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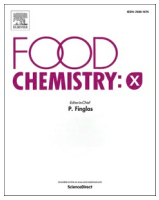

Role of ellagitannins and OTR of the *Quercus petraea* (Matt.) Liebl barrel staves for beverages aging☆

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ABSTRACT

American and French oak are woods commonly used in cooperage to make barrels. This study analyzed 250 French oak staves to determine the relationship between ellagitannin content and oxygen consumption capacity, as well as the importance of wood grain and its oxygen transfer rate. The study found that castalagin is the most abundant ellagitannin, followed by vescalagin in 37 % of the staves and roburin E in 46 % of them. This allows for the creation of ellagitannin content profiles. It was observed that vescalagin and castalagin occupied the top positions in 7 % of the samples. Additionally, the woods with the highest oxygen consumption capacity were found to have castalagin or vescalagin as the main ellagitannins. Samples with castalagin and roburin E as the main ellagitannins had the lowest consumption capacity. Seven percent of the samples had a castalagin and roburin D profile, which was found to be intermediate.

1. Introduction

Oak wood is a natural material with highly favorable properties for barrel manufacturing, particularly for aging wines and spirits del Alamo, Bernal, del Nozal and Gómez-Cordovés, 2000. It enables the production of barrels that contribute distinct compounds and varying rates of oxygenation to the aging beverages (del Alamo-Sanza & Nevares, 2014; Garcia-Moreno et al., 2021). When the barrel is filled with wine, it soaks into the wood and accesses the compounds that are subsequently transferred to the wine or beverage, modifying its chemical and sensory characteristics. In addition to volatile compounds (Chatonnet et al., 2010; Sánchez-Gómez et al., 2020), tannins, bioactive compounds that modify the antioxidant properties of wine, are also incorporated into beverages (Alañón et al., 2011; Canas, 2017; Fernández de Simón et al., 1999; García-Estévez et al., 2017; Martínez-Gil et al., 2020; Navarro et al., 2017; Watrelot & Waterhouse, 2018). The transfer of the different compounds depends not only on the type of wood chosen for each barrel, but also on the grain of the wood and the treatments carried out in the cooperage, especially the toasting profile. During the toasting of the wood, the cell structures are broken down (Hale et al., 1999), which could favor the extraction of compounds from the wood into the wine. Therefore, in order to draw conclusions based on the characteristics of the wood of the staves, it is important to have information on the treatments it has undergone (Garcia-Moreno et al., 2021; Jordão et al., 2007; Martínez-Gil et al., 2020, 2022; Watrelot & Waterhouse, 2018).

Tannins are compounds found in wine that can also be used as additives to stabilize coloring matter during the fermentation process. Depending on the nature of their monomeric units, they are classified into two main groups: hydrolyzable tannins and condensed or proanthocyanidin tannins. The flavan-3-ol units, which form the basis of condensed tannins, can vary in their stereochemical configuration, degree of hydroxylation, and whether they are esterified with gallic acid. Given the wide variety of monomeric units, interflavanic bonds, and degrees of polymerization, the structure of proanthocyanidins is highly variable and, therefore, so is their reactivity. Hydrolyzable tannins are divided into gallotannins and ellagitannins. Gallotannins or gallic

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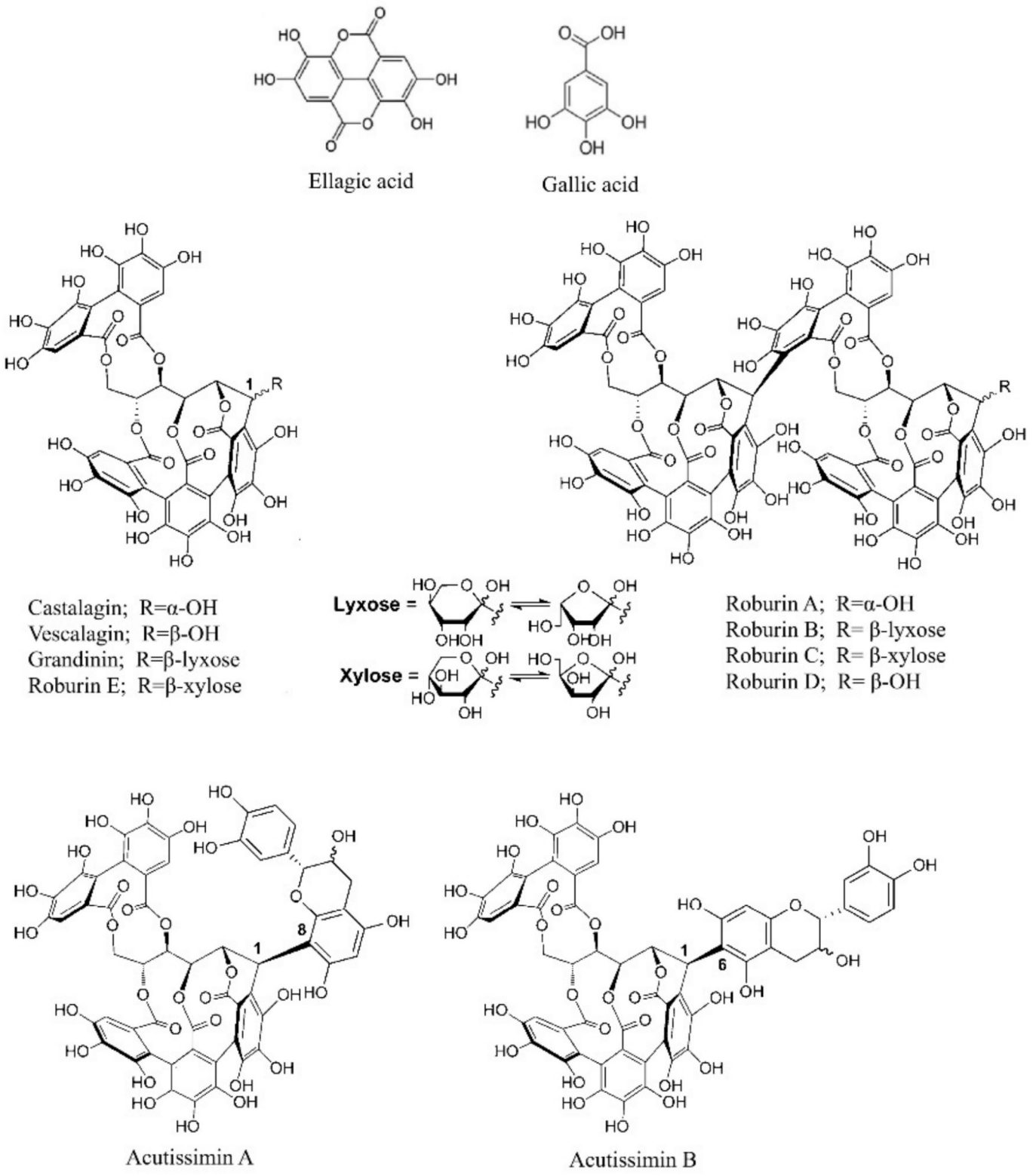


Fig. 1. Structural formulas of main ellagitannins and their precursor phenolic acids. Figure adapted from Jourdes et al. (2011).

tannins are formed by glucose or quinic acid units esterified with one or more molecules of gallic acid or its depsides. Ellagitannins, or ellagic tannins, are formed by glucose units esterified with one or more molecules of ellagic acid or similar compounds. Tannins exhibit a range of properties due to their interaction with salivary proteins, which affects the sensory characteristics of wine. They also possess antioxidant activity, reducing the amount of SO_2 required to preserve the wine. In addition, tannins form chelates with iron, lowering its concentration in wine, and participate in polymerization and complexation processes that contribute to color stabilization (Watrelot & Waterhouse, 2018). Oak wood yields castalagin and vescalagin, the C-lyxoside and C-xyloside conjugates of vescalagin (grandinin and roburin E, respectively),

together with C-glycosidic derivatives such as the quasi-dimers of all the latter compounds (roburins A, D, B, and C, respectively). In addition, new ellagitannins can be formed in wine through reactions between oak ellagitannins and wine components, such as vescalagin adducts with (+)-catechin (acutissimins A and B), ethanol, and even malvidin-3-glucoside (Jordão et al., 2007). The structural formulas of these main ellagitannins and their precursor phenolic acids are presented in Fig. 1. Similar compounds have also been identified in aged spirits and Cognac, including the so-called brandy tannin B, which has been reported to affect the sensory properties of Cognac eau-de-vie (Gadrat et al., 2022). Ellagitannins are oxidizable compounds that degrade when solubilized in a liquid medium, such as in an ethanol:water solution (García-Estévez et al., 2017). In other words, when the barrel is filled with a beverage, part of that beverage penetrates the wood, solubilizing the ellagitannins, which degrade to a greater or lesser extent depending on their structure. These authors have pointed out the indirect contribution of oxygen in the transformation of ellagitannins by enhancing autoxidative reactions, the highest affinity for oxygen, being the most oxidation-prone, showing a marked tendency to undergo oxidation. Subsequently, during aging, the tannins are transferred to the beverage contained in the barrel, and their study has been the subject of various studies in both wines and spirits (Gadrat et al., 2021, 2022). Thus, there are studies reporting total ellagitannin concentrations in wine spirits ranging from 6 to 20 mg mg $\times$ L^{-1} in gallic acid equivalents (Canas, 2017), in Cognac "eaux-de-vie" between 2 and 9 mg $\times$ L^{-1} in vescalagin equivalents (Gadrat et al., 2021) or up to 127 mg $\times$ L^{-1} (Viriot et al., 1993), with variations depending on the type of barrel used. In red wines, total ellagitannin levels vary from 6 mg $\times$ L^{-1} to 120 mg $\times$ L^{-1} (eq. ellagic acid), with higher values in barrels made from French oak than American oak (Watrelot & Waterhouse, 2018).

Furthermore, the oxygen that aged beverages receive in barrels plays an essential role in the aging process due to its role in the reactions between the compounds released by the wood and the compounds in the wine. For this reason, various studies have evaluated the oxygenation rate of different barrels. The oxygenation rate that wine receives during barrel aging depends on the macrostructure of the wood, cooperage practices (including drying, assembly, and toasting), and the aging process itself, which is determined by the interaction between the wine and the barrel (del Alamo-Sanza and Nevares, 2014, 2017; Nevares et al., 2019; Renouf et al., 2016). These results highlight the importance of both the ellagitannin composition of the wood and its oxygenation rate in the evolution of wine.

This study analyzed a collection of 250 staves considered representative of French oak wood *Quercus petraea* (Matt.) Liebl. used in cooperage. This paper reports a multiple analysis of a remarkably large number of *Q. petraea* wood samples, including wood anatomical parameters, iron and ellagitannin contents, total polyphenol index, or oxygen consumption parameters and oxygen transfer rate. All this allows to know the interactions between the evaluated components and thus assess the potential of French oak wood for cooperage.

2. Materials and methods

2.1. Oak wood

The study included 250 staves of French oak, *Quercus petraea* (Matt.) Liebl. (*Q. petraea*), without toasting, subjected to a natural drying process of 18 months at the cooperage wood yard, and supplied by Intona Cooperage (Navarra, Spain). The staves come from different forests in France: Vosges, Tronçais and Bellême forest, measure between 97 and 99 cm. Grain has been calculated as mean value of the width of each ring (mm $\times$ $ring^{-1}$). The density of the different wood pieces was estimated and expressed as the ratio of the oven-dry weight of wood to its volume, with an analytical scale KERN AEJ Version 2.1 (KERN & Sohn GmbH, Balingen, Germany) using the density estimation of KERN YDB-03 Version 1.1. (Bowman & Schoonover, 1967) expressed as g $\times$ cm^{-3} (Bowman & Schoonover, 1967; Nevares et al., 2019).

2.2. Wood oxygen transfer rate measurement

The oxygen transmission rate (OTR) of the oak woods was analyzed using a patented device according to del Alamo-Sanza and Nevares (2012). Therefore, it was necessary to extract a portion of oak wood from each stave. Each wood piece underwent a desorption process consisting of the removal of air contained within its structure. This process lasted seven days and was carried out prior to the determination of its oxygen transmission rate. For the measurement, the wood pieces were maintained in contact with model wine (12.5 % ethanol, pH of 3.5), reproducing similar aging conditions to those found in barrels. A device extensively described in previous studies was used to determine the OTR of the different materials in contact with gas, liquids, or both (Maioli et al., 2024; Nevares & del Alamo-Sanza, 2021). A total of 250 OTR tests were carried out on wood samples.

2.3. Measurement of total phenols, iron, spectrum and capacity to consume oxygen

To determine the total phenolic content, iron concentration, UV–Vis spectrum, and oxygen consumption capacity of the wood, an extraction procedure was required. To facilitate extraction and obtain a representative sample from each stave, a piece of wood was taken from a location close to that used for OTR measurement. The wood sample was then ground into chips to allow for effective extraction of the target compounds. The chips from each piece were homogenized and stored in a sealed bag under controlled temperature and relative humidity conditions (barrel room, 72 $\pm$ 3 % RH, 16 $\pm$ 0.5 °C) until analysis. For extraction, 3 g of wood was added to 25 mL of a methanol-water solution (15,85, %*v*/v), sonicated for 15 min, centrifuged at 4000 rpm at 0 °C for 15 min, and the supernatant was filtered before analysis. The total polyphenol index at 280 nm (TPI) was determined in the wood extract diluted 1:10 using a 1 mm quartz cuvette. For both spectral scanning (300 nm to 500 nm) and absorbance measurement at 420 nm, the undiluted extract was used, employing 10 mm thick quartz cuvettes in both cases. All measurements were performed on a Perkin Elmer LAMBDA 25 UV/ViS spectrophotometer (Waltham, MA, USA). Fe^{+2}, Fe^{+3} ions present in wood extracts can interact with other components, modifying oxygen consumption. Thus, ellagitannins can interact with Fe^{+2} ions, which after being chelated are relatively quickly oxidized to Fe^{+3} ions by the oxygen present. Subsequently, these ions can oxidize the ellagitannins, reducing them back to the Fe^{+2} form. These ions Fe^{2+} and Fe^{3+} were measured in the wood extract using the colorimetric method that exploits the formation of the Fe^{3+} complex with thiocyanate, measurable at 508 nm (Zoecklein et al., 1995). No Fe^{3+} levels were found in the prepared wood extracts, so the results obtained from the evaluation of Fe^{2+} in the prepared wood extracts are presented. Quantification Fe^{2+} with an external standard allowed the results to be expressed as µg $\times$ g^{-1} of Fe^{3+} present in the extract prepared from each wood. All measurements were performed in duplicate.

The oxygen consumption capacity of the wood extract was evaluated following the method described in previous studies (Nevares et al., 2017; del Alamo-Sanza et al., 2021). Briefly, the wood extract was tempered to 35 °C and saturated with air at 35 °C for 5 min, reaching a partial oxygen pressure level between 135 and 235 hPa. The saturated extract was transferred to 3 mL glass vials hermetically sealed with precision valve screw caps (Restek Innovative Chromatography Products, Bellefonte, USA) and equipped with a dissolved oxygen sensor to monitor oxygen consumption. The oxygen consumption kinetics of each extract were performed in quadruplicate, i.e., a total of 1000 kinetics were performed (250 extracts $\times$ 4 repetitions), collecting the dissolved oxygen measurement every 15 min. The evolution of dissolved oxygen (DO) was measured using a SensorDish SDR multi-reader device (PreSens Precision Sensing GmbH, Regensburg, Germany) equipped with 98

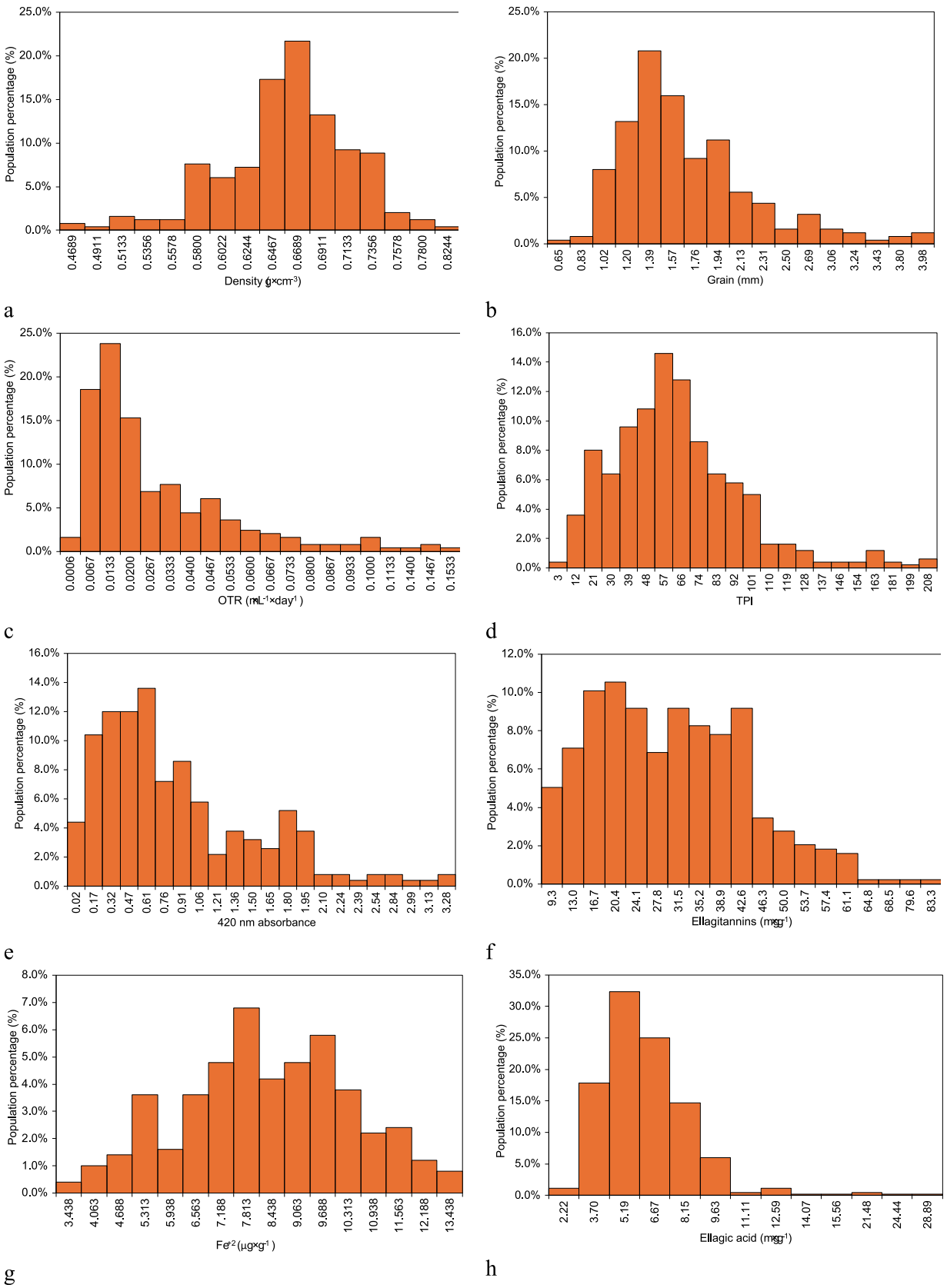


Fig. 2. Distribution of relevant parameters of the wood samples analyzed: (a) density; (b) grain (mm); (c) oxygen transfer rate (OTR, mg × $-L^{1-}$ × day^{-1}); (d) total phenolic index (TPI); (e) absorbance 420 nm; (f) total ellagitannin (mg × L^{-1}); (g) $Fe^{2}+$ (µg × g^{-1}); (h) ellagic acid (mg × L^{-1}) and (i) gallic acid (mg × L^{-1}).

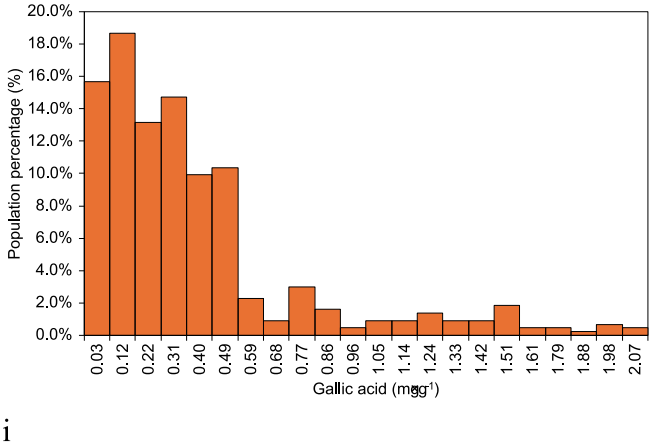


Fig. 2. (*continued*).

vials. Each vial has an SV-PSt5 isolated optical oxygen sensor (PreSens Precision Sensing GmbH, Regensburg, Germany) integrated into the bottom. The oxygen sensors in each vial were calibrated according to the manufacturer's protocol, taking into account the measurement temperature of 16 ± 0.2 °C, with measurements taken at two calibration points: oxygen-free water at a concentration of 0 mg × L^{-1} (0 % air saturation) and saturated air (100 % air saturation). The 1000 kinetics were processed following the method established in previous studies (del Alamo-Sanza et al., 2021), obtaining the characteristic parameters of the curves that describe each oxygen consumption kinetics. The consumption curve data analyzed were those found from the maximum oxygen level reached by each extract to the minimum value, considering the minimum oxygen value found after taking three stable measurements. This time point was considered the end of the oxygen consumption kinetics. Subsequently, the replicates of the same sample were combined to obtain two representative curves: one representing the data obtained from the mean minus the standard deviation and another curve representing the data obtained from the mean plus the standard deviation of the four repetitions performed on each sample. The parameters selected to define the oxygen consumption kinetics were maximum or initial oxygen (hPa) = O_{max}; minimum or residual oxygen (hPa) = O_{min}; total oxygen consumed (hPa) = ΔO_{max_min}; time taken to consume all oxygen (h) = tO_{min}; and oxygen consumption rate (hPa × min^{-1} = V_{con}). In some cases, oxygen measurements are converted to mg × L^{-1} for better understanding, taking into account atmospheric pressure and assuming that the solubility of oxygen in beverages is the same as that in water, although for the study of consumption kinetics it is considered more appropriate to express the results in hPa.

2.4. Ellagitannins analysis

Sample preparation: pieces of wood adjacent to the pieces extracted for OTR analysis were removed. These pieces of wood were ground and sieved using an ultracentrifugal mill Retsch ZM 200 (Retsch GmbH, Haan, Germany), obtaining sawdust of a particle size <1 mm. All samples were stored until extraction under environmental conditions of 16 °C temperature and 75 % relative humidity. Ellagitannins were extracted according to Viriot et al. (Viriot et al., 1994). Briefly, the sawdust from each stave (100 mg) was mixed with 5 mL of a water/acetone solution (30:70, %*v*/v) previously sparged with nitrogen. Extraction was carried out with constant stirring for 180 min. The extracts were centrifuged at 4000 rpm for 10 min at 0 °C, the supernatant was separated and filtered through 0.22 μm hydrophilic PVDF syringe filters. The filtered extracts were concentrated in a N_2 atmosphere and dissolved in 1 mL ultrapure water. All extractions were carried out in duplicate for each stave sample.

Ellagitanins were analyzed according to Navarro et al. (Navarro et al., 2017) method with minor modifications. HPLC separation, identification and quantification of ellagitannins were performed on an Agilent 1100 Series system equipped with DAD detector (Agilent, Waldbronn, Germany). The chromatographic separation was performed on a fused-core, small particle size column (Ascentis Express C18, 150 × 4.6 mm, 2.7 μm particle size) thermostated at 40 °C. The solvent system consisted of solvent A (water/formic acid, 996:4, (‰ v/v) and solvent B (methanol/formic acid, 996:4, v/v). The flow was changed during the elution gradient: 0 min, 0 % B, flow of 0.3 mL/min; 7.5 min, 0 % B, flow of 0.30 mL/min; 8.0 min, 0 % B, flow of 0.55 mL/min; 25 min, 20 % B, flow of 0.70 mL/min; 35 min, 50 % B, flow of 0.70 mL/min; 37 min, 100 % B, flow of 0.70 mL/min; 48 min, 100 % B, flow of 0.70 mL/min; 55 min, 0 % B, flow of 0.55 mL/min; post-run time, 10 min at 0 % B and flow of 0.30 mL/min. The ellagitannins were identified by matching the retention time and spectral data (DAD-UV–vis and MS/MS). The ellagitannins roburin A, roburin B, roburin C, roburin D, roburin E, vescalagin, castalagin, acutissimin A and acutissimin B quantification was performed using the DAD-chromatograms extracted at 235 nm as ellagic acid, gallic acid and grandinin at 280 nm and ellagic acid at 250 nm, and all are expressed as mg of ellagic acid per g of dry wood. Quantification was conducted through the external standard method based on its calibration curve at seven different concentrations using an UV–visible signal (R^2 = 0.99). All analyses were completed in duplicate. The ellagitannins identification was performed using a QTOF orthogonal mass 203 spectrometer (QTOF) equipped with electrospray ionization source (ESI), UPLC 2D-UHPLC 204 Exion Sciex series and X500R model Sciex by QTOF. The chromatographic method and column used were the same, therefore the elution order is expected to remain consistent. The molecular formula of ellagitannins were calculated on the basis of accurate mass of [M-H] − ion and the isotopic pattern The identifications were confirmed by performing MS/MS experiments. In general, the fragmentations confirmed the structures proposed: Two fragments with nominal masses 631 and 613 Da were observed in the MS/MS spectra of vescalagin, and castalagin. The total ion chromatogram also showed signals at *m/z* 924.059 and *m/z* 990.080. They correspond to the m/z signals of [M-2H]2− ions of roburin A/D ($C_{82}H_{50}O_{51}$, MW 1850.132) and roburin B/C ($C_{87}H_{58}O_{55}$, 1982.174 Da), respectively. Other signals at 1065,1046 correspond to roburin E and grandinin ($C_{46}H_{34}O_{30}$). Acutissimin A and B shows very low signals but a fragment of 915 was shown.

2.5. Chemicals

Methanol, ethanol, acetone and formic acid HPLC grade were supplied by Scharlab, SL (Spain). Ellagic and gallic acids were purchased from Sigma-Aldrich (Steinheim, Germany). Potassium thiocyanate (99 % ACS reagent), tartaric acid (ACS reagent), hydrochloric acid (37 %

ACS reagent) and iron (II) chloride 4-hydrate pure were obtained from Panreac (Barcelona, Spain). Deionized water was purified with a Milli-Q system (Millipore, Bedford, MA, USA).

2.6. Statistical analysis

The results of the normal distribution analysis indicate that the population of wood samples studied does not follow a normal distribution and therefore the correlations between variables were obtained using Spearman's correlation test. To assess whether there are significant differences between the parameters studied, a Kruskal-Wallis test was performed. Statistical significance levels of $p \leq 0.05$ were considered significant. Statistical analyses were performed using the Statgraphics Centurion statistical program (version 19.4.02; StatPoint, Inc., The Plains, VA, USA).

3. Results and discussion

3.1. General wood parameters

Fig. 2 shows the distribution in intervals of each parameter studied in the 250 staves of *Q. petraea*. These results are particularly relevant considering that French oak is one of the most widely used woods in cooperage, especially for wine aging. The characteristics of this oak have been studied by various researchers who have evaluated the effect of cooperage treatment of French oak from different geographical areas on the volatile and tannic profile imparted by the barrels (Canas, 2017; Fernández de Simón et al., 1999). During the aging of beverages in barrels, the various antioxidant compounds in the wood, such as ellagitannins, are released and react with the components of the beverages and with the oxygen that enters the barrel (Alañón et al., 2011; Fernández de Simón et al., 1999; Jordão et al., 2007). However, establishing the profile of the content of these compounds is not easy, as it varies according to the properties of the wood, which is a natural material with high inter- and intra-stave variability, this means that the results obtained in this work from a large number of samples can be considered representative.

Most staves (90 %) have a density between 0.580 g $\times$ cm^{-3} and 0.736 g $\times$ cm^{-3}, although 5.2 % of staves have been found with a density below 0.560 g $\times$ cm^{-3} and 3.6 % of samples have a density above 0.750 g $\times$ cm^{-3} (Fig. 2a). Traditionally, wood has been classified according to its grain, defined as the width of the annual growth ring. The French oak woods studied have a grain that varies between 0.65 and 3.98 mm, although most of the samples, 88 %, have a grain between 1.0 and 2.3 mm (Fig. 2b). According to the Pracomtal classification (Guillaume de Pracomtal et al., 2014), there are no large-type (Lc > 5 mm) or wide-grain (4 $\leq$ Lc $\leq$ 5 mm) woods. Twenty-three percent of the population would be average grain (2 $\leq$ Lc $\leq$ 4 mm), 74 % of the population would be tight grain (1 $\leq$ Lc $\leq$ 2 mm) and 3 % of the population would be very tight grain (Lc < 1 mm). Therefore, the population studied corresponds to the three categories of the Vivas classification (Vivas, 1995).

Another property of wood is its ability to transfer oxygen to beverages aging in barrels. The OTR of the woods analyzed in this study ranged from 0.0006 to 0.1533 mg $\times$ L^{-1} $\times$ day^{-1}, although most of the staves (84 %) in the batch studied have an OTR below 0.05 mg $\times$ L^{-1} $\times$ day^{-1} (Fig. 2c). The average of all the samples evaluated is 0.034 mg $\times$ L^{-1} $\times$ day^{-1} with a coefficient of variation of 96 %, which reflects a very high variability, indicating that the selection of wood is essential to ensure that the barrels constructed are homogeneous in relation to the oxygen they transfer to the wines aging in them. The work of (Prat-García et al., 2020) demonstrates that it is possible to classify wood according to its OTR and subsequently build barrels with different oxygenation rates. This classification allows barrels with homogeneous OTR to be obtained, thus reducing the high variability in the behavior of the barrels.

Some substances in wood, such as ellagitannins, low molecular weight compounds, volatile compounds, mineral composition, among others, can be transferred to beverages during aging in oak barrels, thus modifying the chemical and sensory characteristics of the aged beverage (Martínez-Gil et al., 2022). The concentration of these compounds in beverages depends on multiple factors related to oak wood. It is essential to know the level of extractable compounds that a single species can contribute, especially before the toasting process, as this is the most variable factor between different cooperages. Wood has a mineral content that reflects its location, natural environment, and soil composition, factors that affect both tree growth and wood quality. In addition, the toasting carried out in cooperage can also affect its content, however, no studies have been found that demonstrate variations in mineral content due to the toasting process. The Fe^{2+} and Fe^{3+} content of the French oak wood studied was assessed, all of which underwent the same natural air-drying process. This treatment, similar in duration and conditions to those commonly applied in different cooperages, guarantees the representativeness of the material in terms of this aspect of pre-toasting treatment. Fe^{2+} is the most abundant ion in the wood extracts studied. Most of the staves (74 %) show a Fe^{2+} concentration in the range of 0.4–0.7 μg $\times$ g^{-1}, although 10 % of individuals with higher values, between 0.8 and 1 μg $\times$ g^{-1} Fe^{2+} were also found and lower values, between 0.2 and 0.3 μg $\times$ g^{-1} Fe^{2+}, were found in the 16 % of the wood extracts (Fig. 2g). These soluble mineral substances can act as catalysts in the redox mechanism, leading to profound changes in the quality and composition of aged beverages (Vivas et al., 2022). In addition, minerals interact with the water-soluble phenolic fraction, which could adsorb and chelate cations. Therefore, in some cases, barrel aging causes a decrease in the content of metal cations. Therefore, wood can release but also retain mineral elements, and the balance between these two phenomena contributes to the variation in the concentration of these elements in aged beverages.

One of the most important groups of compounds in beverage aging are phenolic compounds. Fig. 2 (f, h, i) shows the overall parameters associated with their concentration in wood extracts. Regarding the ability to release substances, the phenolic compound content of the extracts, reflected in the total phenol index (TPI), showed values between 3 and 208. Approximately 70 % of the extracts had values within the range of 21 to 80 (Fig. 2d), which shows a wide range of concentration and a notable heterogeneity in the transfer capacity of phenolic compounds between the different staves analyzed. The absorbance of the extracts at 420 nm showed values mostly between 0.2 and 1.0, a range in which approximately 65 % of the samples were found. However, 35 % of the extracts had higher values, in the range of 1.21 to 3.30, indicating a high release of phenolic compounds by some staves under aging conditions. These results correlate positively with the total polyphenol index (TPI), reinforcing the usefulness of absorbance at 420 nm as a complementary indicator of overall phenolic content (Fig. 2e). With regard to the ellagitannin content of the samples analyzed, high variability was observed, with values ranging from 9 to 83 mg $\times$ g^{-1}, although most staves had concentrations between 13 and 43 mg $\times$ g^{-1} (Fig. 2f). As expected for this oak species, ellagic acid concentrations were significantly higher than those of gallic acid, in line with previous studies on *Q. petraea*. Specifically, the extracts showed ellagic acid contents between 2.0 and 29.0 mg $\times$ g^{-1}, with more than 75 % of the samples in the range of 3.7 to 6.67 mg $\times$ g^{-1} (Fig. 2h). These values are somewhat higher than those reported in the few references available for naturally air-dried *Q. petraea* wood (Martínez-Gil et al., 2020). The gallic acid content was considerably lower, ranging from 0.03 to 2.07 mg $\times$ g^{-1}, and more than 85 % of the staves analyzed contained between 0.03 and 0.49 mg $\times$ g^{-1} (Fig. 2i), showing, as with all the compounds studied, the wide variability between the different staves. Due to this high variability, in recent years, and to classify wood for barrel construction, an online system has been developed to estimate the total concentration of phenols, mainly ellagitannins (Oakscan, Radoux, France). This classification system was developed to reduce the variability in the content of these compounds in wood selected for barrel construction, relating

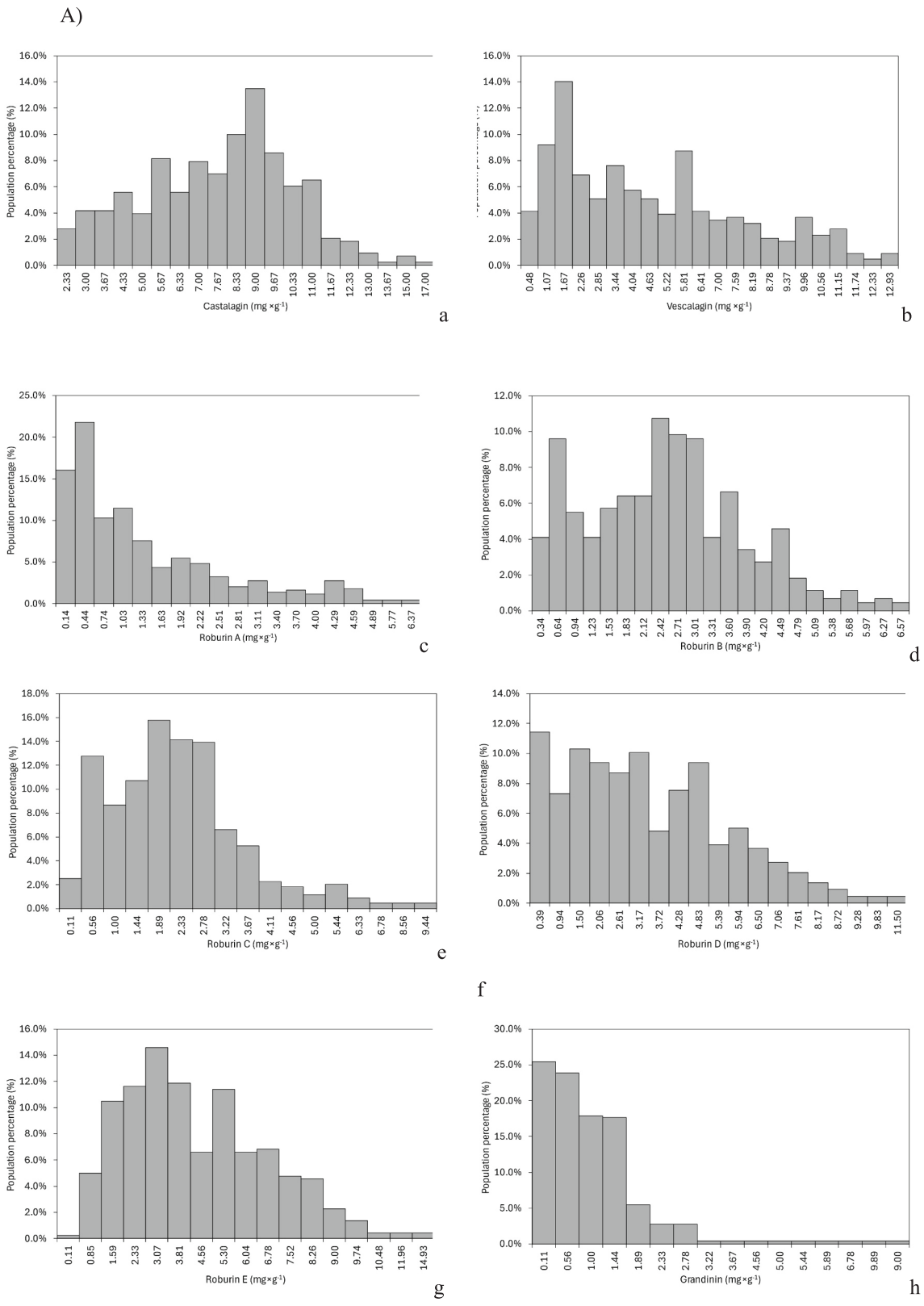
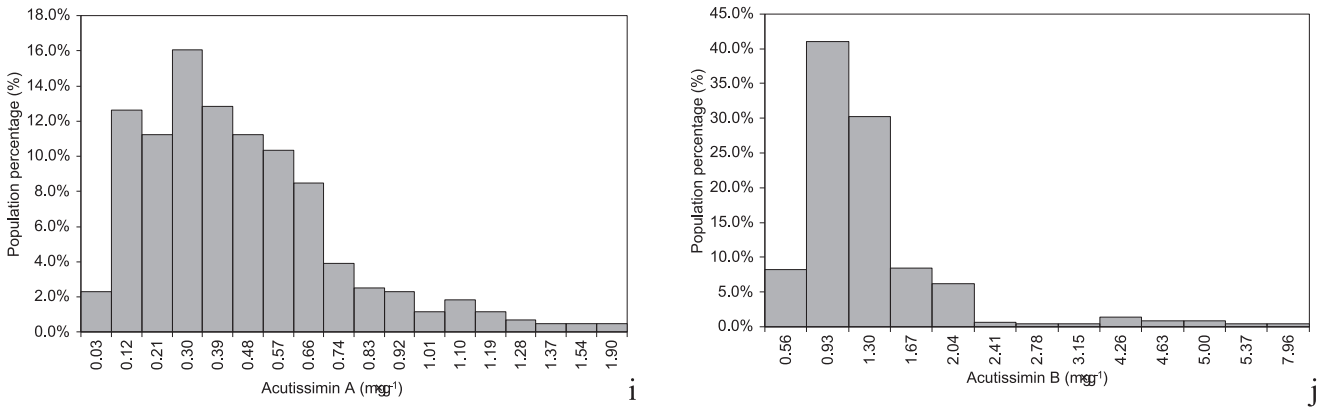


Fig. 3. Distribution of ellagitannin content (a) castalagin, (b) vescalagin, (c) roburin A, (d) roburin B, (e) roburin C, (f) roburin D, (g) roburin E, (h) grandinin, (i) acutissimin A, (j) acutissimin B.



B)

	Percentage (%)	Maximum (mg×g^{-1})	Minimum (mg×g^{-1})	mean (mg×g^{-1})	SD	CV (%)
Monomers						
Castalagin	26.20	15.73	1.64	7.63	2.75	36
Vescalagin	16.30	13.88	0.27	4.76	3.23	68
Pentosylated monomers						
Roburin E	15.10	15.78	0.46	4.42	2.44	55
Grandinin	3.80	9.95	0.03	1.10	1.28	116
Dimers						
Roburin A	4.70	6.26	0.06	1.36	1.26	93
Roburin D	11.60	11.34	0.20	3.39	2.25	66
Pentosylated dimers						
Roburin B	8.60	6.64	0.28	2.52	1.35	54
Roburin C	7.60	9.12	0.22	2.22	1.36	61
Acutissimin A	1.60	1.92	0.05	0.45	0.29	64
Acutissimin B	4.60	8.00	0.50	1.35	0.89	66
Total	100.00	82.23	5.58	29.20	14.04	48

Fig. 3. (*continued*).

the wood to the level of ellagitannins and polyphenols. It is a non-destructive measurement method that uses near-infrared spectroscopy (NIRS) to classify wood. Using this method, staves are classified into three categories according to the Polyphenol Index (PI), which is directly related to the content of ellagitannins.

3.2. Ellagitannins in wood

Next, the percentage representation of each of the ellagitannins with respect to the total content of ellagitannins identified in the wood extracts was analyzed in detail. To contextualize these results, the relative percentages of each compound are compared with those reported in previous studies on naturally dried wood, in which the total number of samples analyzed ranged from 4 to 30 (Cadahía et al., 2001; Viriot et al., 1994). In this case, the analysis was performed on 250 samples, which provides greater representativeness for the characterization of the ellagitannin profile of *Q. petraea*. Fig. 3 shows the histograms corresponding to each of the ellagitannins evaluated, indicating the percentage of samples found within each established concentration range (Fig. 3A). Fig. 3 also shows the results of the quantitative analysis of the ellagitannins in the 250 French oak staves, including the average content, standard deviation, and percentage of each compound (Fig. 3B).

The results obtained highlight the high variability among the 250 samples, with coefficients of variation in the content of each ellagitannin ranging from 36 % to 116 %. The compound that showed the least variability was castalagin, while grandinin showed the highest degree of dispersion among the samples analyzed. The results show that castalagin and vescalagin were the main ellagitannins, with castalagin varying between 1.6 and 15.7 mg × g^{-1}, and vescalagin varying between 0.3 and 13.9 mg × g^{-1} (Figure B), with a castalagin/vescalagin ratio of 1.6. Previous studies on the composition of ellagitannins in *Q. petraea* wood also place castalagin and vescalagin as the most abundant, accounting for 30–50 % and 13–27 % of the total, respectively, with a castalagin/vescalagin ratio between 1.1 and 3.8 (Cadahía et al., 2001; Fernández de Simón et al., 1999; Jordão et al., 2007; Viriot et al., 1994; Vivas et al., 1996), which is in agreement with the findings of the present study.

The next most abundant ellagitannin was roburin E, with concentrations ranging from 0.5 to 15.8 mg × g^{-1} and representing 15.1 % of the total (Fig. 1S). This percentage is like that of vescalagin, indicating that roburin E does not consistently rank third in importance, as previously observed by Fernández de Simón et al. (1999). Next is roburin D, with values between 0.2 and 11.3 mg × g^{-1} (Fig. 3B). Although roburin E is usually the third most abundant ellagitannin, ranging between 12 % and 21 % of the total ellagitannins in wood, like those found in the present study. Previous studies have reported that grandinin usually ranks fourth in terms of contribution, with 5–18 %, while roburin D has lower proportions, generally between 1 % and 6 % (Cadahía et al., 2001; Fernández de Simón et al., 1999; Jordão et al., 2007; Viriot et al., 1994; Vivas et al., 1996), which is consistent with the findings of the present study. Therefore, the percentage of monomeric ellagitannins in relation to the total monomers and dimers found in the 250 staves analyzed was 65 % (castalagin, vescalagin, roburin E, and grandinin), which is in the same range as that reported in other studies, where the percentage of monomers ranges from 46 % to 66 %. Regarding dimers (roburins A, B, C, and D), roburin D was the most abundant, with the percentages described above, followed by roburin B with 8.6 %, roburin C with 7.6 %, and roburin A with 4.7 %. Finally, it was observed that flavano-ellagitannins accounted for 6.2 % of the total ellagitannins (acutissimin A + B) (Fig. 3A). The high variability in the content in wood, due to it being a natural material, makes it interesting to analyze the order of importance of each of them.

As shown in Fig. 3, the histograms allow the distribution of each of

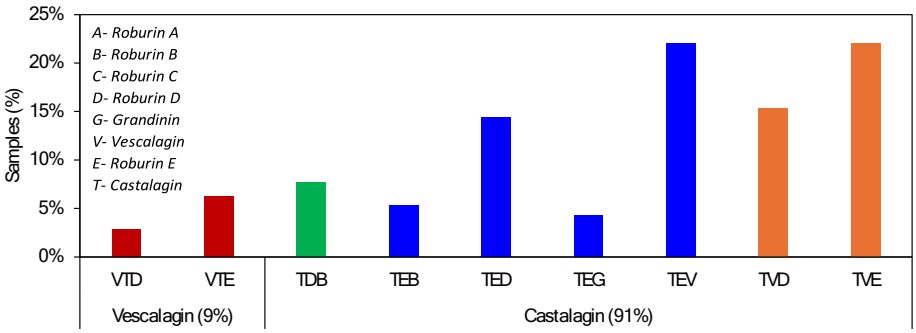


Fig. 4. Profiles with the top 3 ellagitannins in order of contribution: VTD: vescalagin, castalagin, roburin D; VTE: vescalagin, castalagin, roburin E; TDB: castalagin, roburin D and roburin B; TEB: castalagin, roburin E and roburin B; TED: castalagin, roburin E and roburin D; TEG: castalagin, roburin E and grandinin; TEV: castalagin, roburin E and vescalagin; TVD: castalagin, vescalagin and roburin D; TVE: castalagin, vescalagin and roburin E. In red color the VT profile, in green color the TD profile, in blue color the TE profile and in orange color the profile. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

the ellagitannins analyzed to be visualized, providing a detailed view of the degree of dispersion and the relative frequency with which different concentration levels occur in the 250 samples evaluated. The castalagin content varies in the samples between 2 and 16 mg × g^{-1}, with 67 % of the samples between 6 and 10 mg × g^{-1} (Fig. 3a), while the vescalagin content is distributed between 0.3 and 13.9 mg × g^{-1} (Fig. 3b), showing a wider distribution than that recorded for castalagin. 60 % of the samples contain between 0.1 and 1 mg × g^{-1} of roburin A (Fig. 3c), 68 % of the samples contain between 0.6 and 3 mg × g^{-1} of roburin B (Fig. 3d), and 76 % of the samples contain between 0.6 and 2.8 mg × g^{-1} of roburin C (Fig. 3e), 80 % of the samples contain between 0.4 and 3 mg × g^{-1} of roburin D (Fig. 3f), 96 % of the samples contain between 0.8 and 9 mg × g^{-1} of roburin E (Fig. 3g), 85 % of the samples contain between 0.1 and 1.5 mg × g^{-1} of grandinin (Fig. 3h), with almost all samples containing less than 2 mg × g^{-1} of acutissimin A or B (Fig. 3i and 3 j).

It has been reported that in French oak wood, the ellagitannin profile is dominated by castalagin, as shown in the results presented in this study. However, in order of abundance, the ellagitannin in second place varied from one wood to another, causing the order of importance by ellagitannin concentration to vary according to the characteristics of the wood. This distribution would allow the definition of characteristic profiles that reflect the importance of each tannin in *Q. petraea* wood, thus providing a representative view of their presence and variability in this species. Fig. 4 shows the distribution of the different ellagitannins in the samples analyzed, indicating the first three ellagitannins in order of importance in terms of concentration. For example, the VTD profile indicates that in these woods, the most important ellagitannin is vescalagin, followed by castalagin and roburin D. It can be noted that in 91 % of the French oak (*Q. petraea*) samples studied, the most abundant ellagitannin was castalagin. However, in the remaining 9 %, vescalagin is the predominant compound, a relevant finding, since it has not been described in the literature consulted on dry wood; only in green wood in the Allier Forest were similar concentrations of vescalagin and castalagin found (Fernández de Simón et al., 1999). This observation could be due to the high number of samples analyzed in the present study, which

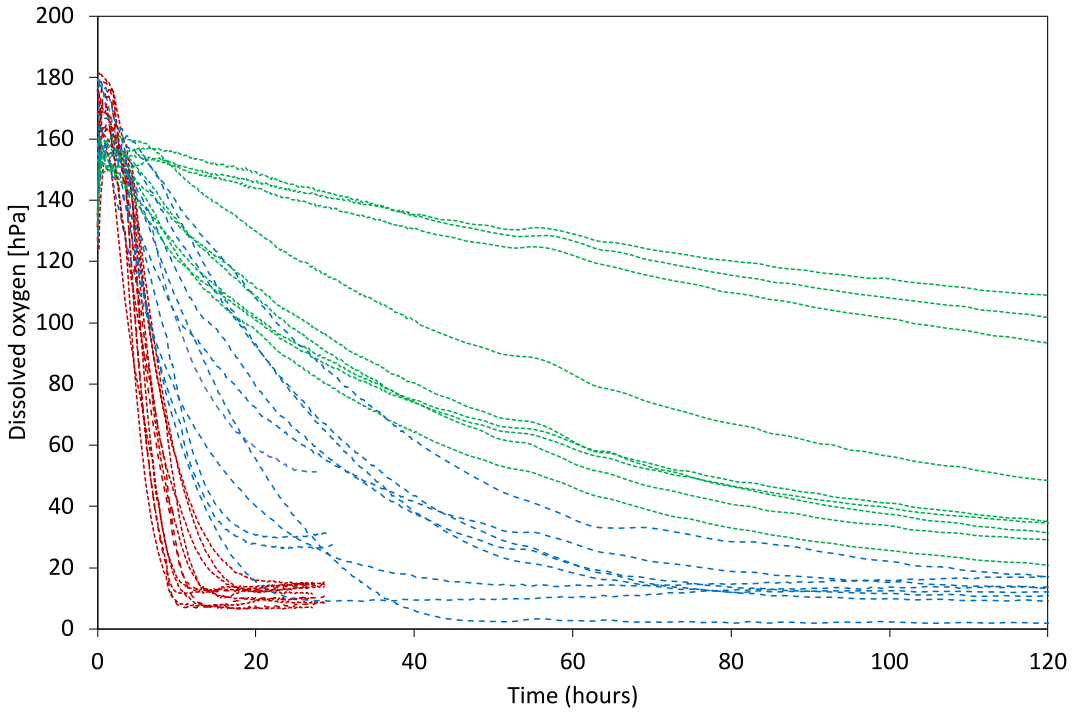


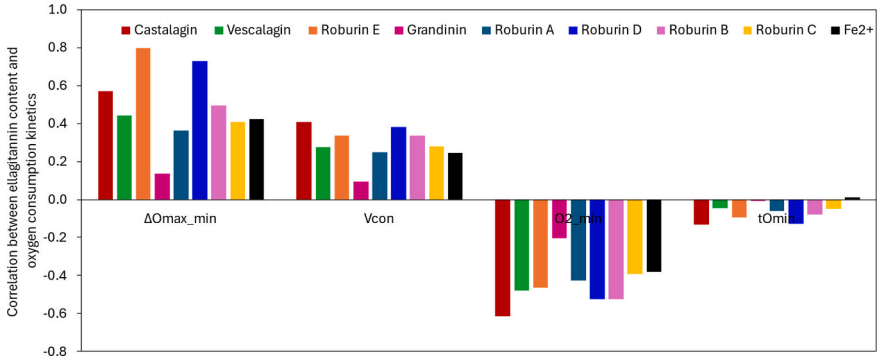
Fig. 5. Oxygen consumption kinetics of Q petraea woods, woods with different oxygen consumption capacity, high capacity in red, medium capacity in blue, low capacity in green. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Table 1
(A) Oxygen consumption kinetics parameters of wood extracts with different ellagitannin profiles. Profiles with the first 2 most abundant ellagitannins: 1-VT: vescalagin, castalagin; 2-TD: castalagin, roburin D; 3-TE: castalagin, roburin E; 4-TV: castalagin, vescalagin. (B) Correlation coefficient between the oxygen consumed by the wood extract, the content of each ellagitannin in the studied woods and Fe^{2+}.

(A)

Ellagitannin profile		1-VT	2-TD	3-TE	4-TV	p-level
	Mean	169.536[b]	161.675[ab]	151.551[a]	163.538[b]	*0.0005*
	SD	9.53	11.69	25.96	15.47	
ΔO_{max_min} (hPa)	CV	5.62 %	7.23 %	17.13 %	9.46 %	
	Min	153.47	138.14	81.24	112.15	
	Max	181.28	175.75	231.68	224.07	
	Mean	178.105	173.697	177.634	176.553	0.647
	SD	10.37	7.23	12.36	12.10	
O_{max} (hPa)	CV	5.82 %	4.16 %	6.96 %	6.86 %	
	Min	161.42	161.99	157.36	159.54	
	Max	196.96	187.16	243.16	231.32	
	Mean	18.070[a]	20.255[a]	34.153[b]	21.310[a]	*0.000*
	SD	5.70	13.72	22.97	9.33	
O_{min} (hPa)	CV	31.56 %	67.74 %	67.25 %	43.78 %	
	Min	9.52	−0.52	5.49	6.57	
	Max	32.5	58.52	108.22	54.46	
	Mean	137.042	146.083	156.227	152.109	0.524
	SD	6.61	40.02	50.31	45.22	
t_{min} (min)	CV	4.82 %	27.40 %	32.20 %	29.73 %	
	Min	116.33	115.67	113.33	113.92	
	Max	140.83	287.67	287.5	287.83	
	Mean	1.152[ab]	1.159[ab]	1.041[a]	1.136[b]	*0.065*
	SD	0.27	0.23	0.28	0.24	
V_{cons} (hPa × min^{-1})	CV	23.27 %	19.60 %	27.04 %	20.74 %	
	Min	0.53	0.48	0.29	0.49	
	Max	1.4	1.52	1.68	1.62	

(B)



allows the detection of less frequent phenomena not observable in studies with less representativeness. In 91 % of the samples in which castalagin was the major ellagitannin, it was observed that in 37 % of the samples after castalagin, vescalagin was the most abundant ellagitannin, defining the TVD and TVE profile. In the remaining 46 % of samples, the second most abundant compound was roburin E (TEB, TED, TEG, TEV profiles). However, this result contrasts with that observed in most previous studies, in which vescalagin is generally described as the second most abundant ellagitannin in *Q. petraea* wood. Only the study of the distribution of ellagitannins in wood from *Q.robur*, forests in Limousin (France) and wood from *Q.petraea* (Fernández de Simón et al., 1999) indicates roburin E as the second most abundant compound in air-dried wood samples. In the present study, it was found that in 8 % of the samples, the second ellagitannin in concentration, after castalagin, was roburin D, castalagin-roburin D (TDE) profile. To our knowledge, this pattern has not been previously described in the literature, suggesting that it is a minority profile only detected when a large number of samples are analyzed.

3.3. Ellagitannin profile in Q. petraea wood and capacity to consume oxygen

The oxygen consumption capacity of different tannins is led by ellagitannins, which are the most protective tannins because they consume available oxygen more quickly than condensed tannins (skin and seed tannins) (Jeremic et al., 2020), although their effect on oxygen consumption tends to decrease rapidly over time (Jeremic et al., 2020). Therefore, some authors indicate that although skin tannins have lower oxygen avidity, when subjected to air saturation cycles, their high content of reactive flavonoids makes them more suitable than ellagitannins for ensuring rapid oxygen consumption (Jeremic et al., 2020). Moreover, the changes undergone by wood ellagitannins in different solutions have been studied in previous studies (García-Estévez et al., 2017), demonstrating that each ellagitannin behaves differently, with its content changing from the moment it passes from the wood to the solution, regardless of the presence or absence of oxygen. During the aging of beverages, the content of each ellagitannin that the wood releases into the beverage affects the greater or lesser consumption of oxygen, thus determining the aging process. For this reason, it is considered interesting to evaluate the relationship between the profile of ellagitannins available in the extract and the kinetics of oxygen consumption, since, as mentioned above, the nature of each ellagitannin affects its oxygen demand.

Fig. 5 shows the average consumption kinetics of some of the woods studied, demonstrating the significant differences that can exist between compounds released by woods from the same batch, which clearly show

different oxygen avidity. It can be observed that in some cases the avidity is so high that the available oxygen is consumed in less than a day (samples in red), while in other cases the rate is much lower and after a week the oxygen consumption process continues without reaching the end (samples in green). These results indicate that the oxidation process of the ellagitannins in the wood extract is a complex process, in which in some cases the substances formed by oxidation have a higher oxygen demand, causing an acceleration in oxygen consumption. In other cases, the oxidation products formed cause a deceleration in consumption until it stops, which causes the remaining oxygen to be higher. Analysis of the consumption kinetics of the extract from each of the woods allows us to extract different parameters that characterize oxygen consumption affinity, speed, etc., thus showing the potential antioxidant capacity of the woods. The characteristic parameters were defined in previous work by our research group and used to analyze the consumption capacity of different samples (del Alamo-Sanza et al., 2021). This study presents the most interesting parameters because they relate to the initial amount of oxygen (O_{max}, hPa), the oxygen consumed (ΔO_{max_min}, hPa), the rate of consumption (V_{cons}, hPa × min^{-1}), the time required to reach the minimum oxygen level (tO_{min}, h), and the residual oxygen that is not consumed by the compounds present in the extract (O_{min}, hPa).

In order to obtain results with a representative number of samples of the profiles described above, four groups of ellagitannin content profiles were established: profile 1 (VT) (Fig. 4-red), which identifies woods in which the major ellagitannin was vescalagin followed by castalagin, accounting for 9 % of the samples studied; profile 2 (TD) identifies woods in which the main ellagitannin was castalagin followed by roburin D, accounting for 7 % of the samples studied (Fig. 4-green); profile 3 (TE) identifies woods in which the main ellagitannin was castalagin followed by roburin E, and which accounts for 46 % of the samples studied (Fig. 4-blue) and finally profile 4 (TV), which identifies woods in which the main ellagitannin was castalagin followed by vescalagin, and accounts for 37 % of the samples studied (Fig. 4-orange). Table 1 shows the different parameters characteristic of the consumption kinetics according to their ellagitannin profile and shows the correlation coefficients between the oxygen consumed by the wood extract and the content of each ellagitannin in the woods studied. It was found that, on average, 46 % of the wood studied, profile 1-VT and profile 4-TV, had significantly higher avidity, as they consumed more oxygen (ΔO_{max_min}; an average of 169 and 163 hPa, respectively) than the other 46 % of the woods with profile 3-TE (151 hPa). The extracts from the woods with profile 2-TD, which represent 7 % of the samples, are in an intermediate position, consuming an average of 161 hPa (Table 1).

With regard to the remaining oxygen, which is not consumed (O_{min}), statistically significant differences were found, with the lowest levels found in extracts from wood with profiles 1-VT, 2-TD, and 4-TV, while 46 % of the wood studied with profile 3-TE showed the highest levels of O_{min}, indicating that oxygen consumption leads to the formation of compounds that cause consumption to slow down until it stops, which leads to higher levels of residual oxygen. Significant differences were also found in the oxygen consumption rate (V_{cons}). As expected, extracts from the woods that consume the least oxygen (profile 3-TE) do so at a significantly lower rate (1.041 hPa × min^{-1}). The extracts from the 1-VT and 2-TD wood profiles showed higher oxygen consumption rates; however, the extracts from the 4-TV wood showed a significantly higher average consumption rate (Table 1). These results confirm the suggestion by Moutounet et al. (1992) who described the oxidation processes of ellagitannins as very slow. These results highlight the significant importance not only of the ellagitannin content of the wood, but also of the type of ellagitannin. All evidence indicates that the presence of high levels of roburin D accelerates oxygen consumption compared to the presence of roburin E, and therefore it can be attributed an important role in ellagitannin-mediated oxidation processes. These results have undoubtedly been obtained due to the large number of samples tested, which has made it possible to evaluate a representative population of *Q petrea* wood. As indicated above, in other studies the results presented were more local, with much smaller populations being studied.

These results are very interesting, as they indicate that each ellagitannin has a very different role. In order to evaluate the importance of each one, correlations were made between the ellagitannin content in the extracts of the different woods and the oxygen consumed by each one. Table 1 also shows this correlation, with positive values indicating a positive relationship between consumption and the content of each ellagitannin, and negative values indicating the opposite relationship. It can be noted that the oxygen consumed by each wood extract has a significant and positive correlation with all the ellagitannins analyzed, except for grandinin, with roburin E showing the highest correlation, followed by roburin D, castalagin, and vescalagin in fourth place. This result indicates that vescalagin is not the ellagitannin with the highest oxygen avidity. In this regard, the literature is quite contradictory. Some authors indicate that oak ellagitannins play an important role as regulators of oxidation in wine, rapidly absorbing dissolved oxygen , while others concluded that the oxidation of ellagitannins, such as vescalagin, is a very slow process. A possible explanation for this apparent contradiction is that the galloyl units of oak glycoside ellagitannins participate in rapid inter- and/or intramolecular oxidation-reduction processes during which their pyrogallol molecules are reversibly converted into semiquinone free radicals (Moutounet et al., 1992). The effect of Fe^{2+} content on oxygen consumption has also been evaluated (Table 1B). Correlations between the different variables of consumption kinetics indicate that higher levels of Fe^{2+} facilitate oxygen consumption, showing significant positive correlations with the amount of oxygen consumed and the rate of consumption, and negative correlations with the level of residual oxygen (O_{min}). Since Fe^{+2} ions can relatively quickly convert to Fe^{+3} ions due to the presence of oxygen and interact with ellagitannins, these ions can oxidize ellagitannins, reducing them back to the Fe^{+2} form.

3.4. The grain of Q. petraea woods and their ellagitannin content

As mentioned above, wood has traditionally been classified according to its grain, defined as the width of the annual growth ring, and based on this classification, the woods analyzed have been classified into three groups. Grain type 1 refers to woods with less than 1.4 mm of annual ring growth (extra-fine and fine oak, $n = 90$), grain type 2 with growth between 1.4 and 2.0 mm ($n = 101$) and type 3 for woods with annual ring growth of more than 2.0 mm ($n = 59$). Given the importance of grain type in the classification and valuation of oak wood in cooperage, the ellagitannin content was analyzed according to the different grain groups established, with the aim of evaluating possible differences associated with this macro-structural variable. The ellagitannin content in grain 1 wood (n = 90) was found to be 24 mg × g^{-1}, ranging from 6 to 67 mg × g^{-1}, while grain 2 staves (n = 101) with an average of 31 mg × g^{-1} showed an ellagitannin level between 6 and 82 mg × g^{-1}. Finally, the third group of wood with grain 3 ($n = 58$) with an average of 34 mg × g^{-1} showed values between 11 and 61 mg × g^{-1}. These variations between the different staves in each grain group are not surprising, since the concentrations of ellagitannins in oak wood depend on the oak species, the origin of the tree, its position in the tree, and the treatment in the cooperage (Fernández de Simón et al., 1999; Viriot et al., 1993). The results obtained do not show a clear trend between grain type and ellagitannin content. Thus, woods classified as grain 3 showed the least variability in this content (n = 58). These results suggest that classifying wood according to grain type does not necessarily imply a higher or lower ellagitannin content.

3.5. Oxygenation rate of Q. petraea woods, ellagitannin content and capacity to consume oxygen

As mentioned above, the wood stave OTR is a fundamental parameter in the aging process of beverages in barrels. In recent years, various

Table 2
Main descriptive statistics of the parameters analyzed: mean value, standard deviation, minimum, maximum and median values and variation coefficient. * for each parameter different letter indicates statistically significant differences.

		Mean	SD	*	Minimun	Maximum	CV			Mean	SD	*	Minimun	Maximum	CV
OTR mg × L^{-1} × day^{-1}	L-OTR	0.012	0.004	a	0.001	0.019	36 %	Castalagin mg × g^{-1}	L-OTR	8.537	2.442	c	15.733	2.890	29 %
	M-OTR	0.034	0.011	b	0.019	0.056	34 %		M-OTR	7.366	2.573	b	12.744	2.062	35 %
	H-OTR	0.093	0.037	c	0.057	0.200	39 %		H-OTR	5.620	2.967	a	11.881	1.636	53 %
	Total	*0.030*	*0.029*		*0.176*	*0.001*	*96 %*		*Total*	*7.635*	*2.748*		*15.733*	*1.636*	*36 %*
D g × cm^{-3}	L-OTR	0.661	0.063	a	0.479	0.815	10 %	Vescalagin mg × g^{-1}	L-OTR	5.867	3.069	c	0.272	13.163	52 %
	M-OTR	0.656	0.052	a	0.476	0.776	8 %		M-OTR	4.256	3.258	b	0.393	13.875	77 %
	H-OTR	0.669	0.047	a	0.506	0.741	8 %		H-OTR	2.795	2.297	a	0.535	8.694	82 %
	Total	*0.660*	*0.057*		*0.176*	*0.815*	*9 %*		*Total*	*4.760*	*3.233*		*0.272*	*13.875*	*68 %*
G mm	L-OTR	1.836	0.605	b	1.000	4.000	33 %	Roburin A mg × g^{-1}	L-OTR	1.773	1.271	c	6.263	0.132	72 %
	M-OTR	1.613	0.506	a	0.604	3.875	31 %		M-OTR	1.177	1.217	b	5.846	0.077	103 %
	H-OTR	1.531	0.690	a	0.830	4.000	45 %		H-OTR	0.610	0.880	a	4.707	0.057	144 %
	Total	*1.697*	*0.594*		*0.604*	*3.980*	*35 %*		*Total*	*1.360*	*1.265*		*6.263*	*0.057*	*93 %*
TPI no units	L-OTR	74.503	33.585	b	16.400	209.350	45 %	Roburin B mg × g^{-1}	L-OTR	2.874	1.201	b	6.240	0.377	42 %
	M-OTR	57.499	28.791	a	12.050	203.350	50 %		M-OTR	2.465	1.387	b	6.636	0.279	56 %
	H-OTR	46.908	34.084	a	7.200	180.250	73 %		H-OTR	1.561	1.202	a	4.884	0.326	77 %
	Total	*63.241*	*33.419*		*7.200*	*209.350*	*53 %*		*Total*	*2.517*	*1.348*		*6.636*	*0.279*	*54 %*
420 no units	L-OTR	0.929	0.723	a	0.170	3.308	78 %	Roburin C mg × g^{-1}	L-OTR	2.471	1.156	b	6.625	0.394	47 %
	M-OTR	0.865	0.616	a	0.005	2.806	71 %		M-OTR	2.230	1.488	b	9.122	0.220	67 %
	H-OTR	0.724	0.660	a	0.016	2.574	91 %		H-OTR	1.420	1.268	a	5.274	0.218	89 %
	Total	*0.870*	*0.673*		*0.005*	*3.308*	*77 %*		*Total*	*2.221*	*1.358*		*9.122*	*0.218*	*61 %*
Fe^{2+} mg × g^{-1}	L-OTR	0.271	0.134	b	0.110	0.888	50 %	Roburin D mg × g^{-1}	L-OTR	4.123	2.259	b	0.511	11.343	55 %
	M-OTR	0.256	0.107	ab	0.103	0.735	42 %		M-OTR	3.093	2.075	b	0.293	9.175	67 %
	H-OTR	0.226	0.127	a	0.105	0.686	56 %		H-OTR	1.954	1.849	a	0.204	7.253	95 %
	Total	*0.258*	*0.123*		*0.103*	*0.888*	*48 %*		*Total*	*3.386*	*2.251*		*0.204*	*11.343*	*66 %*
								Roburin E mg × g^{-1}	L-OTR	4.982	2.469	b	15.783	0.617	50 %
									M-OTR	4.365	2.333	b	9.533	0.459	53 %
									H-OTR	2.849	1.985	a	8.694	0.812	70 %
									Total	*4.421*	*2.443*		*15.783*	*0.459*	*55 %*
Gallic acid mg × g^{-1}	L-OTR	0.451	0.454	a	0.000	2.152	101 %	Grandinin mg × g^{-1}	L-OTR	1.402	1.552	b	9.951	0.054	111 %
	M-OTR	0.370	0.373	a	0.000	1.543	101 %		M-OTR	0.926	1.004	a	6.467	0.034	108 %
	H-OTR	0.302	0.447	a	0.000	2.001	148 %		H-OTR	0.666	0.710	a	3.657	0.044	107 %
	Total	*0.396*	*0.423*		*0.000*	*2.152*	*107 %*		*Total*	*1.099*	*1.275*		*9.951*	*0.034*	*116 %*
Ellagic acid mg × g^{-1}	L-OTR	6.448	1.859	b	3.508	15.068	29 %	Acutissimin A mg × g^{-1}	L-OTR	0.493	0.260	b	1.485	0.092	53 %
	M-OTR	6.563	3.280	b	2.640	26.836	50 %		M-OTR	0.463	0.304	b	1.922	0.053	66 %
	H-OTR	5.296	1.864	a	2.945	11.196	35 %		H-OTR	0.307	0.319	a	1.387	0.052	104 %
	Total	*6.331*	*2.572*		*2.640*	*26.836*	*41 %*		*Total*	*0.454*	*0.293*		*1.922*	*0.052*	*65 %*
∑Ellagin mg × g^{-1}	L-OTR	33.761	13.151	c	6.769	82.231	39 %	Acutissimin B mg × g^{-1}	L-OTR	1.240	0.617	a	5.118	0.536	50 %
	M-OTR	27.831	13.630	b	5.628	61.603	49 %		M-OTR	1.490	1.163	a	7.999	0.500	78 %
	H-OTR	19.078	11.978	a	5.579	50.169	63 %		H-OTR	1.297	0.655	a	4.433	0.687	50 %
	Total	*29.204*	*14.045*		*5.579*	*82.231*	*48 %*		*Total*	*1.351*	*0.893*		*7.999*	*0.500*	*66 %*

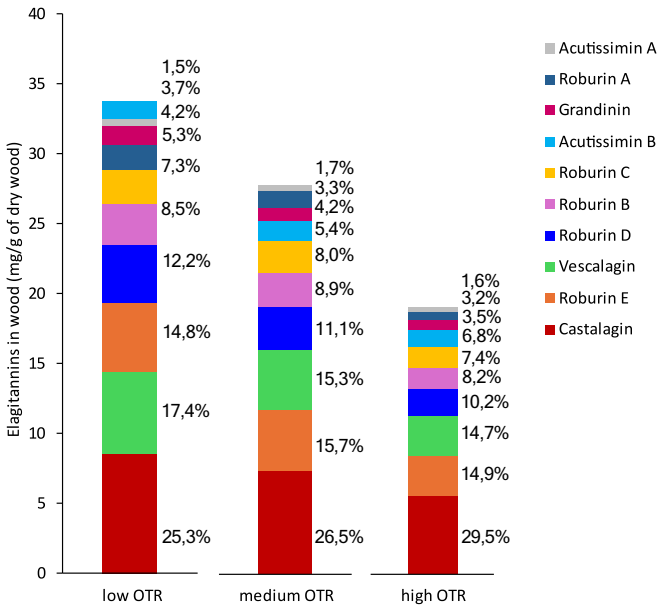


Fig. 6. Cumulative average content of each of the ellagitannins in the woods of each OTR group and percentage of each ellagitannin in each group.

studies have described the characteristics of woods that define their OTR, showing that this rate is a property related to the structure of the wood (Nevares et al., 2019; Nevares & del Alamo-Sanza, 2015). The OTR of the 250 woods studied in this present study ranges between 0.003 and 0.147 mg × L^{-1} × day^{-1}, and given that there is no literature on this subject, it was considered interesting to analyze the relationship between the ellagitannins in the woods according to this property. Previous studies have shown that OTR is highly dependent on wood structure (Nevares et al., 2019; Vivas, 1995), meaning that classification of wood by OTR is an indirect classification of staves by their structure. Three groups of staves have been established: those with L-OTR <0.017 mg × L^{-1} × day^{-1} (L-OTR with an average rate of 0.012 mg × L^{-1} × day^{-1}; $n = 109$), those with medium OTR between 0.018 mg × L^{-1} × day^{-1} and 0.05 mg × L^{-1} × day^{-1} (M-OTR with an average rate of 0.034 mg × L^{-1} × day^{-1}, $n = 100$), and those with H-OTR > 0.05 mg × L^{-1} × day^{-1} (H-OTR with an average rate of 0.093 mg × L^{-1} × day^{-1}, $n = 40$).

Table 2 shows the results of the parameters studied in the wood belonging to the different OTR groups. The mean value and standard deviation of each parameter in each group of wood are presented. The coefficient of variation has also been calculated, as well as the range of values that characterize the group of wood, reflected in the maximum and minimum values. It also indicates whether significant differences were found between the content of the three groups of wood according to OTR. This table shows the wide variability in OTR values, with an average of 0.030 mg × L^{-1} × day^{-1} and a practically equivalent standard deviation (0.029 mg × L^{-1} × day^{-}), representing a coefficient of variation of 96 %. The grain of M-OTR and H-OTR woods was similar to each other and significantly lower than that of L-OTR woods. Similar trends were observed for total phenols (TPI) and the 420 nm absorbance parameter, which are representative of the release of phenolic compounds in the different wood groups; however, in the latter case, no significant differences were found. Regarding mineral content, the Fe^{2+} level in L-OTR wood was significantly higher than in the other H-OTR woods. In the case of Fe^{3+}, significantly different levels were observed among the three groups of wood, with the highest values in L-OTR wood, followed by M-OTR and H-OTR. The presence of these elements will affect oxidation processes and therefore the ability to consume oxygen, as they can act as catalysts for the oxidation of the compounds in the aging beverages themselves and also of the ellagitannins released during the aging process (Vivas et al., 2022). Although the gallic acid content showed a decreasing trend with increasing oxygenation rate (OTR), no statistically significant differences were observed between the groups. In the case of ellagic acid, significant differences were found, woods classified as H-OTR had lower concentrations than those belonging to the L-OTR and M-OTR groups. However, the most pronounced differences were observed in the total ellagitannin content, with a clear pattern of higher concentration in woods with lower OTR, showing significant differences between the three groups. The total concentrations of ellagitannins in L-OTR woods ranged from 6.7 to 82 mg × g^{-1}, while M-OTR staves showed ellagitannin levels between 5.6 and 61.6 mg × g^{-1}. Finally, in the third group of H-OTR woods, it ranged between 5.6 and 50.2 mg × g^{-1}. As indicated above, regardless of their OTR, castalagin was the predominant ellagitannin in 91 % of the oak woods studied, possibly because it is more stable than its isomer, vescalagin, due to the position of the hydroxyl group on carbon 1. Table 2 also shows the average content of each of the ellagitannins in the woods according to their OTR, indicating whether there are significant differences in each group. In addition to finding that woods with lower OTR in general have a higher content of the compounds analyzed, it can also be seen that this content was significantly higher in L-OTR woods. The compounds whose content decreased significantly with increasing OTR, showing differences between the three wood groups, were castalagin, vescalagin, and roburin A. In the case of roburin B, C, D, E, and acutissimin A, a tendency toward higher concentrations in woods with lower OTR was also observed; however, the differences were only statistically significant when comparing the L-OTR and M-OTR groups with the H-OTR group, with no differences found between L-OTR and M-OTR. In the case of grandinin, M-OTR and H-OTR woods had significantly lower content than L-OTR woods. Meanwhile, the content of acutissimin B showed no trend based on wood OTR.

Fig. 6 shows the distribution of ellagitannin cumulative content in wood according to its oxygenation rate. Woods with lower OTR (L-OTR) have the TVE profile (castalagin-vescalagin-roburin E), while woods with higher OTR, M-OTR and H-OTR levels, have TEV profiles (castalagin-roburin *E*-vescalagin). In L-OTR woods, the second most abundant ellagitannin was vescalagin, representing approximately 17 % of the total, followed by roburin E with 15 %. However, in M-OTR and H-OTR

Table 3
Results of the analysis of differences between the parameters of oxygen consumption kinetics in wood extracts of different OTR groups.

	Low OTR					Medium OTR					High OTR					p level
	Mean	SD	CV	Max	Min	Mean	SD	CV	Max	Min	Mean	SD	CV	Max	Min	
tO_{min} (h)	156.82	53.10	34 %	287.50	114.00	148.14	36.48	25 %	287.83	115.75	149.62	51.21	34 %	287.67	113.33	*0.4146*
O_{max} (hPa)	174.78	9.64	6 %	212.75	157.39	178.29	13.37	8 %	243.16	157.36	174.92	11.01	6 %	207.05	160.13	*0.1046*
O_{min} (hPa)	22.09	12.51	57 %	66.13	0.00	27.66	16.88	61 %	103.23	5.49	41.12	31.15	76 %	108.22	6.16	*0.0008*
ΔO_{max_min} (hPa)	161.14	13.88	9 %	205.95	107.44	158.98	22.50	14 %	231.68	84.64	142.48	29.34	21 %	198.11	81.24	*0.0040*
V_{cons} (hPa × min^{-1})	1.11	0.26	24 %	1.52	0.38	1.12	0.23	21 %	1.68	0.32	1.03	0.32	31 %	1.44	0.29	*0.4073*

woods, roburin E ranked second in abundance (15–16 % of the total), followed by vescalagin with a contribution of 15 %, i.e., with contents very similar to those of roburin E. In all cases, the fourth most abundant ellagitannin was roburin D with contributions ranging from 10 to 12 %, followed by roburin B with levels between 8 and 9 % and roburin C with a representation of 7–8 % of the total. Additionally, the contribution of the least abundant ellagitannins varied according to the OTR group. Thus, for H-OTR woods, acutissimin B, roburin A, grandinin and acutissimin A; for M-OTR woods, acutissimin B, A, grandinin and acutissimin A; and for L-OTR woods, roburin A, grandinin, acutissimin B and acutissimin A. As mentioned in the analysis of the ellagitannin content of the woods, the wide variations can be explained by the fact that oak wood is a natural material and the concentrations of ellagitannins depend on the oak species, the origin of the tree, washing by water during the curing of the staves, degradation by microorganisms, and chemical oxidation (Fernández de Simón et al., 1999).

As described above, the role of the ellagitannin profile of *Q. petraea* barrel wood varies according to the type and quantity of ellagitannin, contributing to the protection of the wine during aging, as they will be transferred to the wine and consume more or less oxygen, thus affecting the oxidation of aged wines and the shelf life of the wines obtained. In view of the results described above, it was considered particularly interesting to evaluate the role of ellagitannins in wood according to their OTR group. Table 3 therefore shows the data for each parameter of oxygen consumption kinetics, differentiating the woods by their OTR level. It can be observed that the level of residual oxygen (O_{min}) was significantly lower in L-OTR woods than in woods that oxygenate more (H-OTR). This result indicates that woods with lower oxygenation rates release compounds that interact with aging beverages, causing longer consumption kinetics to consume more oxygen and reach lower levels of unconsumed oxygen. This result is consistent with what has been described above and confirms that the compounds released by L-OTR woods ($n = 109$) make the resulting beverages richer in ellagitannins, showing higher levels of total phenols (TPI) and higher total ellagitannin content. The high oxygen demand of phenols causes greater oxygen consumption, resulting in lower residual oxygen levels (O_{min}). Therefore, although all woods consume oxygen at the same rate, L-OTR woods have longer kinetics, consume more oxygen and reach the lowest level of remaining oxygen (O_{min}). These are therefore woods with greater antioxidant power.

4. Conclusions

This study characterized 250 staves used in cooperage with a grain size between 0.65 and 3.98 mm and an OTR of up to 0.153 mg $\times$ L^{-1} $\times$ day^{-1}. It has been shown that the ellagitannin content of *Q. petraea* wood is not always dominated by castalagin; in 9 % of the wood studied, the main ellagitannin was vescalagin. In addition, it has been found that, in order of importance, in 46 % of the samples, roburin E is the second ellagitannin after castalagin, with vescalagin occupying this second position in 37 % of the wood studied and roburin D in the remaining 7 % of samples. These different ellagitannin profiles define the properties of the wood and therefore determine the aging process of beverages in barrels. Thus, the evaluation of oxygen consumption kinetics indicated that the oxidation of ellagitannins in wood extracts is a complex process. In some cases, the substances formed by oxidation show a greater affinity for oxygen, accelerating its consumption, an effect observed in woods where castalagin and vescalagin are the predominant ellagitannins. In other cases, it could be the oxidation products formed that cause a slowdown in consumption until it stops, resulting in higher residual oxygen, as observed in woods with castalagin and roburin E as the main ellagitannins.

This study revealed differences in ellagitannin composition in woods with varying oxygen transfer capacities, which will undoubtedly impact the production of different wines, not only because of OTR but also due to variations in ellagitannin profiles. In L-OTR woods, after castalagin, the second most abundant ellagitannin was vescalagin, representing approximately 17 % of the total, followed by roburin E with 15 %. However, in M-OTR and H-OTR woods, roburin E ranked second in abundance (15–16 % of the total), followed by vescalagin with a contribution of 15 %.

These results suggest that classifying wood according to grain type does not necessarily imply a higher or lower content of ellagitannins. Understanding the properties of oak wood is essential for better management and more consistent use of French oak wood, enabling the production of barrels with well-defined, customized characteristics tailored to each type of wine. This approach allows for a more objective and systematic use of wood, maximizing its potential, an aspect that is currently underutilized, while adding value and making the process more sustainable and consistent.

CRediT authorship contribution statement

Maria del Alamo-Sanza: Writing – review & editing, Writing – original draft, Supervision, Methodology, Funding acquisition, Conceptualization. **María Asensio-Cuadrado:** Investigation, Formal analysis. **Rosario Sánchez-Gómez:** Investigation, Formal analysis. **Ana M. Martínez-Gil:** Writing – review & editing. **Ignacio Nevares:** Writing – review & editing, Project administration, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fochx.2025.103285.

Data availability

Data will be made available on request.

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