30 storage days in contact with


© 2020 International Viticulture and Enology Society - IVES

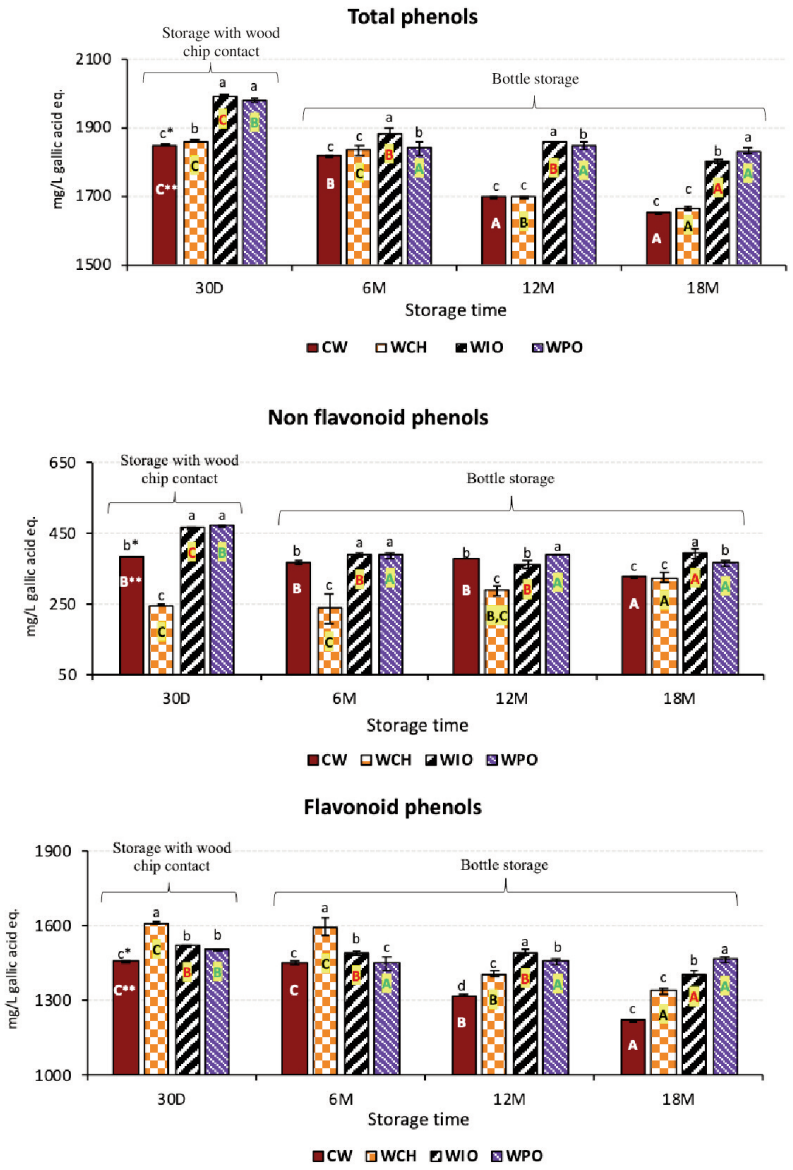



**FIGURE 2.** Total, non flavonoid and flavonoid phenols from red wines after 30 storage days in contact with different wood chip species and during 18 months of bottle storage.
CW - control wine; WCH - wine with cherry wood chip contact; WIO - wine with Iberian wood chip contact; WPO - wine with Pubescent wood chip contact; 30D - after 30 storage days in contact with wood chips; 6M - after 6 months of bottle storage; 12M - after 12 months of bottle storage; 18M - after 18 months of bottle storage; average values with same letter are not significantly different ($p < 0.05$), wherein lowercase letters (* over the bars) are used for wood chips species factor (for each storage time), and capital letters (** inside the bars) are used for storage time evolution factor (for each individual red wine); the error bars represent the standard deviation of the three replicates.

the two oak wood chip species used (Figure 2). Currently, not enough information is available for suitable industrial exploitation of pubescent oak wood; however, Bamber and Fukazawa (1985) previously reported that this oak species shows high levels of extractive compounds. For Bordács *et al*. (2019), *Quercus pubescens* wood is similar to *Q. petraea*, particularly in terms of ring porosity and heartwood colour. In addition, according to these authors, the genetic potential of Mediterranean oak populations (mainly *Q. pubescens*, *Q. virgiliana* and *Q. pyrenaica*, plus *Q. faginea* on the Iberian Peninsula) might assure a continuous gene composition between the populations. This fact may thus help to explain the similar values obtained for the

general phenolic composition of wines stored for 30 days in contact with both of the oak wood chip species used.

During the 18 months of bottle storage, total phenol values decreased for all red wines; this was particularly evident for the control wine and the wine previously in contact with cherry wood chips, with a percentage decrease of 10.7 % and 10.5 % during bottle storage respectively. After 18 months of bottle storage, the levels of total phenols for the control wine and wine previously in contact with cherry wood chips were also very similar (1650 and 1663 mg/L gallic acid equivalent respectively). In addition, red wines previously in contact with oak wood chips showed significantly higher values during the entire bottle storage time. At the end of the bottle storage time (18 months), these two red wines showed total phenol values of between 1800 and 1831 mg/L gallic acid equiv. for wines previously in contact with Iberian and Pubescent oak chips respectively.

In terms of non-flavonoid phenols, after 30 storage days in contact with the different wood chip species, red wines in contact with oak wood chips showed significantly higher values than the remaining red wine samples. These differences were particularly evident between wines stored in contact with oak and cherry wood chips. After 30 storage days in contact with wood chips, non-flavonoid phenol content showed the following tendency in descending order: wine stored in contact with Pubescent oak chips (474 mg/L gallic acid equiv.), wine stored in contact with Iberian oak chips (469 mg/L gallic acid equiv.), the control wine (383 mg/L gallic acid equivalent), and wine stored in contact with cherry wood chips (247 mg/L gallic acid equiv.). The highest non-flavonoid content detected in the red wines stored in contact with oak chips may correspond to a higher potential extraction of several individual non-flavonoid compounds, such as gallic, protocatechuic, vanillic, caffeic, syringic and *p*-coumaric acids, ellagitannins, ellagic acid and other compounds from this oak wood species. In fact, it is well known that European oak wood species are an important source of hydrolysable tannins (Jordão *et al.*, 2007; 2016), while an absence of hydrolysable tannins from cherry heartwood has been noticed by several authors (Sanz *et al.*, 2010b; Jordão *et al.*, 2016). In addition, wine stored in contact with cherry wood chips showed the lowest non-flavonoid phenol content. This could be due to the precipitation of phenolic compounds and oxidation and polymerisation phenomena, in which phenolic leakage from wood plays an important role. According to Chinnici *et al*. (2015), cherry wood promotes a faster evolution of wine constitutive phenols. After bottling, and throughout the 18 months of storage, the values of non-flavonoid phenols generally showed a very slight decrease with some slight oscillations in all the wines (except in the wine previously in contact with cherry wood chips, where there seems to be a slight tendency for the values to increase). In any case, after 18 months of bottle storage, the red wines previously in contact with oak wood chips maintained significantly higher values (393 and 365 mg/L gallic acid equiv. for wines in prior contact with Iberian and Pubescent oak wood chip species respectively), followed by the control wine and the wine in prior contact with cherry wood chips (328 and 324 mg/L gallic acid equiv. respectively).

Finally, with respect to the flavonoid phenols (Figure 2), after 30 storage days in contact with the different wood chip species, there were significantly higher values for the red wine in contact with cherry wood chips (1612 mg/L gallic acid equiv.) compared to the remaining wines. Red wines stored in contact with the two oak wood chip species under study represented the second group with similar values (1506 and 1521 mg/L gallic acid equiv. for wines stored with Pubescent and Iberian oak wood chip species respectively). The control wine showed a significantly lower value (1456 mg/L gallic acid equiv.). The significantly highest values for flavonoid phenols found in red wine stored in contact with cherry wood chips may correspond to a higher potential extraction of several condensed tannins and (+)-catechin. In fact, according to previous studies (Zhang *et al*., 2015; Jordão *et al*., 2016), cherry wood is usually characterised by an abundance of condensed tannins (procyanidin type), as well as by appreciable values for (+)-catechin in relation to the oak wood species. In addition, Fernández de Simón *et al*. (2014a) evaluated several phenolic markers in different varietal wines and found that wines aged with cherry wood chips for two months showed several specific flavonoid phenols, including aromadendrin, taxifolin, isosakuranetin, pruning, eriodictyol, naringenin and significantly higher concentrations of (+)-catechin.


© 2020 International Viticulture and Enology Society - IVES

After bottling and throughout the 18 months of bottle storage, the values of flavonoid phenols showed a tendency to decrease in all wines. However, this tendency was more evident for the control wine and the wines previously in contact with cherry wood chips. In fact, during bottle storage, a drop in the flavonoid phenol values for these two wines corresponded to a percentage decrease of 19.0 and 17.0 % respectively, while for the wines previously in contact with Iberian and Pubescent oak wood chips, the decrease was only between 2.7 and 7.5 % respectively. Thus, after 18 months of bottle storage, wines previously in contact with oak chips showed the significantly highest values (1406 and 1465 mg/L gallic acid equiv. for wines previously in contact with Iberian and Pubescent oak chips respectively), followed by the wine previously in contact with cherry chips (1338 mg/L gallic acid equiv.), and finally the control wine (1221 mg/L gallic acid equiv.).

On the other hand, when the evolution of each red wine was analysed individually during the storage period for the three global phenolic parameters, the quantified values generally showed a significant decrease during the entire bottle storage time. However, between 12 and 18 months of bottle storage, the decreases in the total phenol content for the control wine and the wine previously in contact with Pubescent oak chips were not significantly different. A similar trend was observed for non-flavonoid phenols, in particular for wine with previous contact with Pubescent oak chips, for which it was clear that total phenols were stabilising throughout the time of bottle storage. In addition, individual decreases for each red wine over the storage time were most significant for flavonoid phenols; these decreases in the values were generally significantly different for all red wines, except for the wine previously in contact with Pubescent oak chips, for which the values remained unchanged during the 18 months of bottle storage.

Generally speaking, the decrease in these global phenolic parameters studied during the bottle storage time could be due to the precipitation of phenolic compounds, oxidation and polymerisation phenomena, as phenolic leakage from wood plays an important role, which, in this case, was particularly evident when oak wood chips were used before bottle storage. Therefore, the extracted wood phenolic compounds may have contributed to a greater stability of wine phenolic compounds, reducing their loss during the bottle storage process. This is particularly evident for the red wines previously in contact with oak chips in relation to the red wine previously in contact with cherry chips and the control wine. Previous results have demonstrated the positive impact of oak wood components, particularly ellagitannins and ellagic acid, in terms of preventing the degradation of some wine flavanols, namely (+)-catechin and procyanidin B1 during the aging period (Jordão *et al*., 2006; Jordão *et al*., 2008). In addition, according to Chinnici *et al*. (2015), when compared to oak, cherry wood promotes a faster evolution of wine constitutive phenols, inducing a greater reduction of flavanol and flavonol phenols. These facts could help to explain why, after 18 months of bottle storage, red wine previously in contact with cherry wood chips generally showed similar values to the control wine, as well as lower values than red wines previously in contact with oak wood chips.

### 2.2. Total and polymeric pigments, and tanning power

Figure 3 shows the total and polymeric pigment values for the red wines after 30 storage days in contact with different wood chip species, and their changes during 18 months of bottle storage.

After 30 storage days in contact with wood chips, the wines in contact with oak chips showed significantly higher values for total pigments (36.2 and 34.4 abs. units for wines previously in contact with Pubescent and Iberian oak wood chips respectively), while the wine previously in contact with cherry chips and the control wine showed significantly lower values (29.5 and 28.1 abs. units respectively), with a significant difference between them.

During 18 months of bottle storage, the wines generally maintained the same differences between them as those observed after 30 days of wood chip contact. In addition, all total pigment values showed a tendency to slightly decrease during the bottle storage time; however, this was most pronounced for the wine previously in contact with Pubescent oak chips. Thus, after 18 months of bottle storage, the wines previously in contact with oak chips showed significantly higher values for total pigments (29.9 and 28.9 abs. units for wines with previous contact with Iberian and Pubescent oak chips respectively), followed by the wine previously in

contact with cherry chips (24.4 abs. units) and the control wine (20.8 abs. units). On the other hand, when the evolution of each red wine was analysed individually over the storage period for total pigments, the values quantified generally showed a non-significant decrease for wines previously in contact with Iberian oak wood chips, particularly between 12 and 18 months of bottle storage. In general, the remaining wines showed a significant decrease during the whole bottle storage period.

During red wine aging, tannins and anthocyanins may react either by directly forming flavanol anthocyanin or anthocyanin-flavanol adducts, or by generating molecular aggregates through the intervention of ethanol, whereby hydrolysable tannins extracted from the oak wood play an important role (Fulcrand *et al*., 2006). These formed compounds, called polymeric pigments, are responsible for colour stability in wines over time (Blanco-Vega *et al*., 2014). Thus, the monitoring of these parameters (such as polymeric pigments in red wines) contributes to assessing the changes that may occur in wines in terms of their colour, namely the redness component. The results obtained in our study showed that after 30 storage days in contact with the different wood chip species, the wine in contact with Iberian oak wood chips exhibited the significantly highest value for polymeric pigments (3.6 abs. units). For the remaining wines, there was no clear differentiation in the values (Figure 3). However, during the 18 months of bottle storage, and particularly after 12 months of bottle storage, there was a clear tendency towards a slight increase in polymeric pigments in red wines previously in contact with oak chips compared to the wine previously in contact with cherry chips and the control wine. This tendency confirms previous results obtained by other authors during bottle storage (Giuffrida de Esteban *et al*., 2019) and for bottled wines previously aged in oak wood barrels for 10 and 18 months (Avizcuri *et al*., 2016). After 18 months of bottle storage, the wines previously in contact with oak chips showed significantly higher polymeric pigment values (3.8 and 3.6 abs. units for wines previously in contact with Iberian and Pubescent oak chips respectively), while the control wine and the wine previously in contact with cherry wood chips showed significantly lower values without a clear differentiation between them (2.9 and 2.8 abs. units respectively). In fact, it was clear that there was a significant decrease in polymeric pigments in the latter two wines as from the sixth month of the 18 months of bottle storage.

With respect to the individual evolution of the different red wines, polymeric pigments changes were generally insignificant for up to 6 months of bottle storage. Only after this stage was a significant increase in polymeric pigments detected for wines previously in contact with oak chips. The values for the control wine were maintained between 12 and 18 months of bottle storage, while they significantly decreased for the wine previously in contact with cherry wood chips.

Regarding tanning power (Figure 3), as expected, all red wines in contact with wood chips for 30 storage days showed significantly higher values (particularly the wines stored in contact with oak chips, with values varying between 215 and 220 NTU/mL) than the control wine (150 NTU/mL). The wine stored in contact with cherry wood chips showed intermediate values (190 NTU/mL). This parameter represents the expression of the tannicity of a wine; i.e., the capacity of some tannins (such as proanthocyanidins with particular polymerisation degrees), to interact with proteins, influencing the astringent character of the wine at tasting. Regarding the individual evolution of each red wine during bottle storage, there was a general significant decrease of tanning power values for all wines, particularly as from 12 months of bottle storage.

In fact, the phenolic composition that characterises oak woods (Chira and Teissedre, 2015; Jordão *et al*., 2007; Jordão *et al*., 2012; Jordão *et al*., 2016), and the extraction of these phenolic compounds from wood to wine (in particular ellagitannins that have a high reactivity with proteins), may explain the significantly high tanning power values quantified in red wines kept in contact with wood chips for 30 storage days. In that case, high levels of interactions between these phenolic compounds and human saliva proteins will induce a potentially higher level of astringency in these wines. Moreover, the presence of (+)-catechin and procyanidins in cherry wood could also explain the significantly higher value also obtained for the wine stored in contact with cherry wood chips for 30 days compared to the control wine (Zhang *et al*., 2015; Jordão *et al*., 2016). During bottle storage, wines were generally differentiated for up to


© 2020 International Viticulture and Enology Society - IVES

12 months. However, after 18 months of storage, a marked decrease in the values was detected, despite the wines previously in contact with wood chips showing significantly higher values (particularly those previously in contact with oak chips). This decrease was more evident for wines previously in contact with cherry chips and for the control wine, for which there was a decrease of 39 and 33 % in tanning power values respectively. The tannin power decrease could be explained by the general decrease in phenolic content of the wine and, in particular, the increase in polymeric pigments, which show lower reactivity with human salivary proteins.

### 2.3. Anthocyanins and colour parameters

The results obtained for total and coloured anthocyanins, as well as the colour intensity and hue of red wines during the storage period are shown in Figure 4. After 30 storage days in

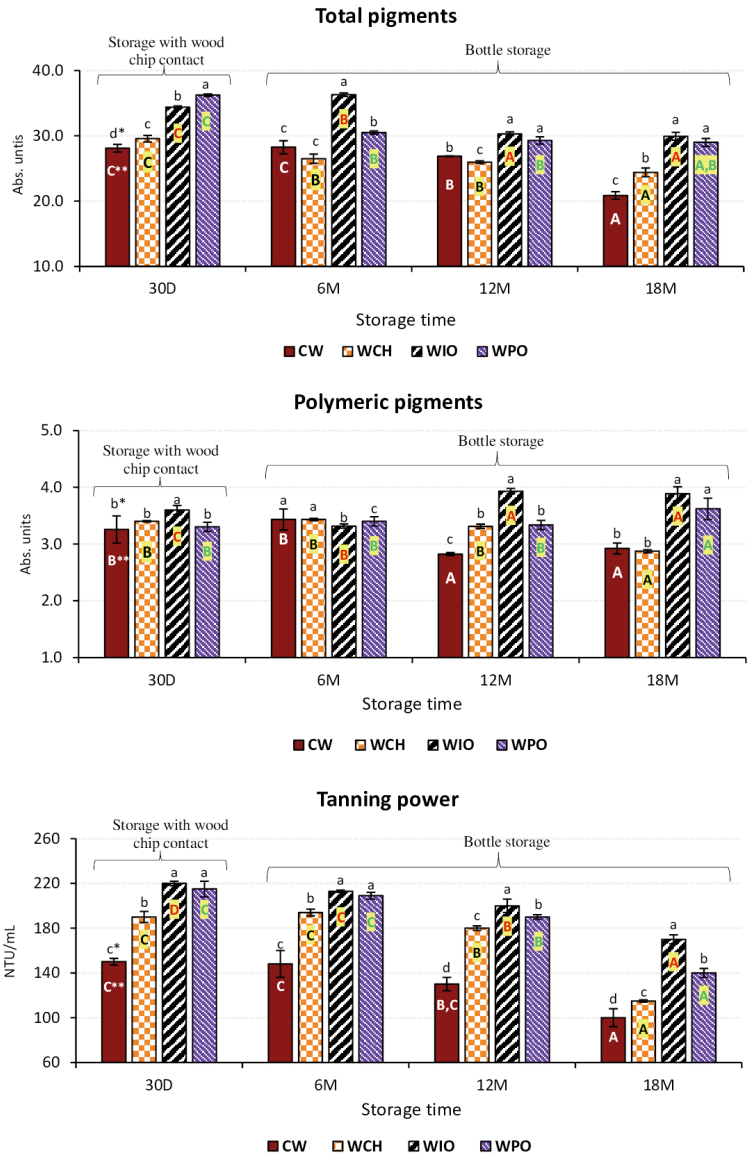



**FIGURE 3.** Total and polymeric pigments, and tanning power from red wines after 30 storage days in contact with different wood chip species and during 18 months of bottle storage.
CW - control wine; WCH - wine with cherry wood chip contact; WIO - wine with Iberian wood chip contact; WPO - wine with Pubescent wood chip contact; 30D - after 30 storage days in contact with wood chips; 6M - after 6 months of bottle storage; 12M - after 12 months of bottle storage; 18M - after 18 months of bottle storage; * average values with same letter are not significantly different ($p < 0.05$); wherein lowercase letters (* over the bars) are used for wood chips species factor (for each storage time), while capital letters (** inside the bars) are used for storage time evolution factor (for each individual red wine); the error bars represent the standard deviation of the three replicates.

 © 2020 International Viticulture and Enology Society - IVES 

contact with wood chips, it was possible to detect similar values for total anthocyanins between the control wine and the wine stored in contact with Pubescent oak wood chips. The values obtained for these two wines were significantly higher compared with the values obtained for the wines stored in contact with cherry and Iberian oak wood chips.

After bottling, and during the 18 months of bottle storage, there was a clear significant tendency for a decrease in total anthocyanins in all individual red wines, which was particularly evident as from 12 months of bottle storage. Red wine previously in contact with Pubescent oak wood chips was the exception, with values staying the same between 12 and 18 months of bottle storage.

The total anthocyanin decrease was more evident for the control wine and for the wine previously in contact with cherry wood chips (a decrease of 45 and 32 % respectively). For the remaining wines previously in contact with oak wood chips, the total anthocyanin decreases during bottle storage varied from 23 to 29 %. After 18 months of bottle storage, the wines with prior oak chip contact showed significantly higher values for total anthocyanins (322 and 319 mg/L of malvidin-3-monoglucoside equiv. for wines with prior contact with Pubescent and Iberian oak chips respectively), followed by the wine previously in contact with cherry chips (290 mg/L of malvidin-3-monoglucoside equiv.) and the control wine (250 mg/L of malvidin-3-monoglucoside equiv.). For coloured anthocyanin content (i.e., the content of free red-coloured anthocyanins in flavylium cation form), a similar tendency to that of total anthocyanins was found during the entire bottle storage period. After 18 months of bottle storage, wines with prior oak chip contact showed the significantly highest coloured anthocyanin values, followed by wine previously in contact with cherry chips and the control wine.

It follows that the aforementioned evolution of total and coloured anthocyanin values was reflected in the evolution of wine colour intensity during bottle storage. After 30 storage

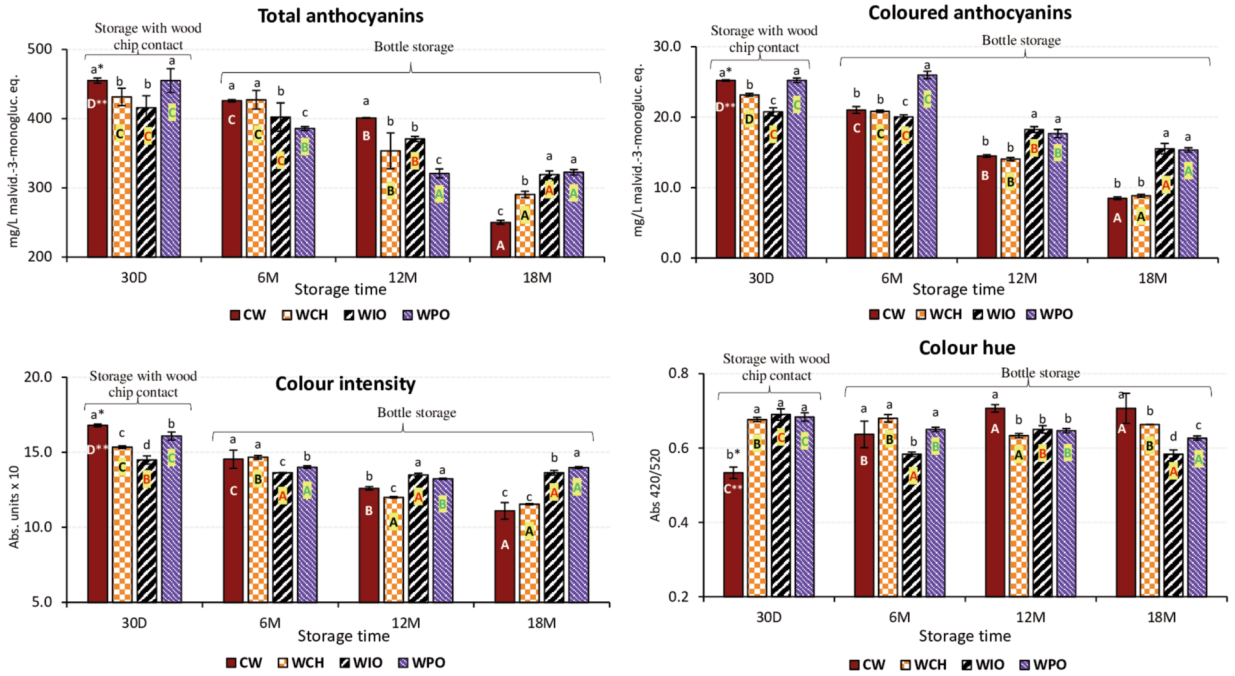



**FIGURE 4.** Total and coloured anthocyanins, colour intensity and hue from red wines after 30 storage days in contact with different wood chip species and during 18 months of bottle storage.
CW - control wine; WCH - wine with cherry wood chip contact; WIO - wine with Iberian wood chip contact; WPO - wine with Pubescent wood chip contact; 30D - after 30 storage days in contact with wood chips; 6M - after 6 months of bottle storage; 12M - after 12 months of bottle storage; 18M - after 18 months of bottle storage; average values with same letter are not significantly different ($p < 0.05$); wherein l letters (* over the bars) are used for wood chips species factor (for each storage time), while capital letters (** inside the bars) are used for storage time evolution factor (for each individual red wine); the error bars represent the standard deviation of the three replicates.


© 2020 International Viticulture and Enology Society - IVES

**TABLE 2.** Individual monomeric anthocyanins from red wines after 30 storage days in contact with different wood chip species and during 18 months of bottle storage.

| Monomeric anthocyanins (mg/L) [(1)] | After 30 storage days with wood chips | | | | After 6 months of bottle storage | | | | After 12 months of bottle storage | | | | After 18 months of bottle storage | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Wines | | | | Wines | | | | Wines | | | | Wines | | | |
| | CW | WCH | WIO | WPO | CW | WCH | WIO | WPO | CW | WCH | WIO | WPO | CW | WCH | WIO | WPO |
| Dp-3-gluc. | 4.32 ±0.04 | 4.23 ±0.05 | 3.01 ±0.19 | 4.21 ±0.29 | 3.59 ±0.02 | 4.10 ±0.05 | 3.54 ±0.22 | 3.21 ±0.40 | 3.01 ±0.22 | 2.10 ±0.03 | 2.33 ±0.32 | 2.34 ±0.30 | 0.55 ±0.02 | 1.10 ±0.02 | 2.54 ±0.20 | 2.21 ±0.10 |
| Cy-3-gluc. | 10.01 ±0.2 | 8.26 ±0.17 | 7.54 ±0.90 | 9.43 ±0.78 | 8.46 ±0.09 | 7.82 ±0.17 | 6.65 ±0.12 | 8.02 ±0.27 | 5.21 ±0.10 | 4.43 ±0.27 | 3.75 ±0.52 | 4.32 ±0.17 | 2.26 ±0.09 | 3.99 ±0.17 | 3.65 ±0.19 | 5.02 ±0.27 |
| Pt-3-gluc. | 18.81 ±1.15 | 15.01 ±0.10 | 10.37 ±0.43 | 14.29 ±1.21 | 14.88 ±0.15 | 13.01 ±0.30 | 11.88 ±0.39 | 13.86 ±1.27 | 10.67 ±0.32 | 7.21 ±0.45 | 8.34 ±1.39 | 5.97 ±1.17 | 4.81 ±0.15 | 3.01 ±0.10 | 9.65 ±0.22 | 5.86 ±0.27 |
| Pn-3-gluc. | 9.35 ±4.14 | 7.10 ±0.11 | 8.98 ±1.12 | 7.79 ±0.45 | 7.50 ±0.14 | 6.36 ±0.11 | 6.33 ±0.51 | 6.57 ±0.85 | 4.50 ±0.56 | 3.36 ±0.21 | 5.21 ±.97 | 3.93 ±0.55 | 2.90 ±0.11 | 3.05 ±0.07 | 4.21 ±0.51 | 3.57 ±0.85 |
| Mv-3-gluc. | 295.0 ±8.1 | 261.60 ±5.7 | 239.21 ±3.3 | 281.32 ±7.2 | 265.30 ±5.0 | 230.30 ±1.7 | 218.80 ±1.1 | 241.80 ±5.2 | 199.31 ±6.01 | 187.21 ±7.72 | 189.21 ±3.11 | 131.20 ±5.21 | 117.21 ±4.09 | 127.30 ±1.7 | 140.10 ±2.1 | 143.80 ±3.2 |
| ∑ Monoglucoside | 337.4 $^{a*}$ ±9.4 $^{D**}$ | 308.7 $^{b*}$ ±6.1 $^{D**}$ | 269.1 $^{c*}$ ±8.9 $^{C**}$ | 317.0 $^{b*}$ ±9.9 $^{D**}$ | 299.0 $^{a*}$ ±5.4 $^{C**}$ | 258.5 $^{b*}$ ±6.4 $^{C**}$ | 269.1 $^{c*}$ ±2.3 $^{C**}$ | 273.4 $^{c*}$ ±7.9 $^{C**}$ | 222.7 $^{a*}$ ±7.2 $^{B**}$ | 204.3 $^{b*}$ ±8.6 $^{B**}$ | 208.8 $^{b*}$ ±8.8 $^{B**}$ | 147.7 $^{c*}$ ±7.4 $^{B**}$ | 127.7 $^{a*}$ ±4.4 $^{A**}$ | 138.4 $^{b*}$ ±2.0 $^{A**}$ | 160.1 $^{c*}$ ±3.2 $^{A**}$ | 160.4 $^{c*}$ ±4.6 $^{A**}$ |
| Cy-3-acetyl. | 10.22 ±0.21 | 8.29 ±1.20 | 10.01 ±1.13 | 12.56 ±1.54 | 9.41 ±0.69 | 9.82 ±0.60 | 9.35 ±0.13 | 10.39 ±0.12 | 6.41 ±0.49 | 5.33 ±0.21 | 6.56 ±0.23 | 4.21 ±0.72 | 1.65 ±0.69 | 1.82 ±0.60 | 3.31 ±0.09 | 4.39 ±0.09 |
| Pt-3-acetyl. | 3.54 ±0.05 | 2.21 ±0.20 | 1.92 ±0.04 | 3.85 ±0.10 | 1.57 ±0.09 | 1.94 ±0.04 | 1.62 ±0.09 | 1.86 ±0.04 | 1.01 ±0.05 | 0.67 ±0.05 | 1.09 ±0.04 | 0.56 ±0.09 | 0.78 ±0.02 | 0.94 ±0.03 | 0.62 ±0.03 | 0.46 ±0.04 |
| Pn-3-acetyl. | 6.18 ±0.11 | 5.03 ±0.07 | 5.42 ±0.02 | 6.95 ±0.07 | 4.48 ±0.04 | 4.54 ±0.02 | 4.42 ±0.02 | 4.86 ±0.03 | 3.12 ±0.08 | 3.10 ±0.06 | 2.98 ±0.04 | 1.86 ±0.02 | 1.48 ±0.04 | 0.99 ±0.02 | 2.42 ±0.02 | 1.97 ±0.02 |
| Dp-3-acetyl. | 2.21 ±0.05 | 1.98 ±0.03 | 1.48 ±0.05 | 2.43 ±0.03 | 1.31 ±0.07 | 1.09 ±0.02 | 1.08 ±0.05 | 1.29 ±0.02 | 0.81 ±0.04 | 0.79 ±0.04 | 0.78 ±0.03 | 0.29 ±0.01 | 0.31 ±0.02 | 0.48 ±0.02 | 0.58 ±0.05 | 0.20 ±0.02 |
| Mv-3-acetyl. | 42.03 ±2.21 | 38.92 ±3.20 | 28.18 ±1.76 | 42.56 ±4.02 | 22.65 ±0.49 | 20.96 ±0.20 | 18.58 ±0.06 | 22.40 ±0.21 | 15.43 ±2.49 | 12.90 ±0.09 | 14.75 ±1.06 | 8.23 ±0.34 | 4.65 ±0.34 | 3.86 ±0.30 | 12.58 ±0.06 | 10.40 ±0.21 |
| ∑ Acetylglucoside | 64.1 $^{a*}$ ±2.6 $^{D**}$ | 56.4 $^{b*}$ ±4.7 $^{D**}$ | 47.0 $^{b*}$ ±3.0 $^{D**}$ | 68.3 $^{a*}$ ±5.7 $^{D**}$ | 39.4 $^{a*}$ ±1.3 $^{C**}$ | 38.3 $^{a*}$ ±0.8 $^{C**}$ | 35.0 $^{b*}$ ±0.3 $^{C**}$ | 40.8 $^{a*}$ ±0.4 $^{C**}$ | 26.8 $^{a*}$ ±3.1 $^{B**}$ | 22.7 $^{b*}$ ±0.4 $^{B**}$ | 26.1 $^{a*}$ ±1.85 $^{B**}$ | 15.1 $^{c*}$ ±1.1 $^{B**}$ | 8.8 $^{a*}$ ±1.1 $^{A**}$ | 8.0 $^{a*}$ ±0.9 $^{A**}$ | 19.5 $^{b*}$ ±0.2 $^{A**}$ | 17.4 $^{c*}$ ±0.3 $^{A,B**}$ |
| Pt-3-p-coum. | 1.92 ±0.03 | 0.98 ±0.04 | 1.21 ±0.31 | 1.35 ±0.03 | 1.12 ±0.03 | 0.92 ±0.04 | 0.98 ±0.11 | 1.05 ±0.03 | 0.34 ±0.02 | 0.32 ±0.01 | 0.38 ±0.01 | 0.25 ±0.02 | 0.12 ±0.03 | 0.12 ±0.02 | 0.14 ±0.04 | 0.15 ±0.03 |
| Pn-3-p-coum. | 2.49 ±0.10 | 2.47 ±0.01 $^{a}$ | 2.10 ±0.04 | 1.94 ±0.02 | 1.75 ±0.10 | 1.47 ±0.01 | 1.39 ±0.04 | 1.64 ±0.02 | 0.76 ±0.10 | 0.47 ±0.03 | 0.79 ±0.05 | 0.63 ±0.02 | 0.20 ±0.10 | 0.22 ±0.02 | 0.40 ±0.04 | 0.44 ±0.03 |
| Mv-3-p-coum. | 18.10 ±1.21 | 15.13 ±0.09 | 13.30 ±0.10 | 17.67 ±0.12 | 14.00 ±0.05 | 12.13 ±0.14 | 10.30 ±0.22 | 13.15 ±0.02 | 10.20 ±0.35 | 8.73 ±0.24 | 9.97 ±0.12 | 7.05 ±0.04 | 3.21 ±0.04 | 4.17 ±0.10 | 4.30 ±0.04 | 4.15 ±0.03 |
| ∑ Coumarylglucoside | 22.5 $^{a*}$ ±1.3 $^{D**}$ | 18.5 $^{b*}$ ±1.0 $^{D**}$ | 16.6 $^{b*}$ ±0.4 $^{C**}$ | 22.3 $^{a*}$ ±1.1 $^{D**}$ | 16.8 $^{a*}$ ±1.1 $^{C**}$ | 14.5 $^{b*}$ ±0.9 $^{C**}$ | 12.6 $^{c*}$ ±0.3 $^{B**}$ | 15.8 $^{ab*}$ ±2.0 $^{C**}$ | 11.3 $^{a*}$ ±0.4 $^{B**}$ | 9.5 $^{b*}$ ±0.5 $^{B**}$ | 11.1 $^{a*}$ ±1.1 $^{B**}$ | 7.9 $^{b*}$ ±2.0 $^{B**}$ | 3.5 $^{a*}$ ±0.1 $^{A**}$ | 4.5 $^{a*}$ ±0.2 $^{A**}$ | 4.8 $^{a*}$ ±0.1 $^{A**}$ | 4.7 $^{a*}$ ±0.2 $^{A**}$ |

CW - control wine; WCH - wine with cherry wood chip contact; WIO - wine with Iberian wood chip contact; WPO - wine with Pubescent wood chip contact; [(1)] individual monomeric anthocyanins expressed as malvidin-3-monoglucoside equivalents; average values with same letter are not significantly different ($p < 0.05$); wherein lowercase letters (*) are used for wood chips species factor (in column for each storage time), while capital letters (**) are used for storage time evolution factor (in line for each individual red wine); ± standard deviation. Dp : delphinidin; Cy - cyanidin; Pt - petunidin; Pn - peonidin; Mv - malvidin; gluc. - monoglucoside; acetyl. - acetylglucoside; coum. - coumarylglucoside.

days in contact with wood chips, the control wine and the wine stored in contact with Pubescent oak chips showed the significantly highest colour intensity values (16.7 and 16.0 abs. units respectively), followed by the wine stored in contact with cherry chips (15.3 abs. units) and the wine stored with Iberian oak chips (14.4 abs. units). During the bottle storage, colour intensity showed a tendency to slightly decrease in all wines. However, this decrease was only statistically different for the control wine. Nevertheless, for wines previously in contact with oak chips, particularly following 12 months of bottle storage, a colour intensity stabilisation was detected. After 18 months of bottle storage, these wines previously in contact with oak chips showed the significantly highest values for colour intensity (values between 13.6 and 13.9 abs. units), followed by the wine previously in contact with cherry chips and the control wine (11.5 and 11.0 abs. units respectively). These results thus clearly exhibit the positive effect of the oak chips used in the study, as well as the potential of the oenological use of *Q. pubescens* wood in red wine aging.

Although there was a tendency for anthocyanin content (including coloured anthocyanins) values to decrease, this fact was not fully reflected in a sharp decrease in colour intensity values during the entire bottle storage period (Figure 4). This is probably due to the increase in absorbance values at 420 nm (one of the components of the colour intensity that quantifies the brown colour) which compensated for the decrease in absorbance values at 520 nm (red). This fact is particularly evident for the control wine, which showed a tendency for a significant increase in colour hue values (32 %) during bottle storage, with significantly higher values after 18 months of bottle storage (0.70). Furthermore, after 18 months of bottle storage, significantly lower colour hue values (0.63 and 0.58 for the wine previously in contact with Pubescent and Iberian oak wood chips respectively) were found for wines previously in contact with oak chips. Intermediate values for colour hue (0.66) were found for the wine previously in contact with cherry chips.

The higher anthocyanin content (total values and coloured forms), and consequently the significantly higher colour intensity values observed after 18 months of bottle storage for the wines previously in contact with oak wood chips, could be explained by the important role of hydrolysable tannins extracted from the oak chips during the prior chip contact in anthocyanin stabilisation and colour protection. In fact, ellagitannins, found mainly in oak wood, as well as in very low levels in cherry wood (Jordão *et al*., 2016), are involved in stabilising anthocyanin structures and demonstrate important antioxidant properties (Vivas and Glories, 1996; Fujieda *et al.,* 2008; Escudero-Gilete *et al.*, 2019). Moreover, ellagitannins have an important role in wine oxidation processes, as they rapidly absorb dissolved oxygen and facilitate the hydroperoxidation of wine constituents, and they also play an important role in the condensation rate of proanthocyanidins and anthocyanins, preventing their degradation and precipitation (Vivas and Glories, 1993; Vivas and Glories, 1996). Moreover, several authors have reported (Monagas *et al*., 2006; Avizcurti *et al.*, 2016) that during bottle storage, red wines have a tendency to increase their colour hue and to develop a brown colour, which is generally explained by a degradation and decrease in anthocyanin content, particularly due to the disappearance of monomeric anthocyanins. This tendency was also confirmed by the evolution of the individual monomeric anthocyanins quantified in the studied red wines during the entire bottle storage period (Table 2).

Thus, as noted for total anthocyanins, a considerable decrease in the different individual monomeric anthocyanins was detected in all red wines during bottle storage (Table 2). However, this decrease was more evident for the control wine and the wine previously in contact with cherry chips in the last two data points (after 12 and 18 months of bottle storage). Nevertheless, all the wines maintained an almost constant anthocyanin profile, with the most abundant group being the group of 3-monoglucosides, followed by the acetylglucoside and coumarylglucoside groups. At the end of the bottle storage period, the wines previously in contact with oak wood chips had the significantly highest content of the 3-monoglucoside group (between 160 and 160 mg/L of malvidin-3-monoglucoside equiv.), followed by the wine previously in contact with cherry wood chips (138.4 mg/L of malvidin-3-monoglucoside equiv.) and the control wine (127 mg/L of malvidin-3-monoglucoside equiv.). However, when the evolution of each wine was analysed individually over the storage period for the three monomeric anthocyanins groups, a


© 2020 International Viticulture and Enology Society - IVES

significant decrease in the values was found for the entire bottle storage time.

It is also important to note that after 18 months of bottle storage, the ratio of total acetylated/coumarylated derivatives was higher for the wines previously in contact with oak chips (ranging between 3.70 and 4.06) than for the control wine (2.51) and the wine previously in contact with cherry chips (1.77). According to several authors (Del Álamo *et al*., 2008; Avizcuri *et al*., 2016) the ratio of acetylated and coumarylated derivatives provides important information about the stability of the red colour. According to Malien-Aubert *et al*. (2001), acylated anthocyanins are very important for wine colour since they participate in intramolecular copigmentation processes, thus increasing the red colour. Therefore, the higher proportion of acetylated anthocyanins compared to coumarylated forms corresponds to a higher red colour stability and a tendency for higher red colour values, as well as for lower colour hue values. This fact could thus help to explain the results shown in Figure 4, in which the highest colour intensity and coloured anthocyanins values and lower values of colour hue can be observed for the wines with the highest ratios of total acetylated/coumarylated derivatives (wines with prior oak wood chip contact) after 18 months of bottle storage in comparison with the control wine and wine with prior cherry wood chip contact. The results obtained also agreed with previous work by Del Álamo *et al*. (2008), who reported a lower decrease in acetylated than non-acetylated anthocyanins in red wines aged in oak barrels. Other authors (Gonçalves and Jordão, 2099; Jordão *et al*., 2019) describe how during wine aging, besides extraction, reactions occur between the wood components and the wine compounds, particularly phenolic compounds (namely anthocyanins), thus leading to a clear decrease in anthocyanins in wines with oak wood contact. In addition, for model wine

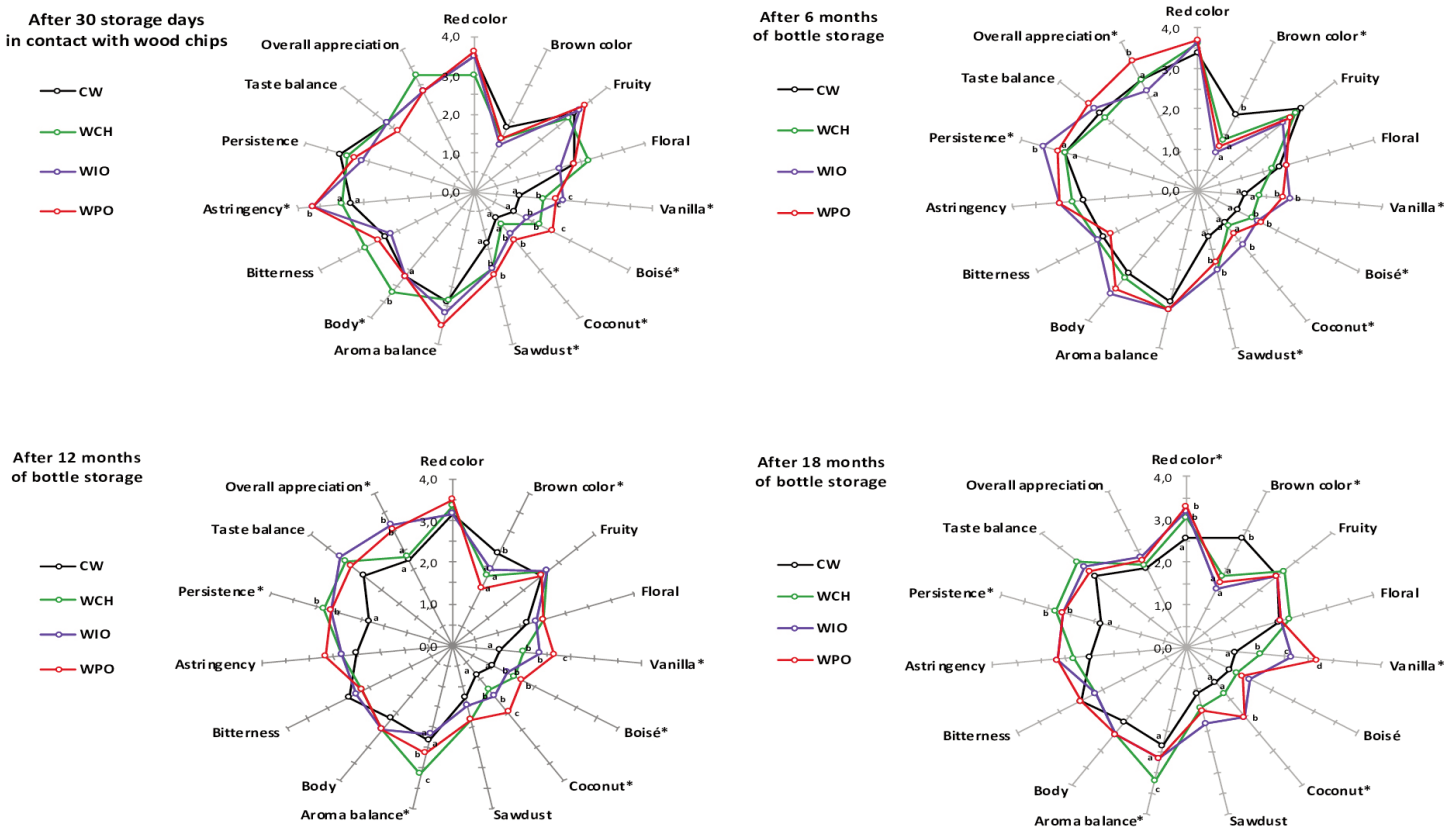



**FIGURE 5.** Sensory profile of red wines after 30 storage days in contact with different wood chip species and their evolution during 18 months of bottle storage.
CW - control wine; WCH - wine with cherry wood chip contact; WIO - wine with Iberian wood chip contact; WPO - wine with Pubescent wood chip contact; * sensory parameters where there are significant differences between the wines and in which values with same letter are not significantly different ($p < 0.05$).

 © 2020 International Viticulture and Enology Society - IVES 

solutions Jordão *et al*. (2008) reported a more pronounced decrease in malvidin-3-monoglucoside in the presence of ellagic acid and oak wood chip extracts.

### 3. Sensory evaluation

Figure 5 shows the spider diagrams of the average values of each descriptor obtained from the sensory analysis of red wines after 30 storage days in contact with wood chips and during bottle storage. Seven wine experts (belonging to a panel of tasters from an official certification organisation, as mentioned previously in the materials and methods section), carried out a sensory evolution of the red wines studied. According to Parr *et al*. (2002), wine experts have a higher recognition memory than wine novices, as well as superior perceptual skills unaffected by verbal interference.

After 30 storage days in contact with the different wood chips, the most marked sensory differences were related to the several aroma descriptors ("sawdust", "coconut", "boisé" and "vanilla") and two taste descriptors ("astringency" and "body"). Thus, red wines in contact with wood chips received significantly higher scores for the two aroma descriptors, "sawdust" and "boisé", than the control wine. Wines kept in contact with oak (*Q. pyrenaica* and *Q. pubescens*) chips received significantly higher scores for "astringency" and the two aroma descriptors, "vanilla" and "coconut". In addition, red wine stored in contact with cherry wood chips obtained the significantly highest scores for "body".

In general, the results obtained for the "astringency" descriptor, showed the same tendency as those obtained for tanning power (Figure 3): red wines previously in contact with wood chips had the highest tannicity values after 30 storage days. According to Chira and Teissedre (2015), wood ellagitannin concentration and other extractable phenolic wood components, particularly from oak wood species, are closely correlated with several wine sensory descriptors, namely "persistency" and "astringency". In addition, the significantly highest scores obtained for "vanilla" and "coconut" aroma descriptors in wines in contact with oak chips is probably a result of *tran*s and *cis*-β-methyl-γ-octalactone extraction from the oak wood chips and transfer to the wines. These two lactones play an important role in wood aroma, giving coconut and vanilla sensory character to wines (Pérez-Prieto *et al*., 2002; De Coninck *et al*., 2006; Guchu *et al*., 2006).

For the panel test, only taste "persistence" and two aroma descriptors, "coconut" and "vanilla", were detected as being statistically different over the entire 18 months of bottle storage. Thus, all red wine samples previously in contact with both types of wood chips obtained significantly higher scores for taste "persistence", while only wines previously in contact with oak wood chips received significantly higher scores for "coconut" and "vanilla" aroma descriptors. Several authors (Fernández de Simón *et al*., 2009; Fernández de Simón *et al*., 2014b; Flamini *et al*., 2007) reported very low amounts of vanillin and lactones in cherry wood which may help to explain the low scores obtained for wine samples previously in contact with cherry wood chips. Only after 18 months of bottle storage did the panel test reveal statistical differences for the "red colour" descriptor, for which all red wines previously in contact with wood chips (oak and cherry) showed significantly higher values than the control wine. In general, these results follow the same tendency observed for total and coloured anthocyanins and colour intensity, with, in particular, significantly higher values being obtained for the red wines previously in contact with oak wood. It was also clear that for the "brown colour" descriptor, the control wine received the significantly highest scores after the entire bottle storage time, which also confirms the results already obtained for quantified colour hue, where the control wine showed the significantly highest values (Figure 4).

In addition, although after 6 and 12 months of bottle storage, wines previously in contact with wood chips (particularly with Pubescent oak chips after 12 months of bottle storage) received significantly higher scores for "overall appreciation", this trend was not observed after 18 months of bottle storage, when all wines obtained similar scores for this descriptor. Finally, the results obtained from the sensory analysis of wine after bottle storage also allowed the wines previously in contact with cherry wood chips to be verified: for the majority of the sensory descriptors analysed, they generally received intermediate scores compared to the control wine and the wines with prior oak wood chip contact. Furthermore, it can also be observed that both the wine previously in contact with Pubescent oak chips and the wine in


© 2020 International Viticulture and Enology Society - IVES

contact with Iberian oak chops received similar scores during bottle storage, particularly for "coconut" and "vanilla" aroma descriptors, as well as for "overall appreciation".

## 4. Principal components analysis applied to wine phenolic and sensory characterisation

To better understand the relationship between the use of different wood chip species, general phenolic parameters and sensorial attributes of red wines, a principal component analysis (PCA) was performed at four time points: after 30 storage days in contact with wood chips, and after 6, 12 and 18 months of bottle storage. The PCA was carried out to obtain a reduced number of linear combinations of the variables that explain the greater variability in the data. Thus, a PCA was calculated on 25 initial variables (phenolic and sensory parameters). The corresponding loading plots that established the relative importance of each variable are shown in Figure 6.

The PCA showed that the first two principal components (PCs) explained 66.34 % of the total variance. The projections of the analysed variables in the PCs are the weighted sum of the original variables and are shown in Figure 6A. The first PC (PC1, 45.40 % of the variance) was positively correlated with all initial variables, except for the "brown colour" sensory attribute. The second PC (PC2, 20.94 % of the variance)

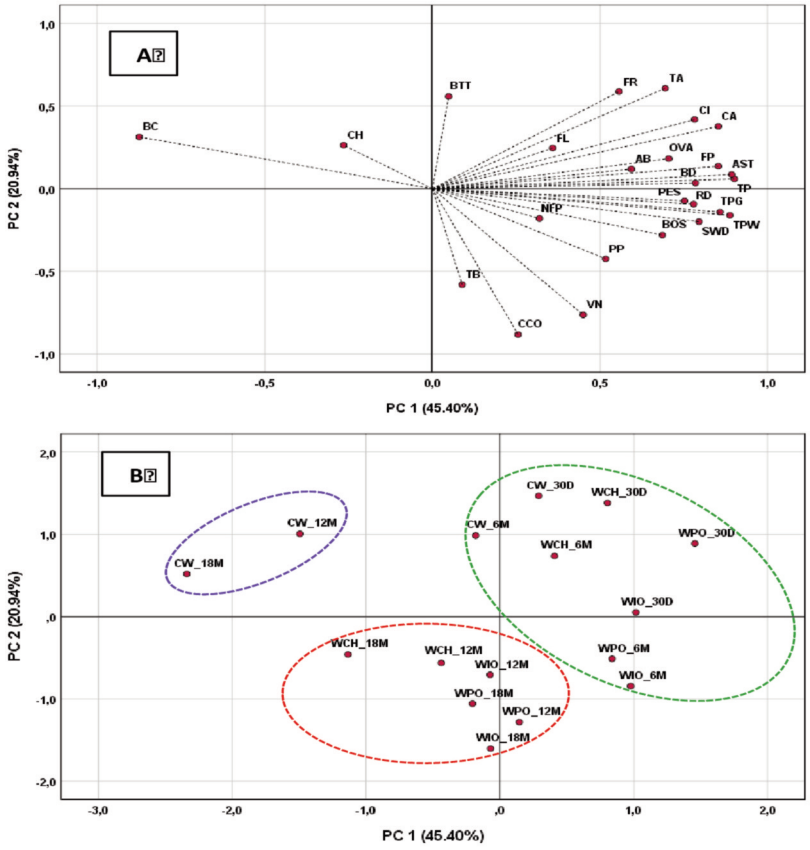



**FIGURE 6.** Principal component analysis (PCA; PC1 and PC2) for different phenolic parameters and several sensorial attributes of red wines after 30 storage days in contact with different wood chip species and during 18 months of bottle storage. (A) Projection of sensorial attributes and phenolic parameters. (B) Projection of red wine samples.
Phenolic parameters: CI - colour intensity; CH - colour hue; TA - total anthocyanins; CA - coloured anthocyanins; TP - total phenols; FP - flavonoid phenols; NFP - non flavonoid phenols; TPG - total pigments; PP - polymeric pigments; TPW - tanning power. Sensorial attributes: RD - red color; BC - brown colour; FR - fruity aroma; FL - floral aroma; VN - vanilla aroma; BOS - boisé aroma; CCO - coconut aroma; SWD - sawdust aroma; AB - aroma balance; BD - body; BTT - bitterness; AST - astringency; PES - taste persistence; TB - taste balance; OVA - overall appreciation. CW - control wine; WCH - wine with cherry wood chip contact; WIO - wine with Iberian wood chip contact; WPO - wine with Pubescent wood chip contact; 30D - after 30 storage days in contact with wood chips; 6M - after 6 months of bottle storage; 12M - after 12 months of bottle storage; 18M - after 18 months of bottle.

 © 2020 International Viticulture and Enology Society - IVES 

was positively correlated with two global phenolic parameters, total anthocyanins and colour intensity, and two sensory attributes ("fruity" and "bitterness"). However, this second PC was negatively correlated with polymeric pigments and with the 3 sensory attributes, "vanilla aroma", "coconut aroma" and "taste balance". Figure 6B gives a spatial distribution of the red wines which had previous wood chip contact in relation to the different parameters considered. After a cluster analysis, three different groups were formed. One group comprises red wines with previous wood chip contact and after 12 and 18 months of bottle storage; these wines are positively related to total pigments and two sensory descriptors associated with aroma, such as, "vanilla" and "coconut". Another group comprises the control wines after 12 and 18 months of bottle storage; these two wines are positively related to the "brown colour" sensory descriptor. Finally, a last third group constitutes all red wine samples analysed after 30 days in contact with the different wood chips (including control wine), as well as all wines after 6 months of bottle storage; these wines are positively related to the majority of the phenolic and sensory parameters studied, except for one phenolic parameter (colour hue) and four sensory attributes ("brown colour", "taste balance", "vanilla" and "coconut" aromas).

All of these results were confirmed by the hierarchical cluster analysis as shown in Figure 7, clearly showing the three distinct clusters comprising the different red wines previously described for Figure 6B.

## CONCLUSIONS

This study revealed a clear influence of the prior contact of wine with wood chips in terms of the evolution of the global phenolic parameters and sensory characteristics of red wine during bottle storage. Compared to red wines previously in contact with cherry chips and the control wine, wines previously in contact with oak chips for 30 storage days showed less decrease in phenolic composition and red colour, as well as a reduction in the development of the brown colour during bottle storage. From a sensory point of view, the wines previously in contact with oak chips also tended to receive higher aroma scores for "vanilla" and "coconut" descriptors, and lower scores for "brown colour", but they showed greater evidence of astringency during bottle storage than wines with prior cherry chip contact and the control wine. However, after 18 months of bottle storage no statistical differences in overall appreciation were found between all the wines. In addition, the wine previously in contact with cherry chips always showed a similar evolution to the control wine. Finally, it is also worth noting that the two oak species used (*Quercus pyrenaica* and *Quercus pubescens*) had similar impacts on red wine characteristics. This is particularly important for oak wood from the *Quercus pubes-*

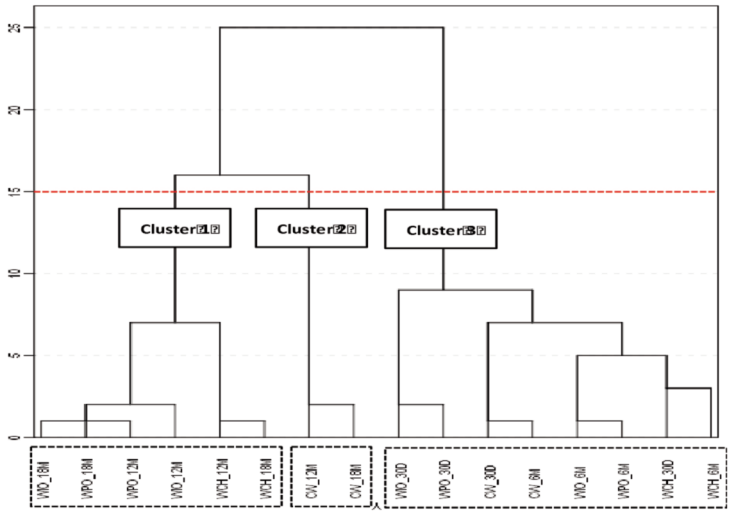



**FIGURE 7.** Diagram with the clusters derived from the hierarchical cluster analysis calculated using the three dimensions of the PCA performed with the red wine samples.
CW - control wine; WCH - wine with cherry wood chip contact; WIO - wine with Iberian wood chip contact; WPO - wine with Pubescent wood chip contact; 30D - after 30 storage days in contact with wood chips; 6M - after 6 months of bottle storage; 12M - after 12 months of bottle storage; 18M - after 18 months of bottle storage.

 © 2020 International Viticulture and Enology Society - IVES 

*cens* species, because currently no information is available on the oenological use of this species.

It can be concluded that the outcomes of our study could be of practical interest to winemakers, allowing them to make better use of different wood chip species and to have a perspective of how red wines can evolve after and during bottle storage. Further research involving more detailed chemical analyses, different wood chip-wine contact times, different wood chip concentrations, and longer bottle wine storage will be necessary to improve our understanding of the potential impact of prior wood chip contact on bottled wine quality.

**Acknowledgements:** The authors wish to thank the AEB Bioquímica Portuguesa company (Portugal) for supplying the cherry wood chips, the J.M. Gonçalves company (Portugal) for supplying *Quercus pyrenaica* oak wood chips, and Barrique Djordjevic Company (Serbia) for supplying *Quercus pubescens* oak wood chips used in this study.

## REFERENCES

Avizcuri, J.M., Sáenz-Navajas, M.P., Echávarri, J.F., Ferreira, V., & Fernández-Zurbano, P. (2016). Evaluation of the impact of initial red wine composition on changes in color and anthocyanin content during bottle storage. *Food Chem.*, 213, 123-134. doi:10.1016/j.foodchem.2016.06.050

Bamber, R.K., & Fukazawa, K. (1985). Sapwood and heartwood: a review. *Forest Prod. Abstr.*, 8, 265-276.

Blanco-Vega, D., Gómez-Alonso, S., & Hermosín-Gutiérrez, I. (2014). Identification, content and distribution of anthocyanins and low molecular weight anthocyanin-derived pigments in Spanish commercial red wines. *Food Chem.*, 158, 449-458. doi:10.1016/j.foodchem.2014.02.154

Bordács, S., Zhelev, P., & Schirone, B. (2019). EUFORGEN Technical guidelines for genetic conservation and use of pubescent oak (*Quercus pubescens*). European Forest Genetic Resources Programme (EUFORGEN), European Forest Institute. 6 pp.

Chira, K., & Teissedre, P.L. (2015). Chemical and sensory evaluation of wine matured in oak barrel: effect of oak species involved and toasting process. *Eur. Food Res. Technol.*, 240, 533-547. doi:10.1007/s00217-014-2352-3

Chinnici, F., Natali, N., Sonni, F., Bellachioma, A., & Riponi, C. (2011). Comparative changes in color features and pigment composition of red wines aged in oak and cherry wood casks. *J. Agric. Food Chem.*, 59, 6575-6682. doi:10.1021/jf2005518

Chinnici, F., Natali, N., Bellachioma, A., Versari, A., & Riponi, C. (2015). Changes in phenolic composition of red wines aged in cherry wood. LWT *Food Sci. Technol.*, 60, 977-984. doi:10.1016/j.lwt.2014.10.029

Clarke, R., & Bakker, J. (2004). Basic taste and stimulant component. Blackwell Publishing (Ed.). Wine flavour Chemistry, pp: 66-119.

Dallas, C., & 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. doi:10.1002/jsfa.2740650416

De Coninck, G., Jordão, A.M., Ricardo-da-Silva, & J.M., Laureano, O. (2006). Evolution of phenolic composition and sensory properties in red wine aged in contact with Portuguese and French oak wood chips. *J. Int. Sci. Vigne Vin*, 40, 25-34. doi:10.20870/oeno-one.2006.40.1.881

De Freitas, V., & Mateus, N. (2001). Structural features of procyanidin interactions with salivary proteins. *J. Agric. Food Chem.*, 49, 940-945. https://doi:10.1021/jf000981z

De Rosso, M., Cancian, D., Panighel, A., Vedova, A.D., & Flamini, R. (2009a). Chemical compounds released from five different woods used to make barrels for aging wines and spirits: volatile compounds and polyphenols. *Wood Sci. Technol.,* 43, 375-385. doi:10.1007/s00226-008-0211-8

De Rosso, M., Panighel, A., Dalla-Vedova, A., Stella, L., & Flamini, R., (2009b). Changes in chemical composition of a red wine aged in acacia, cherry, chestnut, mulberry, and oak wood barrels. *J. Agric. Food Chem*., 57, 1915-1920. https://doi:10.1021/jf803161r

Délia, L., Jordão, A.M., & Ricardo-da-Silva, J.M. (2017). Influence of different wood chips species (oak, acacia and cherry) used in a short period of aging on the quality of 'Encruzado' white wines. *Mitteilungen Klosterneuburg*, 67, 84-96.

Del Álamo, M., Nevares, I., Gallego, L., Martin, C., & Merino, S. (2008). Aging markers from bottled red wine aged with chips, staves and barrels. *Anal. Chim. Acta*, 621, 86-99. doi:10.1016/j.aca.2008.05.014

Del Galdo, V., Correia, A.C., Jordão, A.M., & Ricardo-da-Silva, J.M. (2019). Blends of wood chips from oak and cherry: impact on the general phenolic parameters and sensory profile of a white wine during the aging process. *Vitis*, 58, 159-169.

Dobrei, A., Poiana, M.-A., Sala, F., Ghita, A., & Gergen, I. (2010). Changes in the chromatic properties of red wines from *Vitis vinifera* L. cv. Merlot and Pinot noir during the course of aging in bottle. *J. Food, Agric. Environ.* 8, 20-24. doi:10.1234/4.2010.1553

Escudero-Gilete, M.L., Hernanz, D., Galán-Lorente, C., Heredia, F.J., & Jara-Palacios, M.J. (2019). Potential of cooperage byproducts rich in ellagitannins to improve the antioxidant activity and color expression of red wine anthocyanins. *Foods* 8, 336. doi:10.3390/foods8080336

Fernández de Simón, B., Cadahía, E., & Jalocha, J. (2003a). Volatile compounds in a Spanish red wine aged in barrels made of Spanish, French, and American oak wood. *J. Agric. Food Chem*., 51, 7671-7678. doi:10.1021/jf030287u

Fernández de Simón, B., Hernández, T., Cadahía, E. Dueñas, M., & Estrella, I. (2003b). Phenolic compounds in a Spanish red wine aged in barrels made of Spanish, French and American oak wood. *Eur. Food Res. Technol*., 216, 150-156. doi:10.1007/s00217-002-0637-4

Fernández de Simón, B., Cadahía, E., Sanz, M., Poveda, P., Perez-Magariño, S., Ortega-Heras, & M, González-Huerta, C. (2008). Volatile compounds and sensorial characterization of wines from four Spanish denominations of origin, aged in Spanish Rebollo (*Quercus pyrenaica* Willd.) oak wood barrels. *J. Agric. Food Chem.,* 56, 9046-9055. doi:10.1021/jf8014602

Fernández de Simón, B., Esteruelas, E., Muñoz, À.M., Cadahía, E. & Sanz, M. (2009). Volatile compounds in acacia, chestnut, cherry, ash, and oak woods, with a view to their use in cooperage. *J. Agric. Food Chem*., 57, 3217-3227. doi:10.1021/jf803463h

Fernández de Simón, B., Sanz, M., Cadahia, E., Martinez, J., Esteruelas, E., & Munoz, A.M. (2014a). Polyphenolic compounds as chemical markers of wine ageing in contact with cherry, chestnut, false acacia, ash and oak wood. *Food Chem*., 143, 66-76. doi:10.1016/j.foodchem.2013.07.096

Fernández de Simón, B., Sanz, M., Cadahía, E., Esteruelas, E., & Muñoz, A.M. (2014b). Nontargeted GC-MS approach for volatile profile of toasting in cherry, chestnut, false acacia, and ash wood. *J. Mass Spectrom.*, 49, 353-370. doi:10.1002/jms.3347

Flamini, R., Dalla Vedova, A., Cancian, D., Panighel, A., & De Rosso, M. (2007). GC/MS-positive ion chemical ionization and MS/MS study of volatile benzene compounds in five different woods used in barrel making. *J. Mass Spectrom*., 42, 641-646. doi:10.1002/jms.1193

Fujieda, M., Tanaka, T., Suwa, Y., Koshimizu, S., & Kouno, I. (2008). Isolation and structure of whiskey polyphenols produced by oxidation of oak wood ellagitannins. *J. Agric. Food Chem.*, 56, 7305-7310. doi:10.1021/jf8012713

Fulcrand, H., Dueñas, M., Salas, E., Cheynier, V. (2006). Phenolic reactions during winemaking and aging. *Am. J. Enol. Vitic.*, 57, 289-297.

Gallego, L., Del Álamo, M., Nevares, I., Fernández, J.A., Fernández de Simón, B., & Cadahía, E. (2012). Phenolic compounds and sensorial characterization of wines aged with alternative to barrel products made of Spanish oak wood (*Quercus pyrenaica* Willd.). *Food Sci. Technol. Int.*, 18, 151-165. doi:10.1177/1082013211427782

Gómez-Plaza, E., Gil-Muñoz, R., López-Roca, J.M., Martínez-Cutillas, A., & Fernández-Fernández, J.I. (2002). Maintenance of colour composition of a red wine during storage. Influence of prefermentative practices, maceration time and storage. *LWT - Food Sci. Technol.* 35, 46-53. doi:10.1006/fstl.2001.0809

Gómez-Gallego, M.A., García-Carpintero, E.G., Sánchez-Palomo, E., González Viñas, M.A., & Hermosín-Gutiérrez, I. (2013). Evolution of the phenolic content, chromatic characteristics and sensory properties during bottle storage of red single-cultivar wines from Castilla La Mancha region. *Food Res. Int.* 51, 554-563. https://doi:10.1016/j.foodres.2013.01.010

Giuffrida de Esteban, M.L, Ubeda, C., Heredia, F.J, Catania, A.A, Assof, M.V, Fanzone, M.L., & Jofre, V.P. (2019). Impact of closure type and storage temperature on chemical and sensory composition of Malbec wines (Mendoza, Argentina) during aging in bottle. *Food Res. Int.*, 125, 108553. https://doi:10.1016/j.foodres.2019.108553

Gonçalves, F.J., & Jordão, A.M. (2009). Changes in antioxidant activity and proanthocyanidin fraction of red wine aged in contact with Portuguese (*Quercus pyrenaica* Willd.) and American (*Quercus alba* L.) oak wood chips. *Ital. J. Food Sci.*, 21, 51-64

Guchu, E., Díaz-Maroto, M.C., Pérez-Coello, M.S., González Viñas, M.A., & Cabezudo, M.D. (2006). Volatile composition and sensory characteristics of Chardonnay wines treated with American and Hungarian oak chips. *Food Chem*., 99, 350-359. doi:10.1016/j.foodchem.2005.07.050

ISO 3591 (2016). Sensory analysis-apparatus-wine-tasting glass. Available online: http://www.iso.org/iso/ rss.xlm?csnumber=9002&rss=detail (accessed on 20 November 2017).

Jordão, A.M., Ricardo-da-Silva, J.M., & Laureano, O. (2006). Effect of oak wood constituents and oxygen on the evolution of malvidin-3-glucoside and (+)-catechin in model wine. *Am. J. Enol. Vitic.*, 57, 377-381.

Jordão, A.M., Ricardo-da-Silva, J.M., & Laureano, O. (2007). Ellagitannins from Portuguese oak wood (*Quercus pyrenaica* Willd.) used in cooperage: Influence of geographical origin, coarseness of the grain and toasting level. *Hollforschung*, 61, 155-160. doi:10.1515/HF.2007.028

Jordão, A.M., Ricardo-da-Silva J.M., Laureano, O., Mullen, W., & Crozier, A. (2008). Effect of


© 2020 International Viticulture and Enology Society - IVES

ellagitannins, ellagic acid and some volatile compounds from oak wood on the (+)-catechin, procyanidin B1 and malvidin-3-glucoside content of model wine solutions. *Aust. J. Grape Wine Res.*, 14, 260-270. doi:10.1111/j.1755-0238.2008.00029.x

Jordão, A.M., Correia, A.C., DelCampo, R., & González-SanJosé, M.L. (2012). Antioxidant capacity, scavenger activity and ellagitannins content from commercial oak pieces used in winemaking. *Eur. Food Res. Technol.*, 235, 817-825. doi:10.1007/s00217-012-1803-y

Jordão, A.M., Lozano, V., Correia, A.C., Ortega-Heras, M., & González-SanJosé, M.L. (2016). Comparative analysis of volatile and phenolic composition of alternative wood chips from cherry, acacia and oak for potential use in enology. 39th World Congress of Vine and Wine. *BIO Web of Conferences* 7, 02012. doi:10.1051/bioconf/20160702012

Jordão, A.M., Lozano, V., & González-SanJosé, M.L. (2019). Influence of different wood chip extracts species on color changes and anthocyanin content in synthetic wine solutions. *Foods*, 8, 254. doi:10.3390/foods8070254

Malien-Aubert, C., Dangles, O., & Amiot, M.J. (2001). Color stability of commercial anthocyanin-based extracts in relation to the phenolic composition. Protective effects by Intra- and intermolecular copigmentation. *J. Agric. Food Chem.*, 49, 170-176. doi:10.1021/jf000791

Martínez-Gil, A.M., Cadahía, E., Fernández De Simón, B., Gutiérrez-Gamboa, G., Nevares, I., & Álamo-Sanza, M. (2017). *Quercus Humboldtii* (Colombian oak): Characterization of oak heartwood phenolic composition with respect to traditional oak woods in oenology. *Cienc. Tec. Vitiv.,* 32, 93-101. doi:10.1051/ctv/20173202093

Martínez-Gil, A.M., del Álamo-Sanza, M., Gutiérrez-Gamboa, G., Moreno-Simunovic, Y., & Nevares, I. (2018a). Volatile composition and sensory characteristics of Carménère wines macerating with Colombian (*Quercus Humboldtii*) oak chips compared to wines macerated with American (*Q. alba*) and European (*Q. petraea*) oak chips. *Food Chem.*, 266, 90-100. doi:10.1016/j.foodchem.2018.05.123

Martínez-Gil, A., Del Álamo-Sanza, M., Sánchez-Gómez, R. & Nevares, I. (2018b). Different woods in cooperage for oenology: A review. *Beverages*, 4, 94. doi:10.3390/beverages4040094

Monagas, M., Gómez-Cordovés, C., & Bartolomé, B. (2006). Evolution of the phenolic content of red wines from *Vitis vinifera* L. during ageing in bottle. *Food Chem.*, 95, 405-412. doi:j.foodchem.2005.01.004

Nunes, P., Muxagata, S., Correia, A.C., Nunes, F., Cosme, F., Jordão, A.M. (2017). Effect of oak wood barrel capacity and utilization time on phenolic and sensorial profile evolution of an Encruzado white wine. *J. Sci. Food Agric.*. 97, 4847-4856. doi:10.1002/jsfa.8355

Nunes, I., Correia, A.C., Jordão, A.M., & Ricardo-da-Silva, J.M. (2020). Use of oak and cherry wood chips during alcoholic fermentation and the maturation process of rosé wines: Impact on phenolic composition and sensory profile. *Molecules*, 25, 1236. doi:10.3390/molecules25051236

Kramling, T.E., & Singleton, V.L. (1969). An estimate of the nonflavonoid phenols in wines. *Am. J. Enol. Vitic.*, 20, 86-92.

OIV (2014). International Oenological Codex; Edition Oficielle Organisation International de la Vigne et du Vin: Paris, France.

Parr, W.V., Heatherbell, D., & White, K.G. (2002). Demystifying wine expertise: olfactory threshold, perceptual skill, and semantic memory in expert and novice wine judges. *Chem. Senses*, 27, 747-755. doi:10.1093/chemse/27.8.747

Pérez-Prieto, L.J., López-Roca, J.M., Martínez-Cutillas, A., Pardo-Mínguez, F., & 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. doi:10.1021/jf011505r

Ribéreau-Gayon, P., Glories, Y., Maujean, A., & Dubourdieu, D. (2006). Handbook of Enology. The Chemistry of Wine Stabilization and Treatments (Vol. 2, 2nd ed.). Wiley & Sons Ltd., Chichester, England. https://doi:10.1002/0470010398

Santos, F., Correia, A.C., Ortega-Heras, M., García-Lomillo, J., González-SanJosé, M.L., Jordão, A.M., & Ricardo-da-Silva, J.M. (2019). Acacia, cherry and oak wood chips used on a short aging period of rosé wines: effects on general phenolic parameters, volatile composition and sensory profile. *J. Sci. Food Agric.*, 99, 3588-3603. doi:10.1002/jsfa.9580

Sanz, M., Cadahía, E., Esteruelas, E., Muñoz, M., Fernández de Simón, B., Hernández, & T., Estrella, I. (2010a). Phenolic compounds in cherry (*Prunus avium*) heartwood with a view to their use in cooperage. *J. Agric. Food Chem.*, 58, 4907-491. doi:10.1021/jf100236v

Sanz, M., Cadahía, E., Esteruelas, E., Muñoz, A.M., Fernández de Simón, B., Hernández, T., & Estrella, I. (2010b). Phenolic compounds in chestnut (*Castanea sativa* Mill.) heartwood. Effect of toasting at cooperage. *J. Agric. Food Chem.*, 58, 9631-9640. doi:10.1021/jf102718t

Sanz, M., Fernández de Simón, B., Cadahía, E., Esteruelas, E., Muñoz, A.M., Hernández, T., M., & Estrella, I. (2012a). Polyphenolic profile as a useful tool to identify the wood used in wine aging. *Anal. Chim. Acta,* 732, 33-45. doi:10.1016/j.aca.2011.12.012.

Sanz, M., De Simón, B.F., Cadahía, E., Esteruelas, E., Muñoz, A.M., Hernández, T., Estrella, I., & Pinto, E. (2012b). LC-DAD/ESI-MS/MS study of phenolic compounds in ash (*Fraxinus Excelsior* L. and *F. Americana* L.) heartwood. Effect of toasting intensity at cooperage. *J. Mass Spectrom*., 47, 905-918. doi:10.1002/jms.3040.

Springmann, S., Rogers, R., & Spiecker, H. (2011). Impact of artificial pruning on growth and secondary shoot development of wild cherry (*Prunus avium* L.). *For. Ecol. Manag.*, 261, 764-769. doi:10.1016/j.foreco.2010.12.007.

Somers, T.C., & Evans, M.E. (1977). Spectral evaluation of young red wines: anthocyanin equilibria, total phenolics, free and molecular $SO_2$, chemical age. *J. Sci. Food Agric*., 28, 279-287. https://doi:10.1002/jsfa.2740280311.

Tavares, M., Jordão, A.M., & Ricardo-da-Silva, J.M. (2017). Impact of cherry, acacia and oak chips on red wine phenolic parameters and sensory profile. *OENO One*, 51 (3), 329- 342. doi:10.20870/oeno-one.2017.51.4.1832.

Vivas, N., & Glories, Y. (1993). Les phénomènes d'oxydoréduction liés à l'élevage en barriques des vins rouges: Aspects technologiques. *Rev. Fr. Oenol*., 33, 33-38.

Vivas, N., & Glories, Y., Bourgeois, G., & Vitry, C. (1996). The heartwood ellagitannins of different oaks (*Quercus* Sp.) and chestnut species (*Castanea Sativa* Mill.). Quantity analysis of red wines aging in barrels. *J. Sci. Tech. Tonnelerie*, 2, 25-75.

Vivas, N., & Glories, Y. (1996). Role of oak wood ellagitannins in the oxidation process of red wines during aging. *Am. J. Enol. Vitic.*, 47, 103-107.

Vivas, N. (2005). Manual De Tonelería: Destinado A Usuarios De Toneles; Mundi, Pre: Madrid, Spain.

Zhang, B., Cai, J., Duan, C.Q., Reeves, M.J., & He, F. (2015). A review of polyphenolics in oak woods. *Int. J. Mol. Sci.*, 16, 6978-7014. doi:10.3390/ijms1 6046978


© 2020 International Viticulture and Enology Society - IVES