



Influence of Oak Wood Seasoning Duration and Toasting Degree on Endogenous and Wood-Derived Compounds of *Brandy Italiano*

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Abstract

The Geographical Indication (GI) “*Brandy Italiano*” applies exclusively to brandy produced in Italy through the distillation of wine obtained from grapes cultivated and vinified within the national territory. Its product specification requires a minimum aging of six months in oak casks smaller than 1000 L to be marketable. During aging, the characteristics of the final product are affected by the different treatments applied to oak wood during the cooperage process. This study aimed to assess the impact of different oak wood toasting levels and natural seasoning durations on the whole volatile profile of this GI, including wood-derived compounds as well as varietal and fermentative volatiles. The research was conducted on brandies from *Trebbiano Romagnolo* wines distilled via two-step distillation and aged for 8 months in 350-L oak barrels. Cooperage woods were naturally seasoned for 24 or 36 months before being toasted under light or heavy conditions. The products were analysed via solid-phase extraction coupled with gas chromatography–mass spectrometry to determine their overall volatile composition and via high-performance liquid chromatography with photodiode array and fluorescence detection to quantify wood-derived compounds. Heavy toasting significantly increased the content of most wood-derived aromatic compounds, particularly furan compounds and phenolics from lignin degradation, and induced the formation of 5-acetoxymethyl furfural, a potential marker of short-term aging in distillates from heavily toasted wood. Seasoning duration affected the levels of gallic and ellagic acids, as well as the ratio between *cis*- and *trans*-methyl- γ -octalactones. The wood treatments also influenced certain varietal and fermentation aromas, including terpenes and ethyl esters.

Keywords *Brandy Italiano* · Wood toasting · Wood seasoning · Volatile compounds · Phenolic composition

Introduction

The Geographical Indication (GI) “*Brandy Italiano*” is exclusively reserved for brandy produced in Italy through the distillation of wine made from grapes grown and vinified within the national territory (Ministero delle politiche agricole, alimentari e forestali, 2016; European Parliament and Council of the European Union, 2019). The aromatic profile of brandy derives from a complex interplay of volatile compounds generated throughout its production stages,

beginning with grape selection and vinification, continuing through distillation, and culminating in barrel aging. Primary aroma compounds, such as monoterpenes, benzenoids, and C₁₃-norisoprenoids, originate from the grapes and are influenced by environmental factors and viticultural practices (Flamini & Traldi, 2009a), whereas secondary aroma compounds, including higher aliphatic alcohols, ethyl and acetate esters, and fatty acids, are formed during alcoholic and malolactic fermentations (Flamini & Traldi, 2009b). The distillation process extracts and concentrates volatile compounds to preserve desirable aromas while minimizing off-flavours (Tsakiris et al., 2014). During this phase, additional compounds may also be formed, including ethoxy derivatives, acetals, terpenols, and furans (Mayr Marangon et al., 2021; Zhao et al., 2011). The final stage in brandy production is aging in oak barrels, during which multiple phenomena, including chemical reactions and physical transformations, may occur involving both compounds extracted from the wood and those already present in the distillate

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(Cruz et al., 2013; De Rosso et al., 2009; Mosedale & Puech, 1998; Tsakiris et al., 2014; Van Jaarsveld & Hattingh, 2012). Cooperage practices, particularly wood seasoning and barrel toasting, are considered important factors that may influence these phenomena (Canas, 2017). Natural seasoning is generally carried out in open air over a period of 18 to 36 months, during which the wood loses excess moisture and undergoes chemical changes, including the leaching of water-soluble compounds and a decrease in ellagitannin content (Dousot et al., 2002; Fernández de Simón et al., 2010; Martínez et al., 2008). The subsequent toasting process, commonly classified as light, medium, or heavy on the basis of the temperature–time combination, induces more pronounced chemical transformations within the wood matrix. These modifications, whose extent strongly depends on charring conditions (Canas, 2017) result in the formation of water- and alcohol-soluble aromatic compounds such as aldehydes (e.g., vanillin, syringaldehyde, sinapaldehyde, coniferaldehyde), cinnamic and benzoic acids, and furan derivatives (Canas, 2017; Conner et al., 1992; Mosedale & Puech, 1998). Although considerable research has examined the impact of wood treatments on the chemical composition of wood used in oenology (Alanon et al., 2010; Le Floch et al., 2015), studies specifically addressing their influence on the chemical properties of wine brandies remain relatively scarce. Most investigations have focused on French (Cantagrel et al., 1993; Rabier & Moutounet, 1990) and Spanish brandies (Artajona et al., 1990; Belchior et al., 2001; Guerrero-Chanivet et al., 2023), primarily examining wood-derived compounds and toasting parameters, whereas the role of wood seasoning has received comparatively less attention. Given the outlined context, the present study investigated the effects of wood seasoning duration and toasting level on the volatile profile of *Brandy Italiano*. Focus was placed not only on wood-derived compounds but also on the evolution of varietal and fermentative aroma constituents. The research was conducted on brandies aged for eight months in 350-L barrels made from French Limousin oak. The oak wood used for barrel production was naturally seasoned for either 24 or 36 months and subjected to two distinct toasting levels, light or heavy. Most wood compounds were quantified using HPLC–DAD–FLD, while SPE–GC–MS was employed to analyze both endogenous and other individual wood-derived volatile compounds.

Materials and Methods

Wood Barrels and Brandy Samples

All brandy samples were produced and provided by Villa Zarrì S.R.L. (Castel Maggiore, Emilia Romagna, Italy)

using wines made from grapes of the *Trebbiano Romagnolo* cultivar.

Wine distillation was performed using a two-step discontinuous method with a Charentais-type alembic, yielding a wine distillate with an ethanol content of approximately 70% v/v. Prior to the aging process, the ethanol concentration was adjusted to 60% v/v by dilution with distilled water.

Aging was conducted in four new 350-L barrels made from French Limousin oak, naturally seasoned for either 24 or 36 months and subsequently toasted at two intensity levels: light (60 min at 130 °C) or heavy (50 min at 260 °C). The four barrels were filled with the raw wine distillate adjusted to 60% v/v ethanol, while an unaged distillate sample was retained as a control (t_0). During aging, the cellar temperature was maintained at 18 °C. After eight months of aging, 50 mL of distillate was collected from each barrel and diluted to 40% v/v ethanol with distilled water. The samples were designated as L24, L36, H24, and H36, on the basis of the toasting intensity (L = light, H = heavy) and seasoning duration (24 or 36 months) of the barrel wood in which they were aged. Each sample was analyzed in triplicate.

Chemicals and Standards for SPE–GC–MS Analysis

Reference standards for compound identification were supplied by Sigma–Aldrich (St. Louis, MO, USA), Fluka Chimie AG (Buchs, Switzerland), Carlo Erba Reagents (Milan, Italy), ICN Biomed (United Kingdom), and L’Italiana Aromi (Carate Brianza, Italy), as listed in Table S1 of the supplementary material.

Methanol ($\geq 99.9\%$ purity), dichloromethane ($\geq 99.8\%$ Suprasolv purity), and ethanol ($\geq 99.9\%$ purity) used for sample extraction were purchased from Sigma–Aldrich (St. Louis, MO, USA) and Merck KGaA (Darmstadt, Germany). Ultrapure water was produced using a Milli-Q purification system (EMD Millipore, Bedford, MA, USA).

SPE–GC–MS Sample Preparation and Analysis

Ten milliliters of each sample were diluted with distilled water to a final volume of 50 mL to reduce the alcohol content to approximately 10% v/v. Subsequently, 50 μ L of 2-octanol (500 mg/L in absolute ethanol) was added as an internal standard. The samples were extracted and analyzed by GC–MS following the procedure described by López et al. (2002). Sample solutions were passed through 60 mg/3 mL HyperSep™ Retain PEP SPE cartridges (Thermo Scientific, Waltham, MA, USA), which had been previously activated with 4 mL dichloromethane, 4 mL methanol, and 4 mL of a 10% v/v ethanol–water mixture. The cartridges were then dried under vacuum, and the volatile compounds were eluted with 5 mL of dichloromethane. The obtained extracts were dehydrated using anhydrous

sodium sulphate and stored at $-30\text{ }^{\circ}\text{C}$ until analysis. Prior to GC analysis, samples were filtered and concentrated to approximately $300\text{ }\mu\text{L}$ under a gentle stream of nitrogen.

GC–MS analysis was performed using a Thermo Finnigan Trace GC Ultra gas chromatograph (San Jose, CA, USA) coupled with a Thermo Finnigan Trace DSQ mass spectrometer. The system was equipped with a fused silica Stabilwax-DA capillary column (Restek Corporation, Bellefonte, PA, USA), 30 m in length, 0.25 mm internal diameter and $0.25\text{ }\mu\text{m}$ polyethylene glycol film thickness. The injector temperature was set to $250\text{ }^{\circ}\text{C}$, and $1.5\text{ }\mu\text{L}$ of sample was injected in splitless mode. The oven temperature program was as follows: initial temperature of $45\text{ }^{\circ}\text{C}$ held for 1.0 min ; ramped to $100\text{ }^{\circ}\text{C}$ at $3\text{ }^{\circ}\text{C}/\text{min}$ and held for 1.0 min , and then increased to $240\text{ }^{\circ}\text{C}$ at $5\text{ }^{\circ}\text{C}/\text{min}$ and held for 15 min . Helium was used as the carrier gas at a constant flow rate of $1.0\text{ mL}/\text{min}$. Analytes were detected by electron impact (EI) positive ion mass spectrometry in total ion current (TIC) mode, with an ionization energy of 70 eV . The transfer line and ion source temperatures were set to $220\text{ }^{\circ}\text{C}$ and $250\text{ }^{\circ}\text{C}$, respectively. Mass spectra were acquired over a range of m/z $33\text{--}400$ at a scan rate of 1.3 scans per second. When possible, analytes were identified at Level 1 (Sumner et al., 2007) by comparing their mass spectra and retention times with those of the reference standards. The other compounds were tentatively identified by comparing their mass spectra and linear retention indices (LRIs) with those available in the NIST 2.0 and Wiley 7 databases, as well as in the literature. Mass spectral matches were considered satisfactory when the reverse match exceeded 800 . Relative concentrations of the analytes were calculated with respect to the signal intensity of the internal standard and expressed as $\mu\text{g 2-octanol}/\text{L}$.

Chemicals and Standards for HPLC–DAD–FLD Analysis

The mobile phases consisted of 0.5% acetic acid (eluent A) and pure methanol (eluent B). HPLC-grade methanol and acetic acid used for the mobile phases were supplied by Sigma-Aldrich (St. Louis, MO, USA) and Carlo Erba Reagents (Milan, Italy), respectively.

Reference standards ($\geq 99\%$ purity) were supplied as follows: gallic acid, ellagic acid, furfural, sinapaldehyde, 5-acetoxymethylfurfural, scopoletin, and coniferaldehyde from Sigma-Aldrich (St. Louis, MO, USA); 5-hydroxymethylfurfural, 5-methylfurfural, and vanillin from Merck KGaA (Darmstadt, Germany); vanillic acid from Carl Roth GmbH (Karlsruhe, Germany); and syringic acid and syringaldehyde from Fluka Chimie AG (Buchs, Switzerland). Individual standard solutions were prepared in 50% v/v water/ethanol and subsequently combined, using the same solvent, to obtain a single reference standard solution, in accordance with the official method (Commission of the

European Communities, 2000). The final concentrations in the reference standard solution, as defined by the official method, were approximately as follows: furfural $5\text{ mg}/\text{L}$, 5-hydroxymethylfurfural $10\text{ mg}/\text{L}$, 5-methylfurfural $2\text{ mg}/\text{L}$, vanillin $5\text{ mg}/\text{L}$, syringaldehyde $10\text{ mg}/\text{L}$, coniferaldehyde $5\text{ mg}/\text{L}$, sinapaldehyde $5\text{ mg}/\text{L}$, gallic acid $10\text{ mg}/\text{L}$, ellagic acid $10\text{ mg}/\text{L}$, vanillic acid $5\text{ mg}/\text{L}$, syringic acid $5\text{ mg}/\text{L}$, and scopoletin $0.5\text{ mg}/\text{L}$. An additional compound, 5-acetoxymethylfurfural at $10\text{ mg}/\text{L}$, recently identified in wood-aged brandies (Yuan et al., 2023), was also included in the reference standard solution.

HPLC–DAD–FLD Analysis

The analysis was conducted following the EU reference method for the determination of wood compounds in spirit drinks (Commission of the European Communities, 2000). HPLC separation was performed using a HPLC system (Jasco Corporation, Tokyo, Japan) equipped with a Synergi Fusion RP-C18 column (150 mm length, 4.60 mm internal diameter, $4\text{ }\mu\text{m}$ particle size; Phenomenex, Torrance, CA, USA). Detection was carried out with an MD-910 DAD ($220\text{--}500\text{ nm}$) and an FP-2020 Plus FLD (excitation: 354 nm ; emission: 446 nm) both from Jasco Corporation (Tokyo, Japan). The FLD was used specifically for the determination of scopoletin. Gallic acid, ellagic acid, furfural, 5-hydroxymethylfurfural, 5-methylfurfural, 5-acetoxymethylfurfural, vanillic acid, and syringic acid were detected at 280 nm , whereas vanillin, syringaldehyde, coniferaldehyde and sinapaldehyde were detected at 313 nm . The autosampler was set to inject $10\text{ }\mu\text{L}$ of each sample or standard solution. The column temperature was maintained at $30\text{ }^{\circ}\text{C}$ with a flow rate of $1.0\text{ mL}/\text{min}$. The elution gradient was set as follows: 0 min , 100% eluent A; 50 min , 60% eluent A; 70 min , 0% eluent A; and 77 min , 100% eluent A. The identification of the compounds was carried out by comparing the UV–Visible spectra and retention times of the peaks in the brandy samples with those of the corresponding peaks in the individual standard solutions. According to the reference method (Commission of the European Communities, 2000), quantification was performed by comparing the area of the peaks in the single standard solution with the area of the corresponding peaks in the brandy samples. The results were expressed as mg/L .

Statistical Analysis

Two-way analysis of variance (ANOVA), with a significance level of $p = 0.05$, and principal component analysis (PCA) were performed using the XLSTAT software (version 2024.4.0.1424; Addinsoft, New York, NY, USA).

Results and Discussion

A total of 107 compounds belonging to different chemical classes were identified using SPE–GC–MS and HPLC–DAD–FLD analyses. The 94 volatile compounds identified by SPE–GC–MS are listed in Table S1 of the supplementary material, along with their LRIs and methods of identification. Notably, 30 compounds were identified at level 1 confidence (Sumner et al., 2007), whereas the remaining compounds were putatively identified on the basis of at least two independent criteria.

Influence of Wood Treatments on the Endogenous Volatile Compounds of the Distillates

In Table S2 of the supplementary material, the mean contents of varietal and fermentative volatile compounds are reported according to their chemical classes. A two-way ANOVA was performed to assess the effects of the level of toasting and the duration of seasoning on these endogenous compounds after 8 months of aging. Regardless of the treatment, alcohols were the quantitatively predominant compounds, followed by acids and ethyl esters. Among the primary aromatic compounds, 12 terpenes and 2 norisoprenoids were identified. The most abundant terpenes were α -terpineol, nerolidol, and L-linalool. The *Trebbiano Romagnolo* grape cultivar is considered a neutral variety (Vernocchi et al., 2015), typically producing wines with very low concentrations of varietal aroma compounds that are well below their olfactory threshold (Rienth et al., 2021). After eight months of aging, the concentrations of the most abundant terpenes showed no obvious changes compared to the fresh distillate (C), except for linalool. A study on the fate of selected terpenes in aged wine distillates (Thibaud et al., 2020) highlighted the distinct behaviour of these compounds, depending on the specific molecule, and showed that nerol and linalool generally decrease over time, rearranging into α -terpineol, which can subsequently lead to the formation of 1–8-cineole and α -terpinene (Pedersen et al., 2003). However, while these transformations are favoured in wines due to the acidic environment, in distillates, they may require longer times, up to years, to become clearly evident (Thibaud et al., 2020). This observation may explain why, in our brandies aged for 8 months, terpene concentrations showed no relevant changes compared to the fresh distillate, except for linalool and nerolidol (only in L36 samples). Notably, the L36 samples exhibited a significant reduction in total terpene concentration, mainly due to decreases in linalool, α -terpineol, and nerolidol. Prior studies on aged distillates (Petrozziello et al., 2022) have shown that different toasting levels can influence the wood's sorption of various organic compounds, including terpenes, particularly linalool, with terpene content decreasing under

light toasting, consistent with our findings. Although no specific studies have addressed the influence of seasoning, it can be hypothesized that this process, by contributing to modifying the wood's structure (Le Floch et al., 2015), may also affect its sorption capacity toward terpenes. The presence in the distillates of 2 norisoprenoids, β -damascenone and vitispirane, was confirmed. The former is considered a key odorant because of its very low olfactory threshold (Tsakiris et al., 2014). After eight months of aging, β -damascenone showed a slight but significant increase in all wood-treated samples compared to the fresh distillate, in agreement with previous findings (Hu et al., 2024), whereas no significant change was observed for vitispirane. Considering fermentative volatile compounds, a total of 23 ethyl esters, 19 higher alcohols, and 5 acetate esters were determined. The overall concentration of ethyl esters increased exclusively in the L36 samples, rising from approximately 14.9 mg/L in the control to 20.4 mg/L after 8 months of aging, primarily due to the accumulation of medium-chain fatty acid ethyl esters, namely ethyl hexanoate, ethyl octanoate, and ethyl decanoate. The higher concentrations of medium-chain fatty acid ethyl esters in samples aged in light-toasted wood compared to those aged in heavy-toasted wood are consistent with previous findings reported for red wines aged in oak barrels for one year (Ross-Magahy et al., 2025). In contrast, long-chain fatty acid ethyl esters, specifically ethyl linoleate and ethyl hexadecanoate, increased exclusively in samples aged in heavily toasted wood, regardless of the seasoning duration. It has been reported that the accumulation of ethyl esters in distillates derives from the esterification of their corresponding fatty acids with ethyl alcohol (Onishi et al., 1977; Rodríguez Madrera et al., 2003; Tsakiris et al., 2014). However, the concentration of long-chain fatty acids in the H24 and H36 samples does not appear to fully explain the elevated levels of the corresponding ethyl esters in the distillates, suggesting that esterification may not be the sole factor driving these results. One plausible hypothesis is that the final concentration of long-chain ethyl esters reflects a balance between esterification and sorption phenomena involving the wood matrix. The lignin and hemicellulose fractions of oak wood, in fact, have been shown to participate in the sorption of various volatile compounds, including ethyl esters, primarily through hydrophobic interactions in model wine solutions (Barrera-García et al., 2006; Ramirez-Ramirez et al., 2004). As previously hypothesized in studies on aged distillates (Petrozziello et al., 2022), higher degrees of toasting could reduce the wood's capacity to retain hydrophobic volatiles due to the increase in thermal degradation of lignin and hemicellulose. Those authors concluded that this could lead to higher concentrations of volatile compounds, including ethyl esters, in the distillates aged in heavily charred wood, as also observed in our study. With respect to esters other than acetates and ethyl esters, a significant decrease in their total content was observed during aging under heavy toasting conditions, primarily due

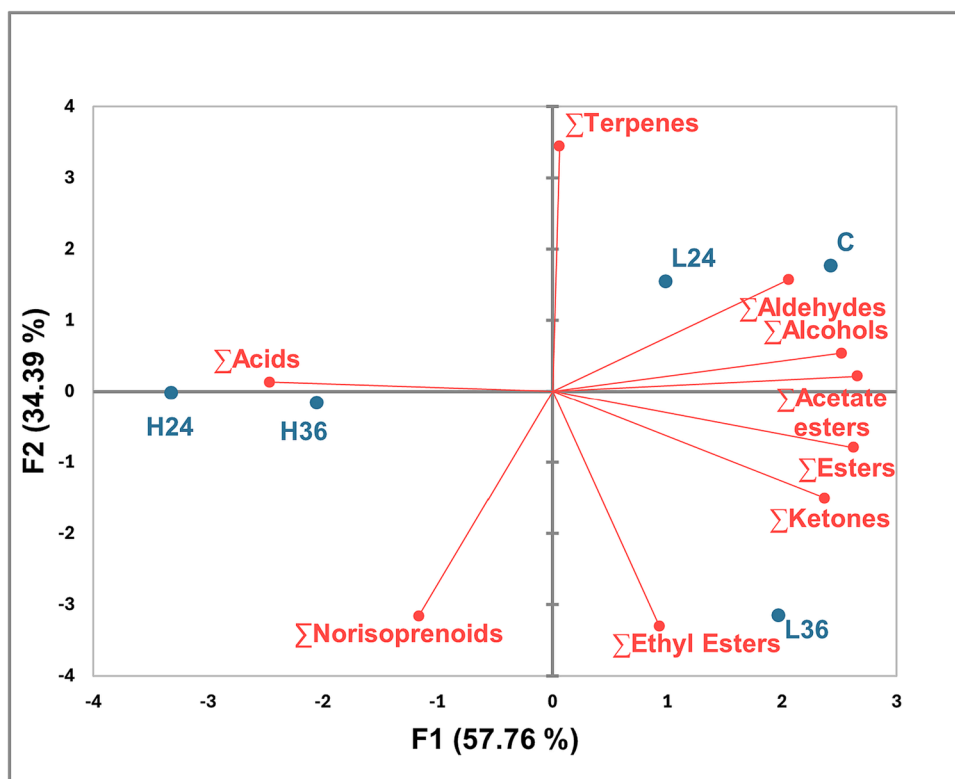
to the reduction in isoamyl lactate. The diminution of this ester, known for its fruit and nutty aromas (Guo et al., 2024), was also enhanced by longer durations of wood seasoning. Among higher alcohols, isoamyl alcohol, phenethyl alcohol, and 1-hexanol were found to be the most abundant, confirming previous findings on wine brandy (Lončarić et al., 2022). After 8 months of aging, a significant decrease in their overall content was observed in samples aged in barrels with a high level of toasting regardless of the seasoning duration. Although not specific to the matrix under study, previous research on grape marc distillates has reported a decrease in higher alcohol content after 12 months of aging in oak barrels (Mayr Marangon et al., 2021). Moreover, research investigating the effect of oak toasting on the volatile composition of red wines has demonstrated that higher toasting levels are associated with a reduction in higher alcohols during aging (Ross-Magahy et al., 2025). Aged distillates exhibited a decrease in total acetate esters regardless of the cooperage treatment, with isoamyl acetate being the main contributor. The reduction in isoamyl acetate and 2-phenylethyl acetate during aging has previously been attributed to acid-catalyzed transesterification between acetate esters and ethanol (Onishi et al., 1977). Ethyl phenylacetate was the only ester that increased, except in the L24 treatment, where no significant change was observed compared to the fresh distillate. The extent of acetate reduction varied according to both barrel toasting level and seasoning time, with greater decreases observed at higher toasting intensities and shorter seasoning periods. Similarly, previous studies (Chen et al., 2025) on high-alcohol red wines (16% vol) aged in oak barrels reported a decrease in acetate ester content with increasing toasting levels, suggesting that heat-induced wood degradation or adsorption processes in heavily toasted casks might play a key role. Since acetate esters are generally associated with fresh and fruity notes, their decline may negatively affect brandy's aromatic profile, although the overall impact on aroma intensity requires further study. Several acids were identified in the samples, with *n*-octanoic and *n*-decanoic acids being the most abundant. The acids that increased the most during aging, particularly in the H24 wood samples, were acetic, palmitic, stearic, and linoleic acids, with increases ranging from approximately 2 to ninefold compared to the unaged distillate. These increases have previously been attributed to wood release, as reported in studies on aged grape marc distillates (Mayr Marangon et al., 2021) and ethanolic wood extracts (De Rosso et al., 2009). In the case of acetic acid, its accumulation during aging has been linked to both ethanol oxidation and the hydrolysis of acetyl groups from wood hemicelluloses (Onishi et al., 1977; Reazin et al., 1976). Two ketones, 2-heptanone and 2-nonanone, were also detected in all the samples. These compounds, associated with the desirable “rancio charentais” character (Watts & Butzke, 2003), showed different evolutions depending on the wood treatments. While the concentration of 2-nonanone remained largely unaffected by

either the heat treatment or the seasoning duration, 2-heptanone levels decreased with increasing toasting intensity. A study investigating the relationship between ketone concentration and Cognac aging proposed a radical mechanism for the formation of ketones from fatty acids involving hydrogen peroxide generated by the oxidation of wood-derived phenolic compounds to quinones (Watts & Butzke, 2003). Although the proposed mechanism does not account for the observed decrease in 2-heptanone relative to the control, it supports the association between ketone presence and specific wood characteristics. Among the aldehydes, only benzaldehyde was identified. Its concentration, after aging, was not significantly different from that of the control, apart from a slight decrease in the H24 distillates, confirming previous findings on other distillates aged in oak barrels (Mayr Marangon et al., 2021). To improve the understanding of latent information and identify correlations among components and variables, a PCA was performed on the sum of the varietal and fermentative compounds. The resulting biplot is shown in Fig. 1. The first two principal components explained more than 92% of the total variance, so the model can be considered representative of the whole variability of the dataset. The first component (PC1) was strongly positively correlated (Pearson coefficient > 0.7) with acetate esters, alcohols, ketones, esters, and aldehydes, and strongly negatively correlated (Pearson coefficient < -0.9) with acids. The second component (PC2) was strongly positively correlated (Pearson coefficient > 0.9) with terpenes and negatively correlated (Pearson coefficient < -0.8) with ethyl esters and norisoprenoids. The control sample was richer in compounds strongly positively correlated with PC1, contained low levels of acids, and exhibited a medium concentration of ethyl esters. Samples H36 and H24 were the richest in acids, poorer in volatiles strongly positively correlated with PC1, and showed medium–low levels of terpenes along with a medium–high concentration of norisoprenoids. The influence of seasoning was more significant in samples aged in lightly toasted wood compared to those aged in heavily toasted wood. Sample L36 was characterized by the lowest terpene contribution, whereas sample L24 was the most similar to the unaged distillate in terms of overall composition.

Influence of Wood Treatments on the Wood-Derived Compounds of Distillates

A total of 23 wood-derived compounds were determined in the brandy samples, 13 of which were quantified using HPLC–DAD–FLD and the remaining ones by SPE–GC–MS. The results are shown in Tables 1 and 2, respectively, where the wood compounds are grouped according to their origin. A two-way ANOVA was performed on the data to study both the individual effects and the interactions of each variable and level. The quantitative data for the unaged sample,

Fig. 1 PCA biplot of endogenous volatiles (Table S2-supplementary material) in brandy samples at t_0 (C) and aged for 8 months in oak wood subjected to different treatments: L24=light toasting, 24-month seasoning; L36=light toasting, 36-month seasoning; H24=heavy toasting, 24-month seasoning; H36=heavy toasting, 36-month seasoning



$C(t_0)$, are shown as a reference but were excluded from the statistical analysis.

Notably, the unaged sample contained three furan compounds, 2-furaldehyde, 5-methyl-2-furaldehyde, and 2-furaldehyde diethyl acetal, as well as one guaiacyl derivative, 4-ethylguaiacol. The presence of furan compounds in unaged distillates is associated with the distillation process, particularly two-stage distillation, which may favor their formation (Rodríguez Madrera et al., 2003; Tsakiris et al., 2014). In contrast, 4-ethylguaiacol may be produced during the winemaking process from grape hydroxycinnamic acids via the metabolic activity of spoilage yeasts (Suárez et al., 2007). After 8 months of aging, the distillate was enriched with a variety of wood-derived compounds of different origins. The main compounds originating from lignin degradation were sinapaldehyde and coniferaldehyde, followed by syringaldehyde, vanillic acid, syringic acid, and vanillin, in accordance with the established thermal degradation pathways of lignin and the formation of their derivatives (Nonier et al., 2006; Sarni et al., 1990). With the exception of 4-ethylguaiacol, the other guaiacyl and syringyl derivatives increased significantly with the degree of toasting, in agreement with previous studies conducted on Spanish and French brandies (Artajona et al., 1990; Canas, 2017; Cantagrel et al., 1993; Puech & Moutounet, 1992). In contrast, seasoning duration did not appear to significantly affect the levels of eugenol or phenolic aldehydes. According to previous research, while toasting is generally associated with an increase in phenolic compounds due to wood degradation,

natural seasoning has been reported to have contrasting effects on the levels of eugenol, vanillin, and other phenolic aldehydes (Doussot et al., 2002; Martínez et al., 2008; Sefton et al., 1993). One possible explanation for this variability is the influence of environmental conditions during the seasoning process. For instance, the amount of vanillin present in oak-aged wines was found to depend on the drying location of the barrel staves (Spillman et al., 1997). These findings highlight the impact of climatic factors, such as temperature and humidity, on the extractive content and quality of seasoned wood. As shown in Table 2, other guaiacyl derivatives, namely, ethyl vanillate, acetovanillone, vanillyl methyl ketone, propiovanillone, and methoxyeugenol, were found at significantly higher concentrations in samples aged in long-seasoned wood, particularly when toasted at higher temperatures. To the best of our knowledge, no studies have specifically examined the effects of natural seasoning on the extraction of these compounds. However, according to Martínez et al. (2008), the progressive depletion of volatile compounds from wood during seasoning, which may result from their dissolution in water, oxidative degradation, or evaporation caused by exposure to meteoric events, may prevail over their generation from lignin breakdown. With respect to compounds resulting from the thermal degradation of cellulose and hemicellulose, furfural was found to be the most abundant, followed by 5-hydroxymethylfurfural and 5-methylfurfural. Notably, 5-acetoxymethylfurfural was also detected, but exclusively in samples aged in heavily toasted wood, with no significant differences between

Table 1 Mean content (mg/L ±SD) of wood-derived compounds determined by HPLC–DAD–FLD and analysed by two-way ANOVA at t_0 and after 8 months of aging as a function of wood toasting level (T): H=heavy, L=light, seasoning duration (S): 24 and 36 months and their interaction (TxS). Asterisks (*) indicate statistically significant effects ($p < 0.05$)

Compound							Effects		
		C (t_0) ¹	H36	H24	L36	L24	T	S	TxS
Lignin derivatives	Guaiacyl derivatives								
	Vanillin	ND ²	1.1 ± 0.1 ^a	1.4 ± 0.2 ^a	0.3 ± 0.1 ^b	0.2 ± 0.0 ^b	*		
	Vanillic acid	ND	3.0 ± 0.2 ^a	2.9 ± 0.4 ^a	2.2 ± 0.2 ^b	2.0 ± 0.2 ^b	*		
	Coniferaldehyde	ND	9.1 ± 0.2 ^a	9.7 ± 0.8 ^a	1.0 ± 0.0 ^b	0.7 ± 0.0 ^b	*		
	Sum	ND	13.1 ± 0.1 ^a	14.0 ± 0.2 ^a	3.5 ± 0.1 ^b	2.9 ± 0.0 ^b	*		
	Syringyl derivatives								
	Syringaldehyde	ND	8.3 ± 0.2 ^a	9.6 ± 0.8 ^a	0.8 ± 0.0 ^b	0.5 ± 0.0 ^b	*		
	Syringic acid	ND	2.8 ± 0.0 ^a	3.6 ± 0.8 ^a	0.3 ± 0.1 ^b	0.3 ± 0.0 ^b	*		
	Sinapaldehyde	ND	41.6 ± 2.3 ^a	45.3 ± 9.8 ^a	2.4 ± 0.1 ^b	1.6 ± 0.0 ^b	*		
	Sum	ND	52.6 ± 2.4 ^a	58.5 ± 11.4 ^a	3.6 ± 0.0 ^b	2.4 ± 0.0 ^b	*		
Hemicellulose derivatives	5-Hydroxymethylfurfural	ND	13.7 ± 0.5 ^a	15.1 ± 0.6 ^a	2.2 ± 0.0 ^b	1.5 ± 0.0 ^b	*		
	Furfural	5.4 ± 0.0	51.3 ± 1 ^a	54.5 ± 1 ^a	10.7 ± 1 ^b	10.7 ± 1 ^b	*		
	5-Methylfurfural	0.2 ± 0.0	6.9 ± 1.6 ^a	7.5 ± 2.1 ^a	0.8 ± 0.1 ^b	0.5 ± 0.1 ^b	*		
	5-Acetoxyethyl furfural	ND	0.2 ± 0.0	0.2 ± 0.0	ND	ND	*		
	Sum	5.6 ± 0.0	52.6 ± 4.1 ^a	58.5 ± 10.6 ^a	3.6 ± 0.2 ^b	2.4 ± 0.1 ^b	*		
Tannin derivatives	Gallic acid	ND	10.4 ± 0.3 ^a	8.8 ± 0.6 ^b	4.8 ± 0.0 ^c	3.4 ± 0.0 ^d	*	*	
	Ellagic acid	ND	25.4 ± 0.0 ^a	19.8 ± 0.6 ^b	10.0 ± 0.9 ^c	6.8 ± 0.6 ^d	*	*	*
	Sum	ND	35.8 ± 0.3 ^a	28.7 ± 0.0 ^b	14.8 ± 0.9 ^c	10.2 ± 0.6 ^d	*	*	*
Coumarins	Scopoletin ³	ND	88 ± 29 ^a	89 ± 11 ^a	58 ± 5 ^a	88 ± 3 ^a			

Within each row, significant differences at $p < 0.05$ are indicated with different letters. ¹Quantitative data for the unaged sample C (t_0) are shown as a reference but were not included in the statistical analysis. ²ND, not detected. ³Expressed in µg/L

Table 2 Mean content (µg/L 2-octanol ±SD) of wood-derived compounds determined by SPE–GC–MS and analysed by two-way ANOVA at t_0 and after 8 months of aging as a function of wood toasting level (T): H=heavy, L=light, seasoning duration (S): 24 and 36 months and their interaction (TxS). Asterisks (*) indicate statistically significant effects ($p < 0.05$)

Compound							Effects		
		C (t_0) ¹	H36	H24	L36	L24	T	S	TxS
Lignin derivatives	Guaiacyl derivatives								
	4-Ethylguaiacol	37 ± 1	40 ± 3 ^b	47 ± 2 ^a	42 ± 1 ^{ab}	38 ± 2 ^b	*		*
	Eugenol	ND ²	30 ± 1 ^a	29 ± 1 ^a	5 ± 0 ^b	7 ± 1 ^b	*		*
	Ethyl vanillate	ND	56 ± 4 ^b	84 ± 14 ^a	13 ± 1 ^c	12 ± 0 ^c	*	*	*
	Acetovanillone	ND	40 ± 2 ^b	65 ± 8 ^a	8 ± 0 ^c	9 ± 1 ^c	*	*	*
	Vanillyl methyl ketone	ND	79 ± 5 ^a	95 ± 12 ^a	10 ± 2 ^b	19 ± 1 ^b	*	*	
	Propiovanillone	ND	78 ± 3 ^b	102 ± 1 ^a	30 ± 4 ^d	41 ± 3 ^c	*	*	*
	Methoxyeugenol	ND	62 ± 6 ^b	94 ± 4 ^a	15 ± 1 ^c	16 ± 1 ^c	*	*	*
	Sum	ND	385 ± 12 ^b	516 ± 30 ^a	121 ± 1 ^c	143 ± 8 ^c	*	*	*
	Lactones	<i>trans</i> -β-Methyl-γ-octalactone	ND	85 ± 1 ^a	84 ± 2 ^a	77 ± 8 ^a	92 ± 17 ^a		
<i>cis</i> -β-Methyl-γ-octalactone		ND	329 ± 7 ^a	219 ± 8 ^b	327 ± 2 ^a	206 ± 7 ^b		*	
Sum		ND	415 ± 7 ^a	302 ± 8 ^b	403 ± 6 ^a	298 ± 10 ^b		*	
Hemicellulose derivatives	Furfural diethyl acetal	7 ± 1	18 ± 4 ^b	28 ± 2 ^a	11 ± 0 ^c	12 ± 0 ^c	*	*	*

Within each row, significant differences at $p < 0.05$ are indicated with different letters. ¹Quantitative data for the unaged sample C (t_0) are shown as a reference but were not included in the statistical analysis. ²ND, not detected

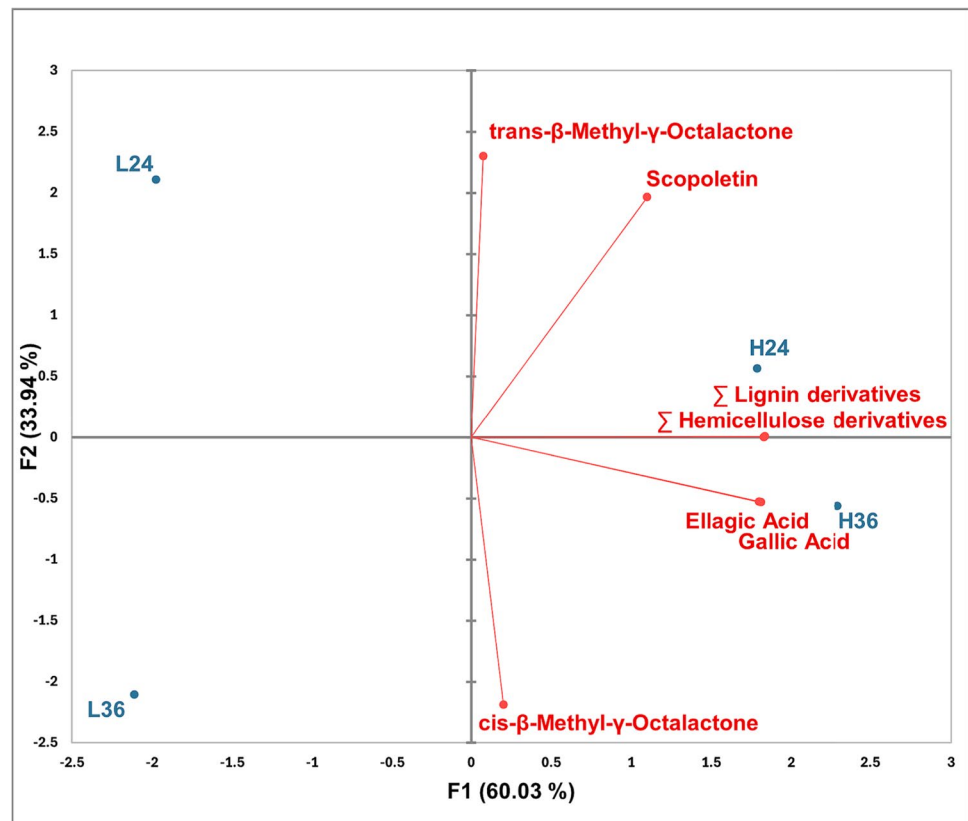
the two seasoning durations. This finding suggests its potential as a marker compound for the use of highly toasted wood in distillates subjected to short-term aging. To the best of our knowledge, only one previous study (Yuan et al., 2023) has reported the presence of this compound in brandy samples, with concentrations comparable to those observed in the present study. In general, the amounts of all the furanic compounds significantly increased with heavy toasting, confirming the results of other studies on the effects of oak wood toasting on furans, particularly furanic aldehydes (Caldeira et al., 2006; Natali et al., 2006). Regarding the influence of seasoning, no significant differences were observed among the different samples, except for diethylacetal 2-furaldehyde, which exhibited a slight increase after 24 months of seasoning. Prior studies on oak wood extracts have also indicated that seasoning exerts only a limited effect on the release of furanic aldehydes in alcoholic matrices (Fernández de Simón et al., 2010; Martínez et al., 2008). GC–MS analysis revealed the presence of both *trans*- and *cis*- β -methyl- γ -octalactones, with the *cis*-isomer occurring in higher concentrations. The resulting *cis/trans* ratios ranged from 2.22 to 4.26 and were consistent with those previously reported for wines aged in French oak barrels (Díaz-Plaza et al., 2002; Luo et al., 2023; Waterhouse & Towey, 1994). Whereas heat treatment had no effect on the content of oak lactones, wood seasoning duration significantly increased the concentration of the *cis*-isomer, leaving the *trans*-isomer unaffected. One previous study on oak wood extracts reported no effect of toasting level (Caldeira et al., 2006), in agreement with the present findings. Concerning the influence of seasoning, our results are consistent with those of Chatonnet et al. (1994), who reported a progressive increase in *cis*- β -methyl- γ -octalactone in French oak seasoned for up to 10 years. In contrast, the *trans*-isomer showed an initial increase, followed by stabilization and a marked decline starting from the third year onward. More generally, the literature presents contradictory results regarding the effects of both toasting and seasoning treatments on oak lactone content (Caldeira et al., 2006; Chatonnet et al., 1989; Fernández de Simón et al., 2010), reportedly due to the unpredictable conversion rate of the lactone precursors in wood, which would be linked to cooperative environmental conditions such as temperature, humidity, and enzymatic activity (Masson et al., 2000; Otsuka et al., 1980; Wilkinson et al., 2004). Ellagic and gallic acids were found to be the most abundant phenolic acids extracted from wood, with mean values consistent with ranges reported in the literature for aged wine spirits (Canas, 2017). The concentrations of both compounds increased significantly with the level of toasting and the duration of seasoning. With respect to the positive correlation with toasting intensity, our findings agree with those of previous studies conducted on French (Rabier & Moutounet, 1990) and Spanish (Belchior et al., 2001; Canas, 2017) brandies, whereas other research reported no influence (Cadahía et al., 2001a; Chatonnet et al., 1989;

García-Moreno et al., 2020) or even a slight decrease in medium to heavy toasted wood (Canas, 2017). It has been suggested that this variability may depend on the relevant susceptibility to oxidation of phenolics, which could undergo multiple oxidation and polymerization reactions, leading to differences in their levels across studies (Chatonnet et al., 1989; García-Moreno et al., 2020). On the other hand, the increase in ellagic and gallic acid concentrations with increasing seasoning duration could be the result of an increased chemical hydrolysis and/or enzymatic degradation of ellagitannins and gallotannins naturally present in the wood (Mosedale, 1995), as also reported by Cadahía et al. (2001b) and Doussot et al. (2002). Among the coumarins, only scopoletin was detected in this study. Its average concentration was consistent with values reported in previous studies on aged wine spirits and mature distilled alcoholic beverages (Canas, 2017; Puech & Moutounet, 1988, 1992). Neither the degree of toasting nor the duration of seasoning had a significant effect on scopoletin levels, as already reported for Italian brandy (Mattivi et al., 1989) and Portuguese chestnut woods seasoned for up to 18 months (Canas et al., 2006). To highlight the compositional differences in wood-derived compounds among the samples in relation to the different wood treatments, a PCA was performed. The results are shown in Fig. 2. The first two principal components accounted for more than 93% of the total variance, indicating that the PCA model is representative of the overall variability within the dataset. PC1 was positively correlated (Pearson coefficient ≥ 0.7) with all the variables except scopoletin and the two oak lactones. PC2 was positively correlated with scopoletin and the *trans* oak lactone, and negatively correlated with the *cis* oak lactone. Brandies aged in heavily toasted woods were positioned on the right-hand side of the biplot, reflecting their high levels of hemicellulose- and lignin-derived compounds. In contrast, samples aged in lightly toasted wood clustered on the left-hand side due to their lower concentrations of these same compounds. With respect to seasoning duration, samples aged in wood seasoned for 36 months were positioned lower on the biplot than those aged in wood seasoned for 24 months, reflecting their higher content of *cis*- β -methyl- γ -octalactone. Furthermore, seasoning had a more pronounced effect on samples aged in lightly toasted wood than on those aged in heavily toasted wood.

Conclusions

This study demonstrated that different wood treatments can significantly influence the volatile profile of *Brandy Italiano* after 8 months of aging. The most pronounced effects were observed in the levels of wood-derived compounds; nevertheless, certain endogenous volatiles were also affected. Aging in heavily toasted barrels led to a significant reduction in total alcohol content (up to 10%)

Fig. 2 PCA biplot of wood-derived compounds (Tables 1 and 2) in brandy samples at t_0 (C) and aged for 8 months in oak wood subjected to different treatments: L24 = light toasting, 24-month seasoning; L36 = light toasting, 36-month seasoning; H24 = heavy toasting, 24-month seasoning; H36 = heavy toasting, 36-month seasoning



and total ester content (up to 20%) compared to lightly toasted barrels, regardless of seasoning duration. Specific combinations of toasting intensity and seasoning duration appeared to affect the composition of endogenous volatiles in different ways. For example, light toasting combined with 3 years of natural seasoning led to the greatest increase (up to 15%) in total ethyl esters. With respect to wood-related compounds, the highest concentrations of lignin, furan, and tannin derivatives were induced by heavy toasting. In particular, the content of sinapaldehyde increased approximately 20-fold, while that of coniferaldehyde and syringaldehyde increased roughly tenfold, compared to light toasting. Additionally, 5-acetoxymethylfurfural, a compound recently identified in brandy, was determined in samples aged in heavily toasted wood, suggesting its potential use as a marker for products that have undergone this technological treatment. The duration of natural seasoning significantly influenced both gallic and ellagic acid levels, as well as the extraction of *cis*- β -methyl- γ -octalactone. For the same toasting degree, barrels seasoned for 3 years yielded distillates with concentrations of this lactone over 30% higher than those from barrels seasoned for 2 years. The results of this study provide additional insights into the chemical and aromatic evolution of *Brandy Italiano* and may offer

useful indications for brandy producers, especially those interested in enhancing flavour development over shorter ageing periods. The use of barrels and distillates supplied by a commercial distillery contributes to the practical relevance of the findings and supports their potential applicability under industrial conditions. Future research should aim to further investigate the mechanisms governing the absorption and desorption of key aromatic compounds, as influenced by distillate characteristics and cooperage treatments, as well as to explore the evolution of volatile compounds over longer ageing periods.

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Author Contributions All the authors carried out the conceptualization and methodology of the research. S.A. performed samples analysis, statistical data processing and wrote the main manuscript text. All the authors reviewed the manuscript.

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Data Availability The data supporting the findings of this study are available in the Institutional Research Repository (AMS-Acta) of the

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Declarations

Competing Interests The authors declare no competing interests.

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