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ORIGINAL RESEARCH ARTICLE

Study of Lignans in Wine Originating from Different Types of Oak Barrels

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ABSTRACT

This study aimed to determine the concentrations of the lignans pinoresinol, secoisolariciresinol, matairesinol and isolariciresinol in white wines aged in twelve different oak barrels originating from different cooperages and regions and with different toasting levels. In a two-year experiment the influence of oak barrels and of variety on lignan content was monitored. The results of this study show that the use of oak barrels affects only syringaresinol concentrations. Wines from low-releasing barrels contained mean concentrations of 20-50 ng/ml of syringaresinol, while wines from high-releasing barrels contained mean concentrations of 140-160 ng/ml of syringaresinol. Wines from the other barrels contained mean concentrations of 70-100 ng/ml of syringaresinol. The amounts of matairesinol and pinoresinol were affected by grapevine cultivar. In Veltliner Grün, the mean matairesinol concentration was significantly higher than in all other cultivars except for Chardonnay (mean concentrations 11, 6 and 3-5 ng/ml in Veltliner Grün, Chardonnay and other cultivars respectively). The amounts of isolariciresinol and secoisolariciresinol and total amount of lignan aglycones were affected by the interaction of cultivar and year. The mean concentrations of both lignans and total aglycone were significantly higher in Chardonnay from 2017 (210, 120 and 460 ng/ml for isolariciresinol, secoisolariciresinol and total aglycones respectively) than in all the other samples (60-130, 0-50 and 180-280 ng/ml for isolariciresinol, secoisolariciresinol and total aglycones respectively).

KEYWORDS: lignans, wine, oak barrels, wine aging

INTRODUCTION

Lignans are a group of phenolic compounds that occur in roots, leaves, seeds, fruits and wooden parts of vascular plants (Ayres and Loike, 1990). As a class of secondary plant metabolites produced from shikimic acid via the phenylpropanoid pathway, they play an important role in plant defence against various biological pathogens and pests, and they participate in plant growth (Ayres and Loike, 1990). Due to their antitumour, anti-inflammatory, immunosuppressive, cardiovascular, antioxidant and antiviral effects, lignans are the subject of extensive studies, for use in and beyond the pharmaceutical industry (Satoshi *et al.*, 2004).

Most plant lignans and lignan glycosides are converted by the intestinal microflora in the upper part of the large bowel into enterolactone and enterodiol, called mammalian or enterolignans. Pinoresinol is converted to lariciresinol and further metabolised to form secoisolariciresinol and matairesinol, which are converted into enterodiol and its oxidised form enterolactone. In addition, syringaresinol is converted into enterolignans (Heinonen *et al.*, 2001). Enterolignans can reduce the risk of certain cancers and cardiovascular diseases due to their antioxidative, estrogenic/antiestrogenic, and antiproliferative properties (Adlercreutz, 2007; Borriello *et al.*, 1985; Setchell *et al.*, 1981).

Some lignans have also been identified in wine: secoisolariciresinol, lariciresinol and isolariciresinol with different conjugations have been identified in Riesling white wine, and secoisolariciresinol, lyoniresinol and matairesinol have been quantified in a few different red wines (Baderschneider and Winterhalter, 2001; Mazur, 1998; Mazur and Adlercreutz, 2000).

In winemaking practices, various oenological processes are often used, such as maceration and fermentation on the skins and stems, which help to increase the extraction of substances that have a positive effect on not only the sensory aspects of wine but also its health effects. In addition to lignan being naturally extracted from various parts of the vine (skin and grape seeds), oak and chestnut wood is used in oenology in the form of chips or wooden barrels, as it is has been found to be an important source of lignans. Several studies have focused on wine and must enrichment by lignans that originated from different wood parts; for example, from spruce knots (Balik *et al.*, 2016; Novotna *et al.*, 2016). In a recent study by Dadakova *et al*. (2021) the origin of lignans in wine was evaluated: resinol-related lignans in must, seeds, stems and wine prepared using stainless steel tanks, oak barrels and qvevri were analysed. The results of this study showed that lignan content in wine can be increased by maturation in contact with grape skins, seeds or stems, or with wood. The contact of wine with wood is one of the ways to naturally increase the content of lignans in wine. Many studies dealing with the study of lignans in oak wood have identified lignans like isolariciresinol, secoisolariciresinol and lyoniresinol, the latter lignan being responsible for a bitter taste (Arramon *et al.*, 2002; Marchal *et al*., 2011; Moutounet *et al*., 1989; Nabeta *et al*., 1987; Nonier *et al*., 2009).

Oak barrels are widely used in oenology due to their impact on aging aroma, colour stabilisation and modulation of sensory perceptions of wine. Therefore, this study focused on the influence of the type of oak barrel on the lignan content of wine (Glabasnia and Hofmann, 2006; Chassaing *et al.*, 2010; Chatonnet *et al.*; Quinn and Singleton, 1985; Ribéreau-Gayon *et al.*, 2006; Tominaga *et al.*, 2000).

The novelty of this study is that we aimed to examine which lignans are released from which type of oak barrel. In the two-year experiment, twelve different oak barrels were used in which five varieties of wine were matured. A total of 108 wine samples were analysed.

MATERIAL AND METHODS

1. Design of experiment

A two-year experiment was performed, in which twelve different types of oak barrels were used for the production of wines from five grape varieties: Sauvignon blanc (SG), Veltliner Grün (VG), Welschriesling (WR), Palava (PA) and Chardonnay (CH). The grapes were harvested manually. Gallic tannin and pyrosulphite (5 + 5 g / 100 kg of grapes) were used in the crushing process. This was followed by pressing, and only free-run must was used for further processing. The must was clarified by sedimentation using Seporit PORE-TEC (ERBSLÖH Geisenheim, Germany) bentonite (80 g / hl). Active dry yeast Oenoferm Klosterneuburg (*Saccharomyces cerevisiae* LW 415-58, ERBSLÖH Geisenheim, Germany) was used for fermentation. Alcohol fermentation and spontaneous malolactic fermentation took place entirely in barrels. The wine were matured on yeast lees. 20 mg/L of SO_2 was added. The wines were matured in barrels for twelve months.

Characterisation of oak barrels:

All of the oak barrels that were used had the following dimensions: height: 94 cm, diameter of the widest part: 69 cm, front diameter: 58 cm, wood thickness: 27 mm. They were used twice: first in 2016 then in 2017. The types of oak barrels differed in terms of origin of the oak, duration of wood maturation and degree of toasting, and some were treated by the Aquaflex method after toasting. A total of 60 oak barrels were used for this study. A characterisation of each of the twelve types of barrel is given in Table 1.

2. Determination of basic analytical parameters

The basic parameters of the resulting wine (alcohol, pH, residual sugar, total acidity and volatile acidity) were determined with an Alpha FTIR analyser (Bruker, Germany) using the attenuated total reflection (ATR) sampling technique. Before the first measurement, the spectrometer was thoroughly rinsed with deionised water and the background was determined using a blank sample (deionised water). For the analyses, 1-mL samples were taken with a syringe;

Table 1. Characterisation of each oak barrel type.

Barrel	Characterisation
Ermitage CM B	Medium toasting, wood originated from 8000 ha part of forest in Bertrange in the area of Nevers, Q.Petraea, fine grain, wood maturation at least 24 months (Ermitage).
Berthomieu CM N	Medium burning, wood originated from Nevers region, fine grain, wood maturation at least 24 months (Berthomieu).
Cadus Access	Medium burning, wood originated from different regions in France, for white and red French-style wines, fine grain, wood maturation at least 30 months (Cadus).
Cadus Equilibre	Medium burning, wood originated from different regions in France, intended primarily for white wines, light, fresh, mineral-style of wine, fine grain, wood maturation at least 30 months (Cadus).
Nadalie White Pearls	Fruité burning, wood originated from central France, intended primarily for fruit-style of white wines, wood maturation at least 24 months (Nadalie).
Francois Freres BM	Medium burning, wood originated from Bertrange, fine grain (Freres). More than 30 months of wood maturation.
Stockinger Y LDL	Long-deep-light burning, wood originated from Ybbstaler-Austria (Stockinger).
Stockinger P LDL	Long-deep-light burning, wood originated from Palatinate-Germany (Stockinger).
Seguin Moreau Fraicheur ML TH	Medium-long burning including front and back part of the barrel, Aquaflex process, wood originated from different parts of France, acacia foreheads, intended for fresh and mineral style of white wines (Moreau).
Seguin Moreau Icon Blanc ML Aquaflex	Medium-long burning, Aquaflex process, wood originated from different parts of France, intended for fresh and mineral-style of wines (Moreau).
Seguin Moreau Icon Blanc ML	Medium-long burning, wood originated from different parts of France, designed for opulent and complex white wines with a spicy and toast tone (Moreau).
Seguin Moreau Caucasian classic M	Medium burning, wood originated from Caucasus, fine grain (Moreau).

0.5 mL was used to rinse the system while the remaining volume of 0.5 mL was analysed three times. The measured values were evaluated automatically using the OpusWine software.

Determination of free and total SO_2 was performed by iodometric titration using a standard iodine solution (Balík, 2004).

3. Determination of lignans by HPLC

Lignan standards were purchased from PhytoLab (matairesinol and pinoresinol, Vestenbergsreuth, Germany), Biopurify Phytochemicals Ltd. (isolariciresinol, Chengdu, China), ChemFaces (syringaresinol, Wuhan, China) and Sigma-Aldrich (secoisolariciresinol and enterolactone, Darmstadt, Germany).

The wine samples were diluted with 0.01 % acetic acid in water (1:1 v/v), spiked with 400 ng/mL enterolactone, filtered with 0.2 µm filter and subjected to LC-MS analysis.

The samples were analysed by LC-MS (6545, Agilent, Santa Clara, CA, USA) using a method published previously (Dadakova *et al.*, 2021). Briefly, analytes were separated using a reverse-phase column (EclipsePlus C18, 2.1 × 50 mm^2, 1.8 µm, Agilent, Santa Clara, CA, USA) and a gradient elution. The mobile phases used were 0.01 % acetic acid in water (A) and methanol (B). The gradient conditions were as follows: 5 to 20 % B from 0 to 10 min, 20 to 100 % B from 10 to 20 min, 100 % from 20 to 30 min and finally, 5 % B from 30 to 40 min at a flow rate of 0.3 mL/min and injection volume of 1 µL. Electrospray ion source was used under the following conditions: acquisition mode 100–1700 m/z, gas temperature 300 °C, gas flow 8 L/min, ion polarity negative, capillary voltage 4000 V and fragmentor voltage 150 V.

The lignans were identified by comparing obtained retention times and mass spectra with those of respective standards, and they were quantified using peak areas of $[M - H]^-$ions, except for lyoniresinol that was semi-quantified using peak area of extracted ion chromatogram combining $[M - H]^-$, $[M + CH_3COO]^-$ and $[M + 2\ CH_3COO]^{2-}$ ions (Table 2). Enterolactone was chosen as the internal standard on the grounds of its similarity to the analytes and assuming its absence in the samples. Calibration curves were drawn using standards in MassHunter Quantitative Analysis software (Agilent, Santa Clara, CA, USA).

4. Statistical evaluation

Statistical analyses and figures were generated using Excel software (manufactured by Microsoft Office, USA) and Statistica 10 statistical software (Copyright © StatSoft). Differences in lignan concentrations between wine samples were assessed using one-way ANOVA and Tukey HSD post-hoc test.

TABLE 2. m/z ratios of the analytes.

Analyte	[M – H]– (*m/z*)	Retention Time (min)
Isolariciresinol	359.1500	16.8
Matairesinol	357.1344	18.4
Pinoresinol	357.1344	18.2
Secoisolariciresinol	361.1657	17.7
Syringaresinol	417.1555	18.1
Lyoniresinol	419.1701	16.3
Enterolactone	297.1132	18.7

RESULTS AND DISCUSSION

1. Determination of basic analytical parameters

In Table 3, the basic analytical parameters of the final wine after maturation in the oak barrels are presented. Residual sugar concentration is expressed in equivalents of glucose and fructose, total acidity in equivalents of tartaric acid, and volatile acidity in equivalents of acetic acid. The results are average values ± standard deviation. There were no significant differences between the concentrations of alcohol of all the different variants. It can therefore be assumed that the values of the alcohol content in the wine had no effect on the release of lignans into the different variants of wine. Differences in terms of free sulfur dioxide content were only found between the Sauvignon and Welsriesling varieties.

2. Determination of lignans by HPLC

Lignans, namely isolariciresinol, secoisolariciresinol, matairesinol, pinoresinol and syringaresinol, were quantified in the wine (Figure 1).

The amounts of syringaresinol were affected by the barrel used. Generally, the barrels can be divided into three groups according to the amount of syringaresinol released from the barrel. The wines from the low-releasing barrels (Stock P LDL, Stock Y LDL and SM Icon Blanc Aqafl. ML) contained mean concentrations of 20-50 ng/ml of syringaresinol. Meanwhile, the wines from the high-releasing barrels (SM Icon Blanc ML, SM Caucasus, SM Fraicheur ML TH and FF B M) contained mean concentrations of 140-160 ng/ml of syringaresinol. The wines from the other barrels contained mean concentrations of 70-100 ng/ml of syringaresinol (Figure 2).

In general, the concentration of lignan present in wood is influenced by many factors: type of wood, locality of growth and maturation time. The SM Icon Blanc ML, SM Caucasus, SM Fraicheur ML TH and FF B M barrels were produced using the medium-long burning method. An FF BM producer will usually mature the wood for barrel production for a longer period of 30 months or more, we can thus assume that the highest concentration of the lignan in FF BM is also influenced by the longer period of wood maturation.

A previous study of different winemaking practices found a high concentration of syringaresinol in Veltliner Grün, a wine aged in qvevri; however, the wine was not aged in steel tanks (Dadakova *et al.*, 2021). Qvevri wines are fermented

TABLE 3. Basic analytical parameters of final wines.

	Alcohol (vol.%)	Sugar (g/L)	Total acidity (g/L)	volatile acidity (g/L)	pH	SO_2 free (mg/L)	SO_2 total (mg/L)
SG 2016	13.51 ± 0.08	0.52 ± 0.37	6.12 ± 0.11	0.56 ± 0.09	3.42 ± 0.02	12.1 ± 0.33	50.3 ± 0.88
SG 2017	13.32 ± 0.07	0.57 ± 0.59	5.41 ± 0.19	0.25 ± 0.07	3.35 ± 0.02	20.3 ± 0.80	55.7 ± 0.72
WR 2016	14.21 ± 0.11	1.09 ± 0.23	3.71 ± 0.22	0.49 ± 0.11	3.58 ± 0.08	15.6 ± 0.25	60.3 ± 0.86
WR 2017	14.62 ± 0.21	1.32 ± 0.31	5.12 ± 0.19	0.32 ± 0.14	3.39 ± 0.02	20.2 ± 0.13	57.3 ± 0.25
CH 2016	14.17 ± 0.09	0.81 ± 0.13	4.23 ± 0.15	0.55 ± 0.26	3.65 ± 0.05	20.3 ± 0.50	80.2 ± 0.81
CH 2017	14.43 ± 0.23	0.34 ± 0.41	4.82 ± 0.23	0.26 ± 0.08	3.58 ± 0.04	20.5 ± 0.69	80.4 ± 0.74
VG 2016	13.71 ± 0.10	0.43 ± 0.26	6.51 ± 0.17	0.65 ± 0.15	3.42 ± 0.09	25.2 ± 0.25	80.1 ± 0.35
VG 2017	14.12 ± 0.08	1.54 ± 0.15	4.92 ± 0.11	0.38 ± 0.23	3.41 ± 0.02	25.1 ± 0.13	80.3 ± 0.27
PA 2016	14.33 ± 0.21	1.82 ± 0.18	4.67 ± 0.19	0.60 ± 0.17	3.42 ± 0.02	25.6 ± 0.08	80.9 ± 0.64

Average values and standard deviations of 12 replicates.

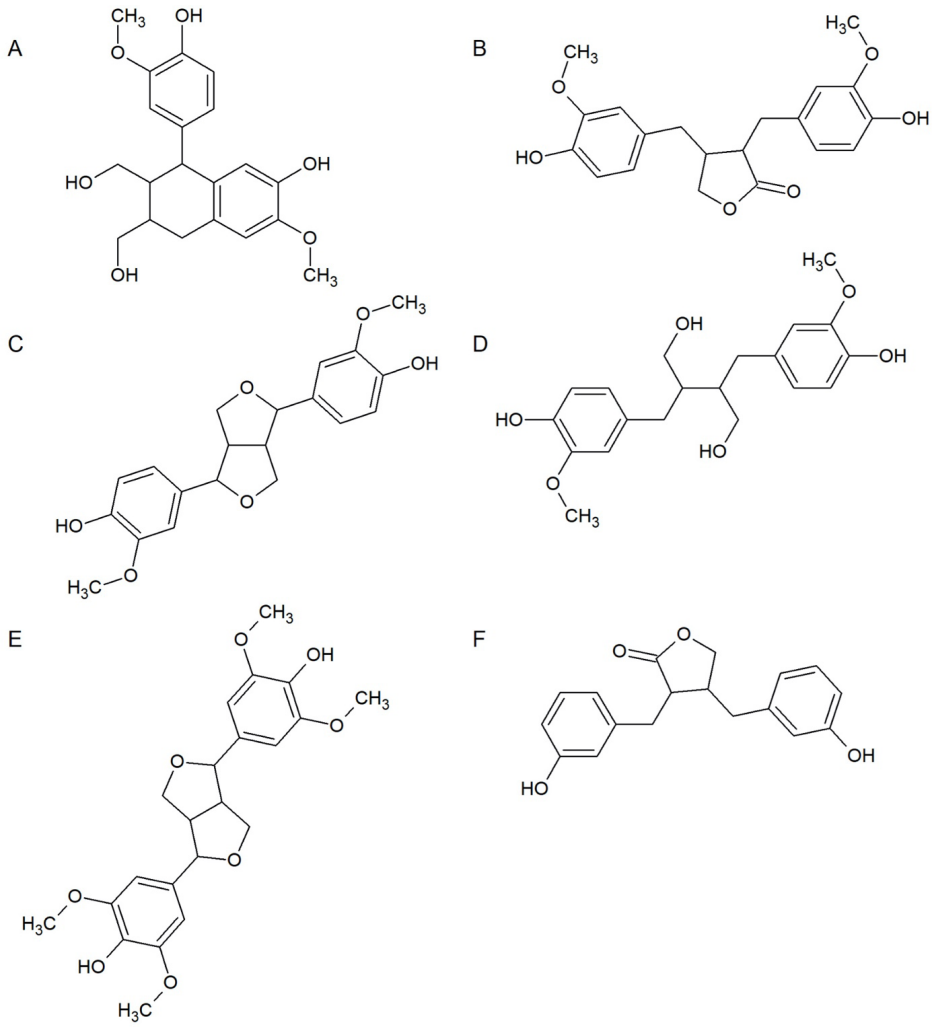


FIGURE 1. Lignan structures. A) isolariciresinol, B) matairesinol, C) pinoresinol, D) secoisolariciresinol, E) syringaresinol, and F) enterolactone.

in contact with some stems; it can therefore be assumed that this lignan, which is not commonly found in white wines, is released into the wine from other parts of the grapevine. Furthermore, a significantly higher amount of syringaresinol was found in a red variety of pinot noir aged in an oak barrel compared to that aged in a steel tank. Therefore, we hypothesise that a significant proportion of syringaresinol is released into the wine from oak wood. Moreover, in a study by Seikel *et al.* (1971), syringaresinol was found in oak wood in high concentrations.

Certain conditions, such as the length and intensity of wood toasting during barrel production, can have an effect on the concentration of lignans in the wood, which can then be released into the wine.

A study by Sanz *et al*. (2012) (Sanz *et al.*, 2012) compared the phenolic composition, including lignans, of acacia (*Robinia pseudoacacia*), chestnut (*Castanea sativa*), cherry (*Prunus avium*) and ash (*Fraxinus excelsior* and *F. americana*) heartwoods by HPLC-DAD/ESI-MS/MS, and identified the changes to each polyphenolic profile as a result of toasting intensity at cooperage. In general, toasting notably changed qualitative and quantitative polyphenolic profiles in proportion to toasting intensity, and led to a minor differentiation among species in toasted woods.

In a study by Windeisen and Wegener (2009), syringaresinol was one of two compounds that significantly increased in concentration during toasting, with no differences between the two species; the results of the study indicate that syringaresinol is a very stable compound thermally.

The major oak lignan lyoniresinol has been the subject of several previous studies (Cretin *et al.*, 2021; Marchal *et al.*, 2015) and was not studied in detail in this study. However, it was putatively identified and semi-quantified in the samples (Figure 3). The peak areas were affected by the barrel used. Interestingly, the barrels that seemed to release high amounts of lyoniresinol (Stockinger P LDL, Seguin Moreau Caucasian classic M, Stockinger Y LDL, Ermitage CM B, and Seguin Moreau Icon Blanc ML Aquaflex) generally released low

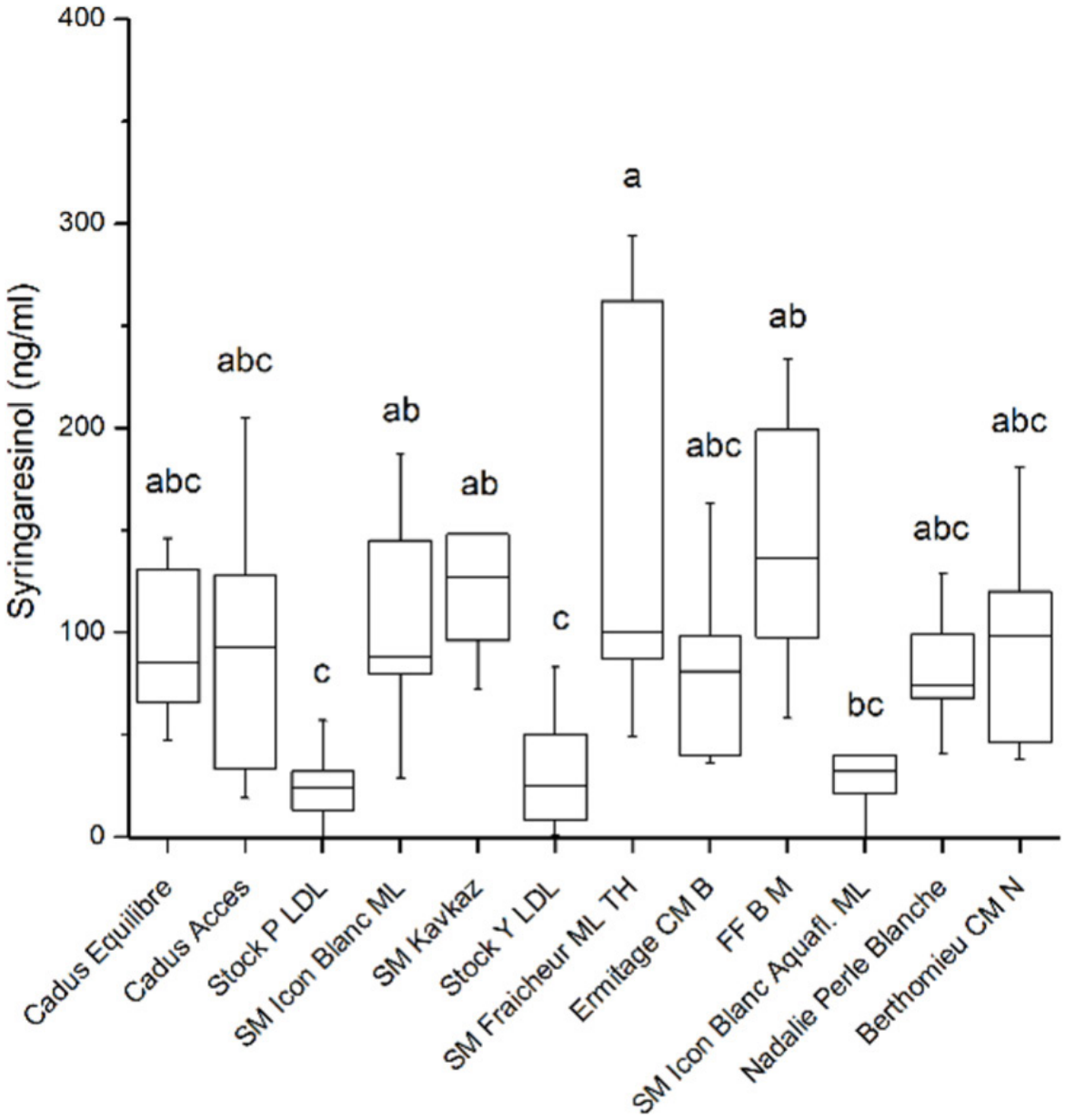


FIGURE 2. Syringaresinol concentrations in wines from different types of oak barrel.

*Results are shown in boxes (25-75 percentile), with horizontal lines showing medians and whiskers showing min-max (excluding outliers) of 9 replicates. Different letters indicate statistically significant differences ($p < 0.05$), as estimated using ANOVA with Tukey HSD post-hoc test.

amounts of syringaresinol; conversely, the barrel that seemed to release low amount of lyoniresinol (Seguin Moreau Fraicheur ML TH) released high amount of syringaresinol.

Based on our results and previous studies, it can therefore be assumed that the concentrations of lyoniresinol and syringaresinol in wine increase after contact with wood, and in addition, the type of oak barrel used has an effect on their release into the wine.

Interestingly, the peak areas of the compound putatively identified as lyoniresinol were affected not only by the barrel used but also by the grapevine cultivar (Figure 3B). On the other hand, they were not affected by the interaction between the cultivar and the barrel. This indicates that the difference in lyoniresinol release rate between each cultivar is not related to the oak barrel but rather to the grape of the different cultivars. Specifically, the Palava variety was the richest in lyoniresinol, with a significantly higher lyoniresinol content than Sauvignon blanc and Welschriesling. Therefore, we can assume that part of the lyoniresinol contained in the wine comes from the barrel and part comes from the grape. This hypothesis is supported by our data from our previous study, in which lyoniresinol was putatively identified in grape stems (data not shown).

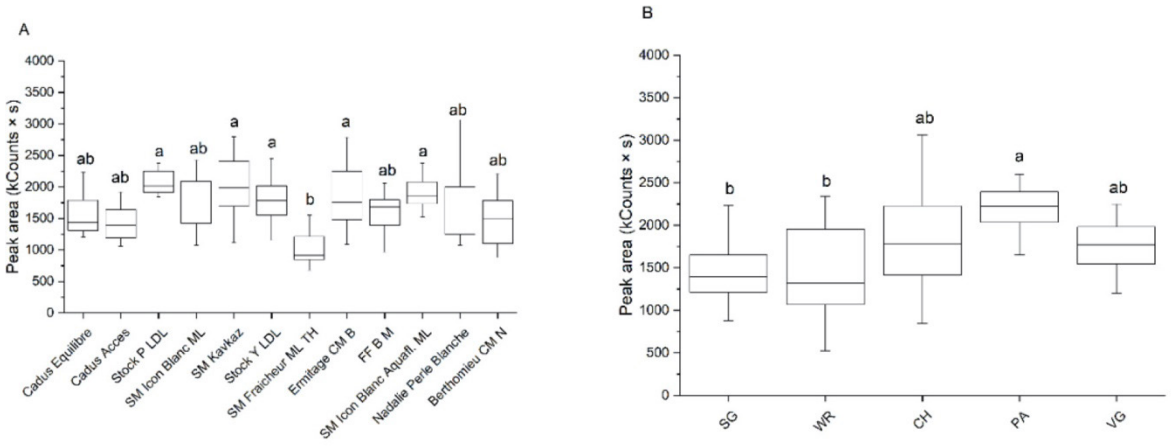


FIGURE 3. Peak areas of extracted ion chromatograms for compound putatively identified as lyoniresinol in wines from different oak barrel types (A) and different cultivars (B).

*Results are shown in boxes (25-75 percentile), with horizontal lines showing medians and whiskers showing min-max (excluding outliers) of 9 replicates (A) and 24 replicates (B, 12 for PA). Different letters indicate statistically significant differences ($p < 0.05$), as estimated using ANOVA with Tukey HSD post-hoc test.

The amounts of matairesinol and pinoresinol in the wines were affected by grapevine cultivar (Figure 4). In Veltliner Grün (VG) wine, the mean matairesinol concentration was significantly higher than in that of all the other cultivars, except for Chardonnay (CH) (mean concentrations of 11, 6 and 3-5 ng/ml in VG, CH and the other cultivars respectively). In contrast to the wines of other cultivars, the Palava wines contained higher amounts of pinoresinol (mean concentration 5 ng/ml).

In a recent study by Dadakova *et al.* (2021), matairesinol was found in low concentrations (less than 12 ng/mL, 9 ng/ mL being the limit of quantification of the method) in all the samples of wine processed by different winemaking methods, with the exception of Traminer Rot, which had been aged in a steel tank. This lignan was not found in must or in the other parts of the grapevine (seeds or stems) (Dadakova *et al.*, 2021). Previous studies found matairesinol in both white and blue grapevine berries and its concentration was dependent on the grape variety. (Kuhnle *et al.*, 2007; Milder *et al.*, 2005; Valsta *et al.*, 2003). Matairesinol was found in red wines and to a lesser extent in white wines, meaning we can hypothesise that this lignan is mainly released from the berry skin (Mazur, 1998; Mazur and Adlercreutz, 2000; Nurmi *et al.*, 2003). Some resinol-related lignans, such as matairesinol, have also been isolated from the barks and woods of different Quercus species (Dada *et al.*, 1989; Kuridze *et al.*, 1981; Marchal *et al.*, 2011; Sohretoglu *et al.*, 2014; Sohretoglu and Renda, 2020). Based on the results of our study, it can be assumed that the concentration of matairesinol in wine can be increased due to the releasing from the barrels, independent of the type of barrel.

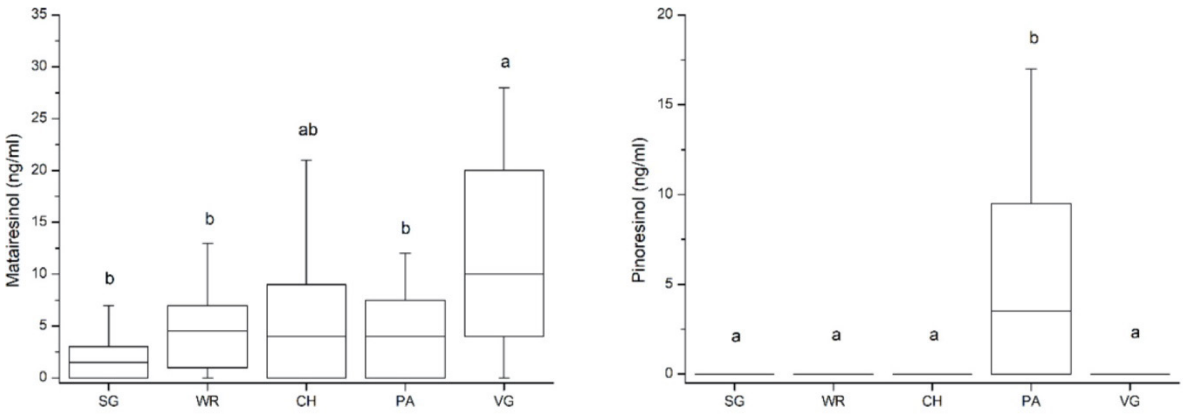


FIGURE 4. Matairesinol and pinoresinol concentrations in wines from different cultivars.

*Results are shown in boxes (25-75 percentile), with horizontal lines showing medians and whiskers showing min-max (excluding outliers) of 24 replicates (12 for PA). Different letters indicate statistically significant differences ($p < 0.05$), as estimated using ANOVA with Tukey HSD post-hoc test.

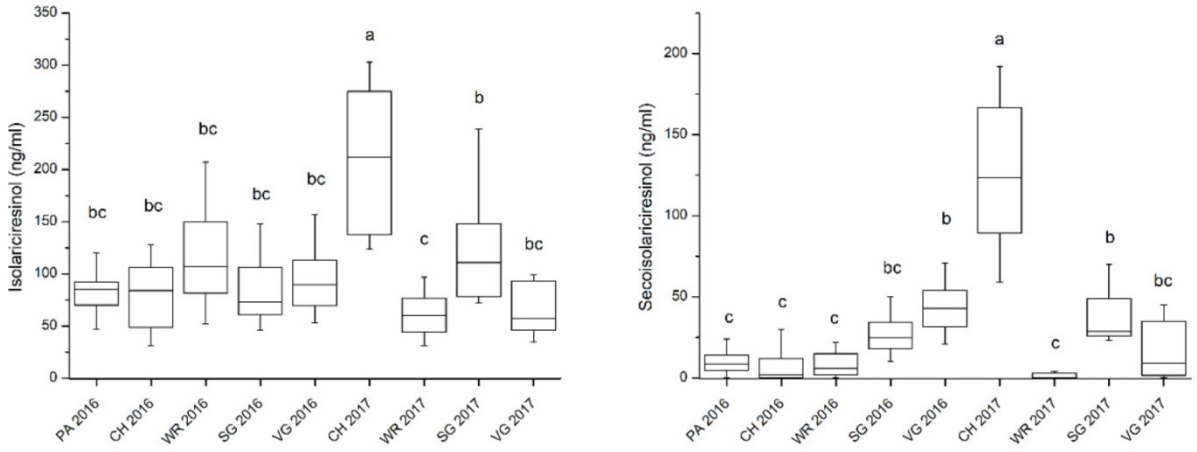


FIGURE 5. Isolariciresinol and secoisolariciresinol concentrations in wines from different cultivars and years.*Results are shown in boxes (25-75 percentile), with horizontal lines showing medians and whiskers showing min-max (excluding outliers) of 12 replicates. Different letters indicate statistically significant differences ($p < 0.05$), as estimated using factorial ANOVA with Tukey HSD post-hoc test.

Pinoresinol was found only in the Palava variety. In previous studies, this lignan was found only in the samples of pinot noir seeds. According to previously reported results, this lignan is apparently not released into the wine (Cecchi *et al.*, 2019, Dadakova *et al.*, 2021). Based on the results of our study, it can be assumed that the type of oak barrel did not affect the concentrations of matairesinol and pinoresinol and their concentrations are dependent on the variety.

The amounts of isolariciresinol and secoisolariciresinol, as well as the total amount of lignan aglycones, were affected by the interaction of cultivar and year (Figure 5). The mean concentrations of the lignans and total aglycones were significantly higher in CH from 2017 (mean concentrations of 210, 120 and 460 ng/ml for isolariciresinol, secoisolariciresinol and total aglycones respectively) than in all the other samples (60-130, 0-50 and 180-280 ng/ml for isolariciresinol, secoisolariciresinol and total aglycones respectively). Furthermore, the mean concentration of isolariciresinol was higher in Sauvignon 2017 than in Welschriesling 2017. The mean concentration of secoisolariciresinol was higher in VG 2016 and SG 2017 than in PA, WR from both years and CH 2016. The influence that the year has on concentrations is indicated by the results from the Chardonnay variety: isolariciresinol and secoisolariciresinol contents were statistically significantly higher in the 2017 vintage when a given barrel was used for a second time (the first being in 2016); it can therefore be assumed that the higher amount of these lignans was not due to them being released from the barrel but rather to an influence of the year 2017. Higher concentrations of secoisolariciresinol were also measured in the variant SG 2017 than in SG 2016. For the other varieties, the influence of year was not observed.

In a recent study, isolariciresinol was found in higher concentrations in wine samples produced by the qvevri method (Dadakova *et al.*, 2021); these concentrations of isolariciresinol were likely significantly increased due to its transfer from seeds or stems during maturation. Isolariciresinol was the most abundant lignan in wines in a study by Nurmi *et al.* (2003), representing – on average – 63 % of the total hydrolysed lignans. Isolariciresinol was also identified in a study by Baderschneider and Winterhalter (2001), in which flavonoids and lignans in Riesling wine were evaluated.

A higher concentration of secoisolariciresinol was found in the CH 2017 variety. This lignan was also found in a previous study in the samples of pinot noir wine, in pinot noir seeds and in the samples of qvevri wines. Therefore, we can assume that maturation in an oak barrel can also increase its concentration. Secoisolariciresinol has previously been found in both white and blue grapevine berries, but there was no specification as to which part of the berry (Kuhnle *et al.*, 2007; Milder *et al.*, 2005; Valsta *et al.*, 2003).

The results of our study indicate that the concentration of isolariciresinol and secoisolariciresinol is not influenced by the type of barrel used, but rather depends on the variety; in the Chardonnay and Sauvignon varieties, the influence of the year has also been confirmed. Previous studies confirm that these two lignans are commonly found in varying concentrations in wine and grape berries.

In many studies, the composition of lignan from *Quercus petraea* oak wood has been described. The results of these studies indicate that lyoniresinol lignans are the major lignans in oak wood, but isolariciresinol and secoisolariciresinol have also been identified as minor lignan components. (Marchal *et al.*, 2011; Nonier *et al.*, 2009; Nurmi *et al.*, 2003; Sohretoglu *et al.*, 2014; Sohretoglu and Renda, 2020). This is also confirmed by the results of our study, in which secoisolariciresinol and isolariciresinol were found in all the wine samples, but their concentration was not significantly influenced by the type of barrel used.

CONCLUSION

This study aimed to determine the concentrations of the lignans syringaresinol, pinoresinol, secoisolariciresinol, matairesinol and isolariciresinol in white wines aged in twelve different oak barrels. The major oak lignan lyoniresinol was putatively identified and semi-quantified in the samples of wine. The results of this study show that syringaresinol concentrations were affected by the barrel used. Generally, the barrels can be divided into three groups according to the amount of syringaresinol released from the barrel. The wines from low-releasing barrels were Stock P LDL, Stock Y LDL and SM Icon Blanc Aqafl. ML; and the wines from high-releasing barrels were SM Icon Blanc ML, SM Caucasus, SM Fraicheur ML TH and FF BM.

The amounts of matairesinol and pinoresinol were affected by grapevine cultivar. In Veltliner Grün, the mean matairesinol concentration was significantly higher than in all other cultivars except for CH. In contrast to the other cultivars, PA wines contained low amounts of pinoresinol.

The amounts of isolariciresinol and secoisolariciresinol in the wines, as well as the total amount of lignan aglycones, were affected by the interaction of cultivar and year. The mean concentrations of both these lignans were significantly higher in CH from 2017 than in all the other samples.

The peak areas of the compound putatively identified as lyoniresinol were affected by the barrel used. The barrels that seemed to release high amounts of lyoniresinol generally released low amounts of syringaresinol, and conversely the barrel that seemed to release low amounts of lyoniresinol released high amounts of syringaresinol. The peak areas of lyoniresinol were affected not only by the barrel used but also by the grapevine cultivar, and they were not affected by the interaction of the cultivar and the barrel.

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