



# Influence of the thickness of oak alternatives on the composition and quality of red wines

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## Abstract

This work studies the influence of the thickness of oak alternatives on the composition and quality of red wines. A red wine was aged in control conditions and also in contact with oak chips, and with thin and thick oak staves for 12 months. As expected, all the wines aged in contact with all the oak alternatives were enriched in total polyphenols and had a higher colour intensity. In addition, the contact with all the oak alternatives enriched the wine in furfural and total furans, vanilla and total aldehydes and ketones, eugenol and total volatile phenols, and in  $\beta$ -methyl- $\gamma$ -octalactones. However, the thickness of the oak alternative seems to play an important role in the composition and quality of the wine. Specifically, the wines aged in contact with the two types of staves had a more intense colour than the wine aged with oak chips, as well as a higher total phenolic index and higher eugenol concentration. Moreover, the  $\beta$ -methyl- $\gamma$ -octalactones concentration was higher in the wine supplemented with thick staves. Finally, the wines supplemented with the two types of staves had a higher intensity of the spicy attribute than the wine aged with oak chips. The wine supplemented with thick staves had a higher intensity of candy/pastry, toasted, smoked, complexity, aromatic intensity, sweetness, structure, and persistence. Finally, the panel preferred the wine aged with thick staves followed by, in descending order, the wines aged with thin staves, oak chips, and the control.

**Keywords** Red wine · Oak alternatives · Thickness · Wine colour · Wine composition · Wine quality

## Introduction

High quality red wines are usually aged in oak barrels because during this process the wine gains in stability and complexity [1, 2]. The oak wood, during the contact with the wine, releases several volatile substances that improve the wine's quality by enhancing its aroma and flavour [3–5]. The wood also releases phenolic compounds that contribute

to the textural sensations [6, 7]. All these compounds are naturally present in the original oak or are formed during the process of toasting the barrels [8].

However, only some of these volatile substances have a substantial influence on the final wine aroma [2, 9]. Among them, the furans, phenolic aldehydes and ketones, volatile phenols and  $\beta$ -methyl- $\gamma$ -octalactones stand out [10, 11].

The furan compound family, which mainly includes furfural, methylfurfural, hydroxymethylfurfural and furfuryl alcohol, contributes to the smoked and toasted nut notes found in wine [12, 13]. Nevertheless, the role played by furans in wine aroma does not seem to be of great importance, because they are usually below their perception threshold [9]. Nevertheless, it has been reported that furfural is a precursor of furfurylthiol, an interesting volatile substance with a very pleasant coffee aroma [14], and therefore its presence cannot be overlooked.

The family of phenolic aldehydes and ketones contributes to the typical vanilla aroma of oak-aged wines, with vanillin being the major contributor [10, 15]. The

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volatile phenol family mainly includes ethylphenol, guaiacol, methylguaiacol, ethylguaiacol, vinylguaiacol, eugenol, and *trans*-isoeugenol. Eugenol provides the highly appreciated spicy note of cloves [1, 16], while the other volatile phenols contribute with smoked/ toasted notes [3, 12]. These volatile phenols can have a negligible effect if their concentrations are below their sensory threshold, a pleasant effect when their presence provides slight notes of toasted bread, and a disagreeable effect when their concentrations are so high that the burnt bread notes predominate [2, 9]. Finally,  $\beta$ -methyl- $\gamma$ -octalactones, also called whiskey lactones, are present in the form of two isomers (*cis* and *trans*). They are both responsible for the coconut flavour but the *cis* isomer has a much lower perception threshold than the *trans* isomer, and therefore contributes much more to the perception of this aroma [17, 18].

Oak wood also releases phenolic compounds such as ellagitannins that contribute to some of the textural sensations of wine, such as body and astringency [7, 19, 20].

All of these substances are released from oak wood into the wine during barrel aging and their proportion depends on various factors, such as the botanical and geographical origin of the oak, seasoning technique, degree of toasting, and the number of times the barrel has been used before [2, 4, 7, 13, 21].

Aging in oak barrels also allows a moderate amount of oxygenation, due to oxygen permeating through the pores in the wood and through the joints between the staves [22, 23]. This moderate diffusion of oxygen through the wood causes different reactions between anthocyanins and proanthocyanidins [24, 25], which lead to the formation of new pigments [26–29]. It is generally accepted that these reactions generated by the oxygen supply stabilize the colour [30, 31] and reduce the astringency in red wines [32, 33].

However, ageing red wines in oak barrels is an expensive and laborious process that can only be applied to wines with a certain added value [34, 35]. Therefore, the use of oak alternatives has progressively increased over recent years, as the processes that take place in the barrels are reproduced more economically and quickly [36, 37]. Several studies have explored how oak alternatives can contribute to wine composition and quality. Aspects such as the influence of the botanical origin [38–40], the toasting level [41, 42], the size [43–45], the contact time [46–48] and even the influence of wood chips that are not oak [49] have been widely studied. Nevertheless, little is known about the influence of their thickness. The aim of this research was therefore to study how different thicknesses of oak alternatives influence the colour, phenolic compounds and volatile composition of a red wine, especially with regard to the substances released by oak wood.

## Materials and methods

### Chemicals

Methanol, acetonitrile, formic acid, acetic acid, ethyl acetate, and absolute ethanol were of HPLC grade and purchased from Panreac (Barcelona, Spain). Acetaldehyde, phloroglucinol, ascorbic acid and sodium acetate were purchased from Sigma-Aldrich (Madrid, Spain). Dichloromethane (LiChrosolv quality) was purchased from Merck (Darmstadt, Germany) and pentane from Fluka (Buchs, Switzerland). The standards for proanthocyanidin analysis were: (+)-Catechin ( $\geq 99\%$ ), (–)-epicatechin ( $\geq 99\%$ ), (–)-epigallocatechin ( $\geq 98\%$ ), and (–)-epicatechin-3-O-gallate ( $\geq 97.5\%$ ). These were purchased from Extrasynthese (Genay, France). The chemical standards for gas chromatography were purchased from Aldrich and PolyScience (Niles, USA). The rest of the chemicals were of high purity and purchased from Panreac (Barcelona, Spain). Pure water was obtained from a Milli-Q purification system (Millipore, USA).

### Oak alternatives

Three types of oak alternatives were used: oak chips (between  $7.5 \times 3 \times 1.5$  and  $20 \times 13 \times 3$  mm), thin staves ( $7 \times 47 \times 960$  mm) and thick staves ( $17 \times 47 \times 960$  mm). All these wood alternatives were from French oak (*Quercus petraea* (Matt.) Liebl) with origins and characteristics that were as similar as possible. All these oak alternatives were medium toasted. All oak alternatives were provided by Tonellerie Radoux – Pronektar (Mézières-en-Brenne, France).

### Experimental design

This study was carried out at the experimental winery of the Universitat Rovira i Virgili (Mas dels Frares, Constantí, Tarragona, Spain) with around 1,200 L of red wine from the 2019 vintage after malolactic fermentation had completely finished. This red wine was a blend of Cabernet Sauvignon, Merlot and Grenache from the AOC Tarragona (Spain). The wine's chemical characteristics were, ethanol content: 13.7% (v/v); titratable acidity: 5.3 g/L (expressed as tartaric acid); volatile acidity: 0.42 g/L (expressed as acetic acid); pH: 3.73; total phenolic index: 54.6; free sulphur dioxide: 28 mg/L; and total sulphur dioxide: 56 mg/L.

The red wine was poured into twelve 100-L plastic tanks with an oxygen permeability similar to oak barrels (Flexcube, Quilinox, Paterna, Valencia, España). Three tanks were supplemented with 2.5 g/L oak chips; three tanks with 5 g/L of thin staves; three tanks with 10 g/L of thick staves; and finally, the last three tanks were kept as controls. These

dosages were chosen based on an equivalent oak impact intensity according to previous experiments (Data not shown). These wines were stocked at  $16 \pm 2$  °C and a relative humidity of 70–80%. The wines were analysed at 2, 6 and 12 months of aging in these conditions. The contact time of wine with oak alternatives is usually shorter (1–3 months), but this time was extended to 12 months to study whether the different oak alternatives were capable of releasing substances for longer times. Wines were also tasted by a trained panel at the end of the ageing time.

## General analysis

Analytical methods recommended by OIV [50] were used to determine ethanol content, titratable acidity, volatile acidity, pH, free sulphur dioxide and total sulphur dioxide.

## Spectrophotometric analysis

Absorbance measurements were performed using a Helios Alpha UV – vis spectrophotometer (Thermo Fisher Scientific Inc., Waltman, MA, USA). Colour intensity (CI), total anthocyanins (TA), and PVPP Index were estimated following the methods described by Glories [51]. The CIELab coordinates – lightness ( $L^*$ ), chroma ( $C^*$ ), hue ( $h^*$ ), red-greenness ( $a^*$ ), and yellow-blueness ( $b^*$ ) – were determined according to Ayala et al. [52]. Data processing was performed with MSCV software [53]. The total colour difference ( $\Delta E_{ab}^*$ ) was calculated according to Martínez et al. [54].

## Anthocyanins, tannins and related parameters

The total phenolic index (TPI) was determined by measuring absorbance at 280 nm, expressed as absorbance units [55]. Total anthocyanins (TA) and the PVPP Index were estimated following the methods described by Glories [51]. Total tannin concentration was estimated by precipitation with methylcellulose [56]. Acid-catalysed depolymerization of proanthocyanidins in the presence of an excess of phloroglucinol (phloroglucinolysis method) was used to analyse the proanthocyanidin content, monomeric composition, and mean degree of polymerization (mDP) of all the wines according to the procedure reported by Kennedy and Jones [57].

## Volatile compound analysis

The volatile compounds released from the oak wood were analysed by GC/MS using a Focus-GC system gas chromatograph coupled to an ISQ mass spectrometer with an electron impact ionization source and quadrupole analyser equipped with a TriPlus autosampler, all from ThermoQuest.

The conditions of the detector were as follows: electron multiplier voltage of 1250 V, impact energy of 70 eV, ion source temperature of 250 °C, and mass scanning range of 40–250 amu. We used a BP21 column (SGE) with an internal diameter of 60 m – 0.32 mm and a free fatty acid phase (FFAP) of 0.25  $\mu$ m thickness (polyethylene glycol treated with nitroterephthalic acid).

The volatile compounds were extracted and analysed using the method developed by Ibarz et al. [58]. Only one analysis was performed by sample since two barrels were included by experimental group. 25 mL of wine was passed through columns filled with 0.2 g of LiChrolut EN (40–120  $\mu$ m, Merck), using 4-nonanol as the internal standard. The columns were then washed with 25 mL of water to remove sugars, acids, and other polar substances. The fraction of volatile compounds was eluted with 15 mL of pentane: dichloromethane (2:1 v/v). The extracts were concentrated to 100  $\mu$ L by distillation in a Vigreux column. They were then kept at – 20 °C until analysis.

The separated compounds were identified by their mass spectra and chromatographic retention times, using commercial products as a standard. Quantification was performed by analysing the characteristic m/z fragment for each compound using the internal standard method with specific calibration curves for the available compounds. The results for the non-available compounds were expressed in concentration units ( $\mu$ g/L or mg/L) as internal standard equivalents, which were obtained by normalizing the compound peak to that of the internal standard and multiplying it by the concentration of the internal standard. Only the main volatile compounds released by oak wood (furanic compounds, vanillin,  $\beta$ -methyl- $\gamma$ -octalactones, and volatile phenols) are shown here because the aim of this study was to focus on the aromas released by wood.

## Sensory analysis

All sensory analyses were performed in the tasting room of the Faculty of Oenology in Tarragona (Universitat Rovira i Virgili), which was designed in accordance with UNE 87004.197 [59]. Tasting was carried out using ISO official tasting glasses [60]. Each sample consisted of 30 mL of wine at room temperature (20 °C), covered with clear plastic petri dishes to minimize the escape of volatile components. The samples were randomly coded with three-digit numbers.

All the samples were tasted by 18 tasters. This panel was made up of ten men and eight women aged between 23 and 60. Ten of them were experts from the Universitat Rovira i Virgili and the other eight were experts from Tonnellerie Radoux – Pronektar. To simplify the tasting, the wines from three similar (triplicate) samples of each experimental group were mixed.

A tasting session was carried out for the descriptive sensory analysis in accordance with ISO-13299 [60]. During this session, the tasters were required to evaluate the intensity of twelve olfactory sensory attributes (Aromatic intensity, Fresh fruit, Ripe fruit, Fresh vegetal, Dry vegetal, Coconut, Vanilla, Candy/Pastry, Toasted, Smoked, Spicy, and Complexity notes) and eight gustatory sensory attributes (Acidity, Sweetness, Mouthfeel, Structure, Astringency, Aggressiveness, Bitterness and Persistence) on a scale of 1 to 10 (1 = slight intensity and 10 = maximum intensity). These attributes were selected based on the experience of the experts from Tonnellerie Radoux – Pronektar. The intensity level of each descriptor was then expressed as the mean value of all the results. Tasters were also required to rank the wines based on their order of preference. A sensory training session was held beforehand so that the panellists could agree on the criteria for each of the different sensory attributes. Samples were served randomly to avoid any influence arising from the tasting order.

## Statistics

All analytical data are expressed as the arithmetic average  $\pm$  standard deviation of three replicates. All sensory data are expressed as the arithmetic mean  $\pm$  standard deviation of the scores of the 18 tasters. One-factor analysis of variance (ANOVA) was carried out using SPSS 15.0 software (SPSS Inc., Chicago, IL).

## Results and discussion

### Colour parameters

Table 1 shows the colour intensity and CIEL\*a\*b\* coordinates of the original wine and the wines obtained after applying the different experimental conditions. In general, the colour intensity (CI), and the green–red component ( $a^*$ ) of the control wine tended to decrease, whereas the lightness ( $L^*$ ) and the blue–yellow component ( $b^*$ ) increased. This is the expected colour evolution of the red wine over time because the colour loses intensity and changes its hue towards more yellowish nuances. Other authors have found similar results [62–65]. However, the supplementation with

oak alternatives seems to modify this trend. In fact, the CI,  $a^*$ , and  $L^*$  of all the wines supplemented with all the types of oak alternatives after 12 months were statistically similar to the corresponding values of the original wine. Furthermore, CI was significantly higher and  $L^*$  significantly lower than the corresponding values of the control wine after 12 months of ageing, which indicates a more intense colour in the case of all the wines supplemented with oak alternatives. This apparent colour-protecting effect of oak alternatives could be because oak wood releases phenolic compounds [6–8, 20, 65] capable of exerting a copigment effect [66–68] and which favour colour stabilization [69, 70]. It is known that anthocyanins can form association structures with other colourless compounds. These complexes adopt a sandwich-like structure that protects the flavylium cation against nucleophilic attack by water, avoiding the formation of the colourless hemiketal [66, 67]. Consequently, this phenomenon, called copigmentation, increases wine colour intensity (hyperchromic effect).

Figure 1 shows the total difference of the colour ( $\Delta E_{ab^*}$ ) between the different wines at the different ageing times to better reflect the effect of oak alternatives on wine colour. The  $\Delta E_{ab^*}$  did not exceed the threshold value of 3 units between the control wine and the wine supplemented with oak chips at any time (Fig. 1.A). Therefore, no differences between the colours of the two wines could be distinguished by the human eye throughout the experimentation time [54]. In contrast, the  $\Delta E_{ab^*}$  values between the wines supplemented with thin (Fig. 1.B) and thick staves (Fig. 1.C) and the control wine were clearly higher than 3 units at all times. These data indicate that the colour of the wines supplemented with the two types of staves is not only more intense but also clearly distinguishable from the control wine at first glance. The comparison between the wine supplemented with chips and the wine supplemented with thin (Fig. 1.D) or thick staves (Fig. 1.E) indicates that the  $\Delta E_{ab^*}$  value was greater than 3 units at all times. This shows that the higher colour intensity of wines supplemented with the two types of staves can be differentiated from the wine supplemented with oak chips by the human eye. In contrast, the  $\Delta E_{ab^*}$  between wines supplemented with thin and thick staves (Fig. 1.F) was always below 3 units, which indicates that the colours of these wines are indistinguishable from each other.

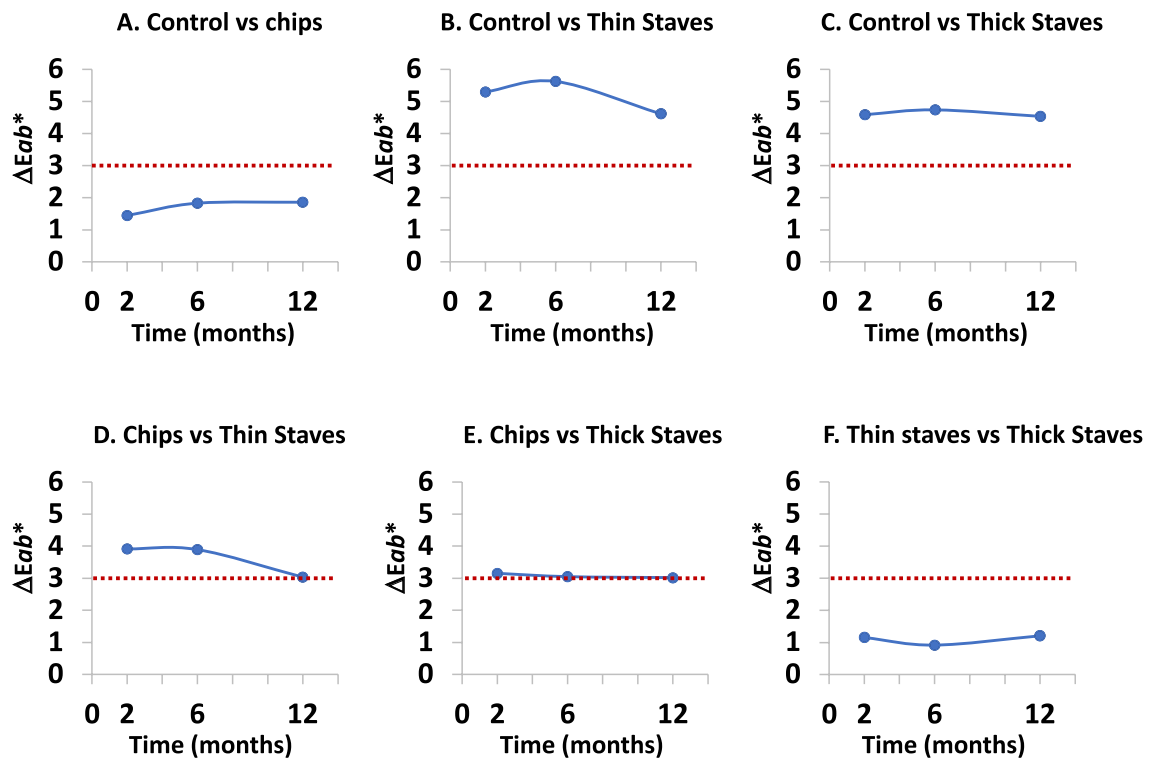
### Anthocyanins and related parameters

Table 2 shows the anthocyanin concentration measured by spectrophotometry and HPLC as well as the PVPP and the ionization index of the different wines. The total anthocyanin concentrations determined by HPLC were much lower than those obtained by spectrophotometry. This could be logical as spectrophotometric analysis is known to overestimate the

**Table 1** Colour parameters

| a | s | d |
|---|---|---|
| 1 | 2 | 3 |

Results are expressed as mean  $\pm$  standard deviation of three replicates. Different letters indicate the existence of statistical difference ( $p < 0.05$ )



The red dotted line indicates the  $\Delta E_{ab}^*$  value below which the human eye cannot distinguish between two samples.

**Fig. 1** Total colour differences ( $\Delta E_{ab}^*$ ). The red dotted line indicates the  $\Delta E_{ab}^*$  value below which the human eye cannot distinguish between two samples

**Table 2** Anthocyanins and related parameters

| q | w |
|---|---|
| 5 | 6 |

Results are expressed as mean  $\pm$  standard deviation of three replicates. Different letters indicate the existence of statistical difference ( $p < 0.05$ )

total anthocyanin concentration because it includes the contribution from other pigments in its measurement, whereas only free anthocyanins are detected by the HPLC method [71].

As expected, the total anthocyanin concentration decreased over time in all the experimental conditions, which confirms that oxygen can degrade anthocyanins, as has been previously reported [72]. This trend was observed both in anthocyanins determined by spectrophotometry and by HPLC. In general, the decrease in the anthocyanin concentration was higher in the wines supplemented with oak alternatives than in the control wine. This difference can be attributed to the absorption of anthocyanins by oak wood [73, 74].

The PVPP index tended to increase over time in all the wines. Since the PVPP index indicates the percentage of anthocyanins combined with flavanols [51], these results confirm that micro-oxygenation that takes place due to the oxygen permeability of the plastic tanks has induced the formation of ethyl bridged flavanol-flavanol adducts [24, 27, 31]. No significant differences were found between the PVPP index of the wine supplemented with oak chips and the control wine at any time. However, the two wines supplemented with staves had significantly higher PVPP Index values than the control wine and the wine supplemented with oak chips. It has been reported that the contact between red wine and oak wood favours the union of anthocyanins and flavanols [69, 70]; however, according to our results only the staves seem to exert this effect. In any case, the formation of these new pigments has been associated with the stabilization of the red wine colour since they are more stable than free anthocyanins [24, 31, 32, 51]. Table 2 also shows the ionization index, which indicates the percentage of anthocyanins that contribute to the red wine colour [51]. The ionization index for the control wine seems to remain stable over time. However, the ionization index of the wine supplemented with oak chips and especially of the two wines

supplemented with staves tended to increase with the ageing time. This augmentation of the percentage of the anthocyanins contributing to the red colour is probably related to the higher combination of these anthocyanins with flavanols, as the PVPP index indicates. As it is well known, pH exerts a large effect on the percentage of the different forms of anthocyanins. The percentage of the red coloured form, the flavylium cation, is greater when the pH is lower [51]. It has also been described that the anthocyanin-flavanol adducts contribute more to the red colour than the free anthocyanins at the same pH [75, 76]. Consequently, the higher PVPP index is associated with a higher colour intensity and ionization index. In addition, the above mentioned copigmentation effect of the phenolic compounds released by oak alternatives [7, 20, 68, 70] can also contribute to the higher colour intensity and ionization index of the wines supplemented with oak alternatives.

### Tannins and related parameters

Table 3 shows the total phenolic index (TPI), the proanthocyanidin concentration and related parameters of the different wines. It includes two different measurements for tannins: first, the tannin concentration as measured by the methylcellulose method [56], and second, the more specific analysis of proanthocyanidins by phloroglucinolysis [57], which also provides information about the mean degree of polymerization (mDP) and the percentages of prodelfphinidins (%PD), and galloylation (%GAL).

The total phenolic index (TPI) of the different wines behaved erratically with slight but sometimes significant differences. This erratic behaviour may be due to the complicated balance between the release of phenolic compounds from the oak alternatives and the precipitation of a part of the colour matter of the wines. In any case, the TPI of the wines supplemented with oak alternatives seems to be slightly higher than that of the control wine.

The tannin concentration measured by the methyl cellulose precipitation method generally followed a similar trend to that observed for the TPI, being the final tannin content

slightly but significantly higher in the wines supplemented with the two types of staves.

The values of the proanthocyanidin concentration determined by phloroglucinolysis were lower than those of the tannin concentration measured by the methyl cellulose precipitation method. These results are completely logical since phloroglucinolysis cannot cleave all the monomers present in proanthocyanidin polymers and therefore always underestimates their concentration. In general, three types of non-depolymerizable inter-flavonoid bonds have been reported: A-type bonds, oxidative-type bonds leading to B-ring bond formation, and bonds involving an anthocyanin unit [77, 78]. In any case, no significant differences were found in the proanthocyanidin concentration in any of the wines or at any time. This result therefore indicates that the presence of oak alternatives did not exert any influence on this parameter. However, certain statistically significant differences were observed in the mDP that tended to increase over time in the case of the control wine, whereas it decreased in all the wines supplemented with oak alternatives. Significant differences were also observed in the percentage of prodelfphinidins (%PD), which tended to decrease in all wines but to a greater extent in wines supplemented with oak alternatives. Likewise, the percentage of galloylation (%GAL) remained stable in the control wine while it tended to increase in all the wines supplemented with oak alternatives.

### Volatile substances

Figure 2 shows the furfural (Fig. 2.A) and the total furan concentrations (Fig. 2.B) of the different wines. As expected, the concentration of furfural and total furans was negligible in the control wine, while these substances were clearly present in the wines supplemented with oak alternatives. In the case of the wine supplemented with oak chips the concentration of furfural and total furans significantly increased between two and six months of ageing to subsequently decrease until, at 12 months, it reached levels similar to those at two months. In contrast, the furfural and total furan concentrations tended to increase during the entire ageing time. It should be noted that the furfural and total furan concentrations were in all cases much below the threshold of these substances [2, 9, 13], and consequently their contribution to wine aroma is negligible in all cases.

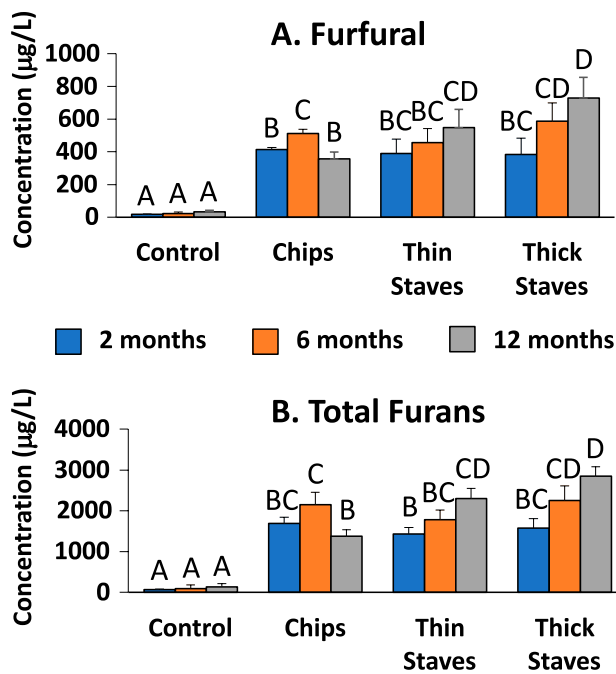
Figure 3 shows vanillin (Fig. 3.A) and total volatile aldehydes and ketones (Fig. 3.B) of the different samples. As was the case with furans, vanillin, and the total volatile aldehydes and ketones levels were almost non-existent in the control wine. In contrast, the concentration of these compounds increased over time in all the wines supplemented with oak alternatives. This tendency to increase was very similar in all the oak alternatives. In all cases, the vanillin levels were above the reported sensory threshold [2, 9, 13]

**Table 3** Tannins and related parameters

| z | x |
|---|---|
| 5 | 6 |

Results are expressed as mean  $\pm$  standard deviation of three replicates. Different letters indicate the existence of statistical difference ( $p < 0.05$ )

TPI total phenolic index, mDP mean degree of polymerization, %PD percentage of prodelfphinidins, %GAL percentage of galloylation



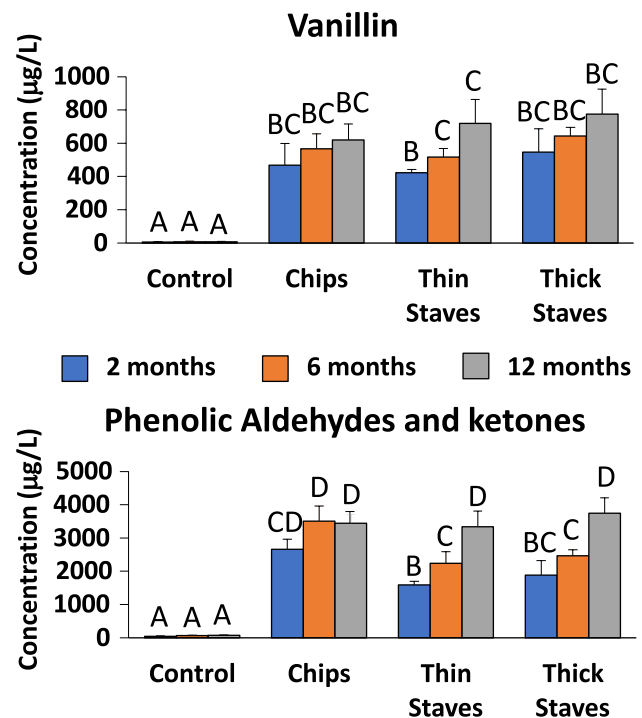
Results are expressed as mean  $\pm$  standard deviation of three replicates. Different letters indicate the existence of statistical difference ( $p < 0.05$ ).

**Fig. 2** Furfural and total furans. Results are expressed as mean  $\pm$  standard deviation of three replicates. Different letters indicate the existence of statistical difference ( $p < 0.05$ )

and therefore its aroma should be perfectly perceived in all the wines supplemented with oak alternatives.

Figure 4 shows the eugenol levels (Fig. 4.A) of the various experimental conditions. Eugenol was present at a very low concentration in the control wine. It is generally acknowledged that the presence of eugenol in wine is mainly due to aging in contact with wood [2, 6, 8, 9]. However, its presence at very low concentrations has been reported in wines without contact with oak [79–81]. In any case, the eugenol concentration in the control wine was always below its sensory threshold [82] and therefore its impact on the aroma of the control wine was insignificant. The eugenol levels increased over time in all the wines supplemented with oak alternatives. The eugenol concentration was higher than its threshold in all these wines, and was therefore sensorially perceptible [82]. The eugenol levels were similar after two months of aging in all the wines supplemented with oak alternatives; however, after six months of aging the eugenol concentration was higher in the wines supplemented with the two types of staves than in the wine supplemented with oak chips. This indicates that the staves continue to release eugenol after this time, while the oak chips appear to be depleted.

Figure 4 also shows the total volatile phenol concentrations (Fig. 4.B) of the different wines. As expected, the

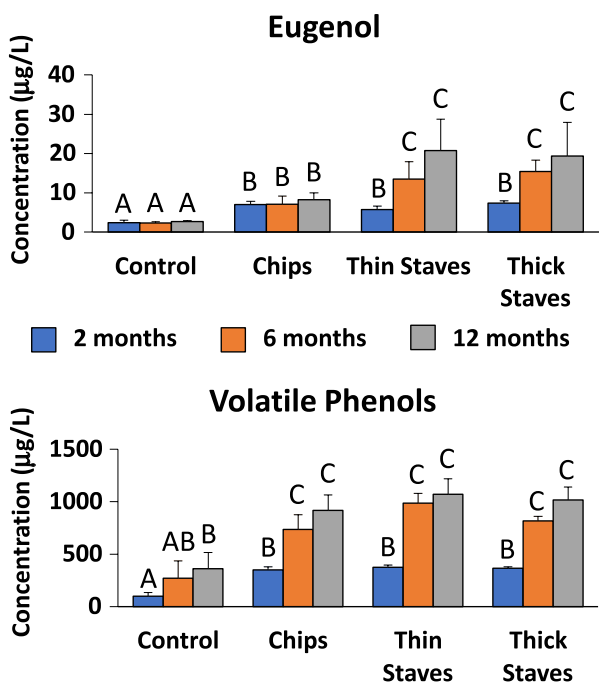


Results are expressed as mean  $\pm$  standard deviation of three replicates. Different letters indicate the existence of statistical difference ( $p < 0.05$ ).

**Fig. 3** Vanillin and total phenolic aldehydes and ketones. Results are expressed as mean  $\pm$  standard deviation of three replicates. Different letters indicate the existence of statistical difference ( $p < 0.05$ )

control wine also contained a significant concentration of total volatile phenols. It is well known that wines without contact with oak have a certain content of volatile phenols [2, 11, 55]; however, the concentration of volatile phenols increases greatly due to the action of certain microorganisms [1, 2, 83] or due to contact with oak wood [3–5, 8–10]. In fact, the concentration of total volatile phenols increased in all the wines over time. In the case of the control wine, this augmentation is only attributable to the action of microorganisms, whereas in the other wines it is attributable to both processes (microorganisms and contact with wood). In any case, no differences were found in total volatile phenols among the wines supplemented with the different oak alternatives. Although it is very difficult to determine an average threshold for all volatile phenols, it can be considered that the characteristic toasted/smoked notes of these compounds could be perceived in all the wine supplemented with oak alternatives. Furthermore, it can also be considered that the perception of these toasted/smoked notes increased similarly over time regardless of the type of oak alternative.

Figure 5 shows the total  $\beta$ -methyl- $\gamma$ -octalactones concentration (Fig. 5.A) of the different samples. As expected, these substances were not detected in the control wine,

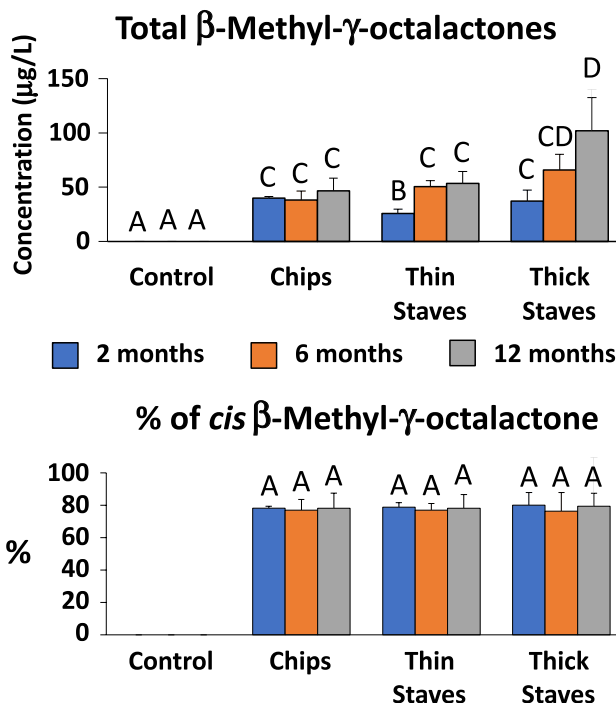


Results are expressed as mean ± standard deviation of three replicates. Different letters indicate the existence of statistical difference ( $p < 0.05$ ).

**Fig. 4** Eugenol and total volatile phenols. Results are expressed as mean ± standard deviation of three replicates. Different letters indicate the existence of statistical difference ( $p < 0.05$ )

while significant amounts were present in all the wines supplemented with oak alternatives. At two months of ageing, the total  $\beta$ -methyl- $\gamma$ -octalactones concentration in the wine supplemented with chips was similar to that of the wine supplemented with thick staves and significantly higher than that of the wine supplemented with thin staves. However, the total  $\beta$ -methyl- $\gamma$ -octalactones concentration increased as the aging time lengthened in the wines supplemented with staves, while it remained stable in the wine supplemented with chips. It therefore seems that the chips had already depleted all their  $\beta$ -methyl- $\gamma$ -octalactones after two months of ageing, while the staves continued to release it. However, the total  $\beta$ -methyl- $\gamma$ -octalactones levels at 12 months were significantly higher in the wine supplemented with thick staves than in the wine supplemented with thin staves. This indicates that thick staves have a greater releasing potential of this interesting aroma than thin staves.

Figure 5 also shows the percentage of the more aromatic *cis* isomer of  $\beta$ -methyl- $\gamma$ -octalactones. In all cases this percentage remained constant at values slightly below 80%. This value coincides with the percentage of the *cis* isomer in French oak described by other authors [4, 6, 9, 11, 65].

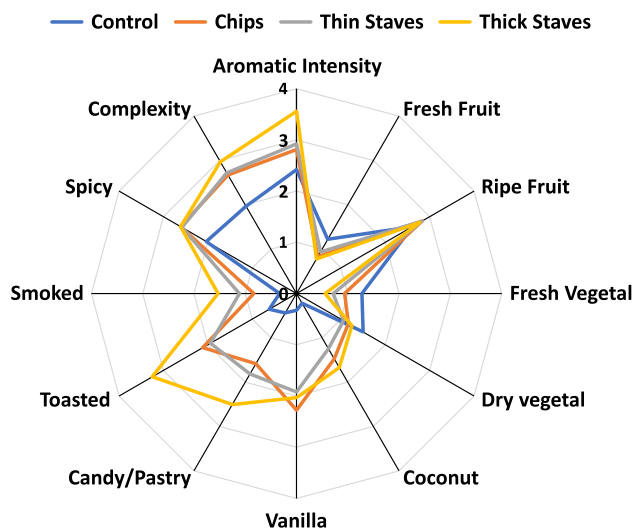


Results are expressed as mean ± standard deviation of three replicates. Different letters indicate the existence of statistical difference ( $p < 0.05$ ).

**Fig. 5** Total  $\beta$ -Methyl- $\gamma$ -octalactones and percentage of *cis* isomer. Results are expressed as mean ± standard deviation of three replicates. Different letters indicate the existence of statistical difference ( $p < 0.05$ )

**Sensory analysis**

Figure 6 shows a spider web chart to illustrate the assessment of the olfactory attributes obtained through sensory



**Fig. 6** Spider web diagram of olfactory attributes

analysis of the wines after 12 months of aging. Tasters considered that the fresh fruit, fresh vegetal, and dry vegetal attributes were more intense in the control wine than in any of the wines supplemented with oak alternatives. In contrast, the panel found that the intensity of coconut, vanilla, candy/pastry, toasted, smoked, spicy, complexity, and aromatic intensity attributes were clearly higher in all the wines supplemented with oak alternatives than in the control wine. These results are completely logical since it is well-known that contact with oak wood enriches the wine with these sensory attributes [1–3, 8–10, 16]. Tasters considered that all the wines supplemented with oak alternatives had similar intensities of coconut and vanilla. In contrast, the panellists found that the wines supplemented with the two types of staves had a higher intensity of the spicy attribute than the wine supplemented with oak chips. In addition, the panel considered that the wine supplemented with thick staves clearly had a higher score in candy/pastry, toasted, smoked, complexity, and aromatic intensity than the wines supplemented with thin staves or oak chips.

Figure 7 shows a spider web chart to illustrate the assessment of the gustatory attributes obtained through sensory analysis of the wines after 12 months of aging. Tasters considered that the sweetness, mouthfeel, structure, bitterness, and persistence of the control wine were lower than in all the wines supplemented with oak alternatives. In contrast, they found that acidity was slightly higher in the control wine. The panellists also considered that the wines supplemented with thick staves had a greater intensity in sweetness, structure, and persistence than the wines supplemented with thin staves or oak chips.

Finally, the panellists were required to rank the wines based on their order of preference. The panel preferred wine

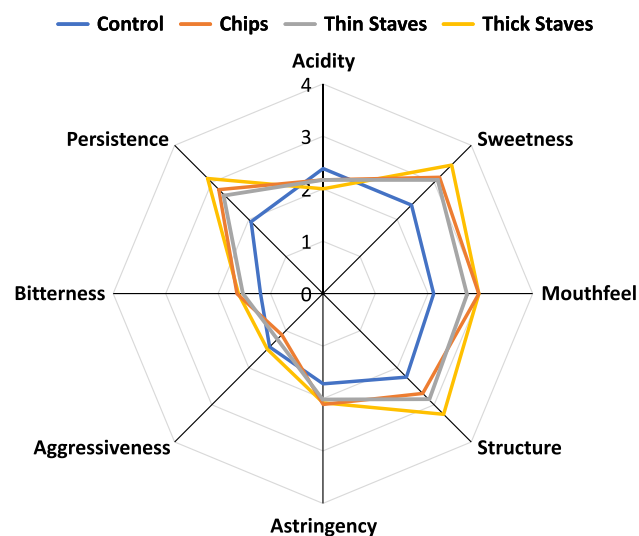


Fig. 7 Spider web diagram of gustatory attributes

aged with thick staves, followed by, in descending order, the wines aged with thin staves, oak chips, and the control.

## Conclusions

In this study, we analysed how three types of oak alternatives of different thicknesses (oak chips, thin staves, and thick staves) influence wine composition and quality. The results indicate that the supplementation with all oak alternatives had a positive effect on colour intensity although at 12 months of ageing the human eye can only perceive the difference in the two types of staves in comparison with the control wine and the wine supplemented with oak chips. This better colour can be attributable to the copigmentation effect exerted by phenolic compounds released from oak wood and also to a greater combination of anthocyanins and flavanols (PVPP Index) favoured by the presence of wood. No large differences were found in the concentration of proanthocyanidins; however, the total phenolic index and the total tannin concentration tended to be higher in wines supplemented with oak alternatives, especially in the case of the two types of staves. In general, the contact with oak alternatives enriched the wine in all the studied volatile substances and their concentration increased over time. Only slight differences were found in furfural, total furans, vanilla, total phenolic aldehydes and ketones, and total volatile phenols among the wines supplemented with the different oak alternatives. However, the eugenol concentration was significantly higher in wines supplemented with the two types of staves than in wines supplemented with oak chips after 6 months of aging. Furthermore, the  $\beta$ -methyl- $\gamma$ -octalactones concentration was higher in the wine supplemented with thick staves than in the wines supplemented with thin staves or oak chips after 12 months of ageing.

In relation to the sensory analysis, the trained panel considered that all the wines supplemented with oak alternatives had a higher intensity of coconut, vanilla, candy/pastry, toasted, smoked, spicy, complexity, and aromatic intensity attributes than the control wine. In addition, they considered that the sweetness, mouthfeel, structure, bitterness, and persistence of the control wine were lower than in all the wines supplemented with oak alternatives. However, some differences in the intensity of these attributes were found in relation to the thickness of the oak alternatives. Specifically, the wines supplemented with the two types of staves had a higher intensity of the spicy attribute than the wine aged with oak chips. Furthermore, the wine supplemented with thick staves was considered to have a higher intensity of candy/pastry, toasted, smoked, complexity, aromatic intensity, sweetness, structure, and persistence than the wines supplemented with thin staves or oak chips. Finally, the panel preferred the wine aged with thick staves followed by,

in descending order, the wines aged with thin staves, oak chips, and the control. It therefore seems that the thickness of the oak alternative plays an important role in the composition and quality of the wine because the wine is considered better when the thickness of the oak alternative is greater.

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**Data availability** The authors confirm that the data supporting the findings of this study are available within the article.

## Declarations

**Conflict of interest** Two of the authors work for Tonnellerie Radoux – Pronektar, which is the manufacturer of the oak alternatives used in this work. The rest of the authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

**Ethical approval** All authors were compliant and followed the ethical guidelines, according to the requirements of European Food Research and Technology.

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