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Characterization of volatile compounds and sensory properties of spine grape (*Vitis davidii* Foex) brandies aged with different toasted wood chips

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Linalool (PubChem CID: 6549)

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ABSTRACT

Aging is an important process for improving wine and brandy quality. In this study, the chemical characterization and sensory properties of spine grape brandies were compared after aging with various species of wood chips, including French oak (FO), American oak (AO), Mongolian oak (MO), Japanese blue oak (JO), chestnut, catalpa, and cherry. The results showed that high color intensity and significant concentrations of tannins and polyphenols were observed in the brandies aged with FO, AO, and chestnut chips. The volatile compounds, such as ethyl decanoate, ethyl 2-methylbutanoate, ethyl octanoate, methyl salicylate, (*Z*)-2-hexenol, and furfural, contributed to the floral, fruity, and roasted/smoky attributes of the brandies aged with FO, AO, and chestnut chips. The 1-butanol, 1-propanol, phenylethanol, phenylethyl acetate, isoamyl acetate, and linalool contributed to the fruity, honey, and floral attributes of the brandies aged with JO and cherry chips. These findings are extremely useful for the production of differentiated and high-quality spine grape brandies.

1. Introduction

Spine grape (*Vitis davidii* Foex) is a wild *Vitis* species native to East Asia with hundreds of years of cultivation history in South China (Meng et al., 2013). This grape variety is densely covered with 1–2 mm thorns on one- or two-year-old canes, and the plants have very strong stress tolerance, such as disease resistance, low light resistance, and high humidity and heat resistance (Gutiérrez-Gamboa, Liu, Sun, & Fang, 2020; Meng et al., 2013). In addition, spine grape berries are characterized by a neutral aroma, low sugar content, and high acidity and are suitable for producing wines and distilled spirit-based beverages (Kong et al., 2019; Xiang et al., 2020). Aroma is a crucial quality parameter of brandies and is derived mainly from the grape variety, fermentation process, and storage in oak barrels; thus, these technological parameters directly determine the key flavor characteristics of the resulting products (Tao, García-Martín, & Sun, 2014). Numerous methods, such as cold maceration, enzyme application, and wine aging, have been developed for aroma improvement and to obtain a desirable and pleasant wine flavor. Previous studies have reported the key odor-active volatile compounds in spine grape berries and distilled spine grape spirits (Meng et al., 2013; Xiang et al., 2020). However, research on the aroma characteristics of spine grape wine and its derivative products during the aging process has not been reported.

Aging is a crucial process for improving the quality of final products and the organoleptic characteristics of alcoholic beverages. Traditionally, aging is carried out by storing wines and brandies in oak barrels for micro-oxygen aging, during which the beverages undergo important modifications that improve their physicochemical stability and increase the complexity of their mouthfeel and flavor (Canas et al., 2016; Tao et al., 2014). Oak-responsible aromatic compounds and astringency-related phenolic compounds are gradually released and transferred into wines or spirits during the aging process and are enhanced or modified by synergistic or masking effects. In addition, certain compounds are gently oxidized and degraded by atmospheric oxygen permeation through the walls of oak barrels, resulting in delicate mouthfeel and changes in color (Cerdán & Ancín-Azpilicueta, 2006; Tao

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et al., 2014). Specifically, the brandies aged in wood chips presented the highest intensities of greenish and topaz color, toasted and coffee odors, whereas the brandies aged in wooden barrels presented the highest intensities of golden color, alcohol odor and bitter taste (Caldeira, Anjos, Portal, Belchior, & Canas, 2010). Thus, the application of wood materials in the aging of spirit-based beverages, including fruit brandies, has a great influence on their final color, taste and aroma characteristics (Coldea et al., 2020; Yan et al., 2024). However, owing to the high cost, long production cycle, and complexity of barrel stock management in traditional natural aging processes, new materials and several alternative technologies have been used to shorten the aging period of wine products and achieve a satisfying color, taste and aroma (Coelho, Teixeira, Tavares, Domingues, & Oliveira, 2021; Tao et al., 2014). Among them, wood alternatives such as sessile oak (*Q. petraea*), chestnut (*C. sativa*), mulberry (*M. alba* and *M. nigra*), cherry (*P. avium*), fir (*A. alba*), and walnut (*J. regia*) have shown potential for replacing traditional *Q. robur* and *Q. alba* (Coldea et al., 2020). Although several species of wood chips, as alternatives to oak barrels, can be considered suitable for aging, the specific effects of these woods on the chemical characteristics and sensory properties of wines and brandies are need to be further elucidated.

Several studies have reported the evolution of volatile and phenolic compounds in wines and spirits following aging with oak chips (Coelho et al., 2021; Han, Tian, Zheng, Jiang, & Bian, 2024; Schumacher, Alañón, Castro-Vázquez, Pérez-Coello, & Díaz-Maroto, 2013). In most cases, the application of wood chips or staves can accelerate the extraction of wood-derived phenolic and volatile compounds and shorten the aging period. Indeed, since wines can completely soak and penetrate wood chips, the extraction efficiency and rate of wood-related compounds, such as volatiles, tannins, and phenols, are accelerated during the aging process in the presence of wood chips (Alencar et al., 2019; Yan et al., 2024). These compounds present important correlations with several sensory attributes, such as smoke and toasted odors, astringency, and bitter taste, which are positively correlated with the overall quality of the brandies (Caldeira et al., 2010). In apple brandies, the main esters, such as ethyl acetate, isobutyl acetate, ethyl decanoate, isoamyl-2-methylbutyrate, ethyl benzoate and ethyl nonanoate were accentuated by aging with mulberry, chestnut and cherry chips (Coldea et al., 2020). Toasted French oak chips enhanced the astringency, sweetness and richness of apple brandy and reduced the acidity and bitterness (Yan et al., 2024). In terms of organoleptic properties, wood chips or staves are desirable alternatives to oak barrels for producing short-term aged wines and brandies with satisfactory sensory quality (Tao et al., 2014). However, the effects of various species of wood chips on the chemical characterization and sensory properties in the spine grape brandies are still unclear.

Spine grape spirits are produced and widely appreciated by local consumers (Xiang et al., 2020). In this study, seven different species of toasted wood chips were used for spine grape distilled spirit aging to produce spine grape brandies. The color intensities, total tannin and polyphenol contents, volatile compound contents, and sensory properties of wood-aged brandies were investigated to comprehensively assess the potential of wood chips in spine grape brandy during the aging process. The results of this study may provide potential technology for the development of differentiated and high-quality spine grape brandies by aging with wood chips.

2. Materials and methods

2.1. Chemicals

Sodium chloride (NaCl), sodium bicarbonate ($NaHCO_3$), and anhydrous sodium sulfate (Na_2SO_4) were obtained from Tianjin Chemical Works (Tianjin, China). GC-grade ethanol ($\geq$ 99.8 %), methanol ($\geq$ 99.9 %), dichloromethane ($\geq$ 99.8 %), and chemical standards for quantification and identification analyses (Supplementary Table S1) were purchased from Sigma–Aldrich (Shanghai, China). 4-Methyl-2-pentanol ($\geq$ 98 %) was employed as an internal standard.

2.2. Oak and substitute wood materials

Seven species of wood chips, including French oak (*Q. robur*, FO), American oak (*Q. alba*, AO), Mongolian oak (*Q. mongolica*, MO), Japanese blue oak (*Q. glauca*, JO), chestnut (*C. sativa*), catalpa (*C. bignonioides*), and cherry (*P. avium*), were obtained from Changyu Winery (Changyu Group Co., Ltd., Yantai, China). All wood chips were $20 \times 5 \times 5$ mm^3 and were toasted at 180 °C for 40 min in an oven before being used as toasted chips.

2.3. Fermentation, distillation and aging modalities

The fermentation and distillation of spine grape (*Vitis davidii* Foex, ‘Xiangzhenzhu’) wines were performed according to previous studies (Duan et al., 2021; Xiang et al., 2020). Briefly, 1500 kg of healthy spine grapes (reducing sugar content, 177.33 g L^{-1}; titratable acidity, 5.61 g L^{-1}) were harvested on Sep. 20, 2021, from a commercial vineyard (31°54′90″ N, 105°02′35″ E; elevation 458 m; subtropical monsoon climate) of Liangqi Winery in Xujia County, Mianyang City, Sichuan Province, China. These grapes were immediately destemmed, crushed, and cold macerated overnight. The must was obtained by squeezing and then fermented at 20–22 °C with an industrial *Saccharomyces cerevisiae* yeast strain (200 mg L^{-1}, AC, Laffort Inc., France). The spine grape wines were fermented to ‘dryness’ (reducing sugar content <4 g L^{-1}) for approximately 7 to 8 days. After alcoholic fermentation, the basic chemical parameters (pH, 3.12; total acidity, 8.35 g L^{-1}; alcohol content, 8.50 % vol) of the spine grape base wine were determined.

The distillation of base wine was carried out by double distillation with the Charente pot distillation method to obtain the distilled spirit. On the basis of alcohol content, the heads (65 % vol), brouillis (40 % vol) and tails (15 % vol) distillates were obtained from 50 L of the base wine. The heads and tails were subsequently mixed with another portion of base wine (50 L) for further distillation. A total of 20 first-stage distillations were performed. The condenser temperature was maintained at 24 °C. After the first-stage distillations, the total collected brouillis were secondly distilled to obtain the heads (85 % vol), hearts (66 % vol), seconds (15 % vol) and tails (5 % vol) distillates. The hearts and seconds were proportionally mixed until the alcohol content of the blend reached to 40 % vol.

Aging with various species of wood chips was performed according to a previous report (Coelho et al., 2021), with slight modifications. A total of seven groups were applied in this study, i.e., toasted FO, AO, MO, JO, chestnut, catalpa, or cherry chips were added to the distilled spirit at a proportion of 4 g L^{-1} for the aging process. Aging was conducted in glass containers (10*L*) at 16–18 °C, and the mixtures were agitated daily during the aging period. Each experimental group was tested in triplicate. The seven groups of spine grape brandies were aged with different species of wood chips for 6 months, and distilled spirits without aging were used as the control samples (Control). All aged spine grape brandies were filtered and bottled for further chemical and sensory analyses.

2.4. Determination of physicochemical indicators of the spine grape brandies

The titratable acidity (expressed as tartaric acid equivalent, g L^{-1}), volatile acidity (expressed as acetic acid equivalent, g L^{-1}), pH, and alcohol content (% vol) of the spine grape brandies were determined according to the OIV standards (OIV, 2017). The concentrations of total tannins and polyphenols were determined via a UV–Vis spectrophotometer (Cary 60 UV–Vis, Agilent Technologies, Santa Clara, CA, USA) according to previous methods (Alencar et al., 2019; Yan et al., 2024). The total tannin content (expressed as epicatechin equivalents, mg L^{-1})

was measured using the methyl cellulose precipitation method at 280 nm. The total polyphenol content (expressed as gallic acid equivalent, mg L^{-1}) was measured using the Folin–Ciocalteu method at 765 nm. The CIELab parameters of brandy chromaticity, such as lightness (L^*), redness–greenness (a^*), yellowness–blueness (b^*), chroma (C^*), and hue angle (h), were measured using a colorimeter (CM-5, Konica-Minolta, Tokyo, Japan), and the total color difference (ΔE) was calculated as follows: $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$.

2.5. Determination of volatile compounds

Volatile compounds of spine grape brandies were analyzed by a headspace solid–phase microextraction system combined with gas chromatography–mass spectrometry (HS–SPME–GC–MS) following previously reported methods (Li, Yang, Tian, Zou, & Li, 2020; Xiang et al., 2020). For sample analysis, 5 mL of each brandy sample was added to 1 g of NaCl and 10 μL of an internal standard solution (4-methyl-2-pentanol, 1.0083 g L^{-1}) in a 15 mL headspace bottle. The vial was immediately sealed with a lid with a polytetrafluoroethylene (PTFE)-silicon septum. The mixture was homogenized with a magnetic stirrer (1 cm) at 400 rpm, and the volatile compounds were extracted at 40 °C for 30 min. An automatic SPME fiber (Supelco, Bellefonte, PA, USA; 50/30 μm DVB/CAR/PDMS) was then immersed in the headspace, and the mixture was extracted for 30 min. The volatiles trapped in the fiber were thermally desorbed in the GC injector (250 °C) for 8 min.

GC–MS analysis was performed on an HP-INNOWAX column (60 m × 0.25 mm, 0.25 μm) using an Agilent 6890 GC coupled with a 5975C MS detector (Agilent Technologies, Wilmington, USA). The oven temperature was initially set to 40 °C for 2 min, increased to 210 °C at a speed of 3 °C min^{-1}, and increased again to 250 °C with an increase of 5 °C min^{-1}, after which the temperature was maintained at 250 °C for 5 min. Ultrapure helium (> 99.999 %) was used as a carrier gas at a constant flow rate of 1.0 mL min^{-1}. The temperatures of the injector and ionization source were maintained at 250 °C. The ionization source conditions were an electron energy of 70 eV and a mass scan ranging from m/z 30 to 350. The identities of the volatile compounds were carefully confirmed by comparing the retention times, MS fragmentation patterns of the existing standards, and mass spectra in the National Institute of Standards and Technology Mass Spectral Library. Standards of aroma compounds were dissolved in ethanol (GC-grade), and fifteen different concentrations of standards were serially diluted in alcohol solution (40 % vol; pH 3.8–4.0) to establish standard curves of volatile compounds for the HS–SPME–GC–MS analysis. The quantitative determination of the identified compounds was carried out using an internal standard method with 4-methyl-2-pentanol, and the results were calculated from the calibration curves in Supplementary Table S1. All analyses were conducted in triplicate for each sample.

The odor activity value (OAV), which measures the contribution of each volatile compound to the characteristic aroma, was calculated by dividing the relative concentration of a certain compound by its absolute odor threshold (Bowen & Reynolds, 2012).

2.6. Sensory analysis

The tasting panel consisted of sixteen panelists (aged 20 to 35 years; eight males and eight females), and they were recruited from the Professional Tasting Panel of the College of Enology, Northwest A&F University. All panelists were involved in flavor chemistry research on wines and had the winetasting experience. Before tasting, each panelist received eight consecutive sensory tasting trainings, including clarity, color, aroma, taste, style, and aftertaste, for 1 h each time to standardize the criteria among these panel members, according to our previous descriptions (Duan et al., 2021). The spine grape brandies were presented in ISO standard tasting glasses, numbered and provided to the panelists in a random order in an isolated tasting room (20–22 °C). The sensory evaluation was performed in triplicate for each brandy sample. The brandy samples were evaluated for several quality attributes, i.e., clarity, color, aroma, taste, style, and aftertaste, using an 11-point quantitative scale, where 0 indicated no perceived descriptor (absence) and values from 1 to 10 corresponded to intensities ranging from low to maximum. The specific aroma descriptors used were divided into seven classes: fusel/solvent, green/plant, fruity, sweet, roasted/smoky, sweaty/fatty, and floral.

2.7. Statistical analysis

The data are shown as the mean ± standard error ($n = 3$). Statistically significant differences in the concentrations of physicochemical indicators or volatile compounds among the spine grape brandy samples were evaluated by one-way analysis of variance (ANOVA) and Duncan's multiple range tests at $p < 0.05$ using SPSS 22.0 (IBM, New York, USA). Calibration curves of volatile compounds were obtained using standard solutions of fifteen different concentrations, and the linear correlation coefficients (R^2) and linear ranges were calculated. All descriptors of the sensory profile were mean-centered per panelist and scaled to unit variance. The clustered heatmap and principal component analysis (PCA) were performed using R 3.6.1 software.

3. Results and discussion

3.1. Acidity, tannin and polyphenol contents, and chromaticity of spine grape brandies

To determine the physicochemical changes in spine grape brandies aged with different species of wood chips (wood-aged brandies), the acidity, pH, alcohol content, tannin content, polyphenol content, and chromaticity of the resulting spine grape brandies were determined (Table 1). The brandies aged with FO and AO chips had higher values of titratable acidity (0.59 and 0.52 g L^{-1}, respectively) than that of other brandy groups. The values of volatile acidity and pH of all the brandy samples were in accordance with OIV standards. The alcohol contents were similar (~40 % vol) among all the wood-aged brandies, as they were produced from the same fermentation and distillation processes and under similar aging modality.

Compared with those in the control samples, the concentrations of tannins and polyphenols were substantially greater in the wood-aged spine grape brandies. Specifically, the brandies aged with FO, AO, and chestnut chips contained the most tannins (302.27, 311.01, and 304.40 g L^{-1}, respectively), followed by the brandies aged with cherry (117.68 g L^{-1}) and MO (35.99 g L^{-1}) chips, but the lowest tannin contents were detected in the brandies aged with catalpa (14.12 g L^{-1}) and JO (11.27 g L^{-1}) chips. With respect to the polyphenol contents, spine grape brandies aged with FO and AO chips contained the most polyphenols (325.50 and 331.45 g L^{-1}, respectively), followed by the brandies aged with chestnut (315.57 g L^{-1}), cherry (153.73 g L^{-1}), catalpa (94.18 g L^{-1}), and MO (50.43 g L^{-1}) chips, and the lowest polyphenol content was detected in the brandies aged with JO (25.39 g L^{-1}) chips. During the aging process, several wood-derived compounds are gradually extracted from the hydroalcoholic matrix, which has a positive impact on the properties and final quality of alcoholic beverages (Coldea et al., 2020; Tao et al., 2014). The chestnut as an alternative wood induced a significantly greater accumulation of phenolic compounds, showing a high potential for the aging of wine spirits, confirming previous results that higher levels of gallic acid and ellagitannins are distinctive features of chestnut wood (Canas, Caldeira, Anjos, & Belchior, 2019; de Simón et al., 2014). Previous studies have shown that increasing the concentrations of condensed tannins and polyphenols in wines can affect the perception of astringency by enhancing the sensation of dryness and grip in the mouth (Coldea et al., 2020; Ortega-Heras, Pérez-Magariño, Cano-Mozo, & González-San José, 2010; Watrelot, Kuhl, & Waterhouse, 2019), indicating that spine grape brandies aged with FO, AO or chestnut chips obtained in the present study would be driven to more

Table 1
Physicochemical parameters of spine grape brandies after aging with wood chips.

Parameters	Control	FO	AO	MO	JO	Chestnut	Catalpa	Cherry
Titratable acidity (g L^{-1})	0.41 ± 0.04b	0.59 ± 0.04a	0.52 ± 0.02a	0.33 ± 0.03b	0.34 ± 0.04b	0.37 ± 0.03b	0.42 ± 0.03b	0.40 ± 0.02b
Volatile acidity (g L^{-1})	0.30 ± 0.02ab	0.34 ± 0.03a	0.21 ± 0.05b	0.33 ± 0.01a	0.29 ± 0.03ab	0.34 ± 0.02a	0.26 ± 0.02b	0.26 ± 0.01b
pH	3.99 ± 0.01b	3.88 ± 0.05b	4.01 ± 0.08b	4.23 ± 0.05a	4.42 ± 0.12a	3.91 ± 0.10b	4.23 ± 0.03a	4.10 ± 0.08b
Alcohol content (% vol)	39.97 ± 0.12a	39.47 ± 0.37a	39.03 ± 0.21a	39.60 ± 0.24a	38.83 ± 0.12a	39.73 ± 0.19a	39.83 ± 0.09a	39.73 ± 0.25a
Tannins content (mg L^{-1})	tr	302.27 ± 15.17a	311.01 ± 17.53a	35.99 ± 4.76c	11.27 ± 5.87d	304.40 ± 8.66a	14.12 ± 1.96d	117.68 ± 8.77b
Polyphenols content (mg L^{-1})	tr	325.50 ± 4.57a	331.45 ± 9.27a	50.43 ± 3.27e	25.39 ± 1.22f	315.57 ± 3.75b	94.18 ± 2.04d	153.73 ± 10.51c
*L**	100.03 ± 0.81a	96.18 ± 0.12c	95.97 ± 0.06d	98.79 ± 0.21ab	99.36 ± 0.50a	97.18 ± 0.15b	98.73 ± 0.54ab	97.48 ± 0.20b
*a**	0.12 ± 0.01a	−1.44 ± 0.16d	−1.70 ± 0.12e	−0.49 ± 0.01c	−0.29 ± 0.03b	−1.56 ± 0.07d	−2.03 ± 0.16f	−0.23 ± 0.02b
*b**	0.05 ± 0.01 g	9.06 ± 0.06b	10.38 ± 0.28a	1.89 ± 0.02e	0.65 ± 0.01f	7.17 ± 0.39c	4.89 ± 0.53d	6.39 ± 0.05c
C*	0.13 ± 0.02 g	9.18 ± 0.09b	10.52 ± 0.28a	1.95 ± 0.02e	0.71 ± 0.01f	7.33 ± 0.39c	4.40 ± 0.05d	7.18 ± 0.56c
h	1.16 ± 0.05a	−1.41 ± 0.02d	−1.41 ± 0.01d	−1.32 ± 0.05c	−1.15 ± 0.01b	−1.36 ± 0.01c	−1.28 ± 0.02b	−1.52 ± 0.01e
ΔE	0.00	9.89 ± 0.19b	11.24 ± 0.16a	2.25 ± 0.25f	0.92 ± 0.35 g	7.80 ± 0.19c	5.36 ± 0.21e	6.83 ± 0.12d

FO, French oak; AO, American oak; MO, Mongolian oak; JO, Japanese blue oak. 'tr' means trace. Different letters in the same row means significant differences among the spine grape brandy samples according to Duncan's multiple range tests ($p < 0.05$).

intensities of mouthfeel attributes than spine grape brandies aged with other wood chips.

After aging, the brandy has a yellowish brown or amber color, reflecting its ripens aging and its desirable quality (Canas et al., 2019; Yan et al., 2024). In this study, the *L**, *a**, and h values of the color parameters were significantly greater in the brandies aged with MO, JO, catalpa, and cherry chips than in the other brandies, except for the *a** value in the catalpa aged brandies and the h value in the cherry aged brandies, whereas the *b**, C*, and ΔE values were significantly greater in the brandies aged with FO and AO chips, followed by those aged with chestnut and cherry chips. These results, which are in accordance with those of a previous study (Caldeira et al., 2010), indicate that the brandies aged in French oak, American oak, and chestnut present a more discriminant and more mature color. During the aging process, the differences in the chromatic characteristics of the wine distillate might be due to the melanoidins produced by the Maillard reactions during wood roasting being transferred to the liquor (Herzfeld et al., 2011), the condensation reactions between tannins mediated by acetaldehyde (Picariello, Gambuti, Picariello, & Moio, 2017), and the oxidative phenomena of phenols, such as flavan-3-ol monomers ((+)-catechin and (−)-epicatechin), flavan-3-ols polymers (proanthocyanidins, also known as condensed tannins) and ellagitannins, leading to the formation of new brown color pigments (Canas et al., 2019; Flamini, Panighel, & De Marchi, 2021), indicating that the intensities of color and astringency perception of brandies are markedly modified after aging with different wood chips.

3.2. Quantification of volatile compounds and OAV analysis

Identification of the volatile compounds in spine grape brandies aged with toasted wood chips was achieved by HS–SPME–GC–MS analysis (Table 2). A total of 38 main volatile compounds in the spine grape brandies were tentatively identified and were significantly affected after aging with wood chips. The most abundant volatile compound in the wood-aged brandies was isoamyl alcohol (from 2412.10 to 2601.11 mg L^{-1}), followed by isobutanol (from 1342.76 to 1547.95 mg L^{-1}), 1-propanol (from 712.17 to 866.29 mg L^{-1}), and ethyl acetate (from 612.56 to 875.07 mg L^{-1}). Similarly, the quantification results revealed that the relative contents of isoamyl alcohol, isobutanol, and ethyl acetate were the highest among the aroma compounds in alcoholic beverages, especially in fruit brandies (Coldea et al., 2020; Xiang et al., 2020; Yan et al., 2024).

Difference tests confirmed that the concentrations of volatile compounds were significantly differ among wood-aged brandies because of differences in inherent wood characteristics (Coldea et al., 2020; Picard, Nonier, Vivas, & Vivas, 2021). Compared with those in the control samples, the concentrations of isoamyl alcohol, isobutanol, ethyl lactate, ethyl hexanoate, ethyl 2-methylbutanoate, and linalool were significantly greater in all the wood-aged brandies, and there were no significant differences among these brandies; however, the concentrations of (*E*)-3-hexenol, ethyl acetate, ethyl butanoate, ethyl octanoate, α-terpineol, acetic acid, benzaldehyde, nonanal, and octanal were markedly lower in these wood-aged brandies. Specifically, the concentrations of 3-methyl-1-pentanol, 4-methyl-1-pentanol, furfural, and octanoic acid were significantly greater in the brandies aged with FO and AO, chestnut, catalpa, and cherry chips than in the other brandies, except for 4-methyl-1-pentanol in the chestnut aged brandies. The concentrations of 1-propanol and isoamyl acetate were significantly greater in the brandies aged with MO, JO, catalpa, and cherry chips than in the other brandies. In addition, the concentrations of ethyl decanoate, methyl salicylate, furfural and phenol were very high in the brandies aged with FO and AO chips. Interestingly, the concentrations of 1-butanol were the highest in the brandies aged with JO chips, followed by cherry chips; moreover, the concentrations of (*Z*)-2-hexenol and propanoic acid were the highest in the brandies aged with AO, FO, and cherry chips. A recent study shows that alcohols, including propanol and 2,3-butanediol, can further affect the complexity of the aroma and taste of mulberry brandy by oxidation reactions to produce aldehydes, such as acetaldehyde, furfural and vanillin, and acids (Han et al., 2024). During the wood aging process, the inconsistency in the increase of ester concentration may be attributed to the direct transfer of wood-derived compounds (e.g., vanillin, guaiacol, and eugenol) from different species of wood and the reactions between volatile compounds and distillates (Caldeira et al., 2010; Yan et al., 2024). However, Ramirez et al. (2001) reported that the concentrations of volatile compounds did not depend on the solubility of these compounds but rather on the acid, base and polarity properties of the solution matrix. These previous findings provide some explanations for our results concerning the differences in volatile compounds in different wood-aged spine grape brandies.

The OAVs of the volatile compounds were analyzed to determine the potential contributions of the specific volatile compounds to the aroma or flavor characteristics of the wood-aged spine grape brandies (Table 3). In the wood-aged brandy samples, ethyl octanoate had the highest OAVs (from 882.01 to 1119.79), followed by (*Z*)-2-hexenol (from 106.78 to 136.61), ethyl hexanoate (from 110.53 to 123.09), isoamyl acetate (from 43.77 to 53.97), and ethyl acetate (from 19.98 to

Table 2

Concentrations of volatile compounds in the spine grape brandies after aging with wood chips by HS-SPME-GC–MS.

Number	Volatile compounds	Concentration (μg L^{-1})							
		Control	FO	AO	MO	JO	Chestnut	Catalpa	Cherry
1	Isobutanol	904,939.04 ± 3413.13b	1,344,817.44 ± 17,541.07a	1,453,403.74 ± 48,783.42a	1,508,217.60 ± 23,539.15a	1,445,964.77 ± 132,553.47a	1,342,762.68 ± 27,080.96a	1,441,225.81 ± 12,585.36a	1,547,950.63 ± 72,009.07a
2	Isoamyl alcohol	1,508,784.31 ± 15,318.68b	2,440,105.82 ± 4752.49a	2,526,385.17 ± 108,654.62a	2,412,101.37 ± 16,219.56a	2,428,208.41 ± 58,739.17a	2,441,542.28 ± 53,199.49a	2,562,875.97 ± 3990.70a	2,601,107.98 ± 83,603.46a
3	1-Octanol	70.26 ± 3.70c	170.54 ± 2.81a	157.03 ± 2.66ab	141.67 ± 3.32b	157.98 ± 6.07ab	164.74 ± 5.12a	154.66 ± 1.49ab	156.36 ± 7.50ab
4	1-Propanol	538,114.70 ± 27,202.52d	735,606.29 ± 12,914.42c	772,368.15 ± 24,107.28b	858,221.71 ± 39,288.06a	801,185.74 ± 5185.38a	712,170.97 ± 17,739.90c	805,760.31 ± 17,994.94a	866,285.59 ± 26,717.47a
5	3-Methyl-1-pentanol	510.35 ± 24.12c	932.75 ± 27.46a	907.61 ± 34.24a	862.10 ± 5.71b	884.65 ± 18.79ab	915.72 ± 11.28a	908.31 ± 24.16a	937.68 ± 49.93a
6	4-Methyl-1-pentanol	221.92 ± 10.70c	371.38 ± 17.39a	375.95 ± 19.69a	351.39 ± 0.71b	358.70 ± 0.17b	349.81 ± 4.79b	381.29 ± 13.63a	372.71 ± 6.17a
7	Phenylethanol	tr	19,121.66 ± 347.17b	22,092.28 ± 352.72ab	22,190.69 ± 1264.54ab	26,943.31 ± 1698.19a	28,630.37 ± 5447.26a	24,564.13 ± 1350.39a	35,357.18 ± 3770.63a
8	1-Butanol	4016.04 ± 460.75d	5701.70 ± 88.43c	6925.24 ± 482.87b	6021.65 ± 75.22c	9521.58 ± 419.76a	5445.54 ± 122.49c	5825.25 ± 315.26c	9353.15 ± 190.98a
9	1-Hexanol	181.20 ± 0.84c	2111.98 ± 98.56a	2041.12 ± 156.25a	1839.81 ± 0.19b	1953.49 ± 55.74a	2069.26 ± 33.09a	2087.22 ± 1.10a	2043.43 ± 118.28a
10	(*Z*)-2-Hexenol	119,496.72 ± 13,318.96d	162,089.09 ± 6305.69b	171,724.51 ± 244.40a	150,067.92 ± 460.51c	137,929.84 ± 13,446.33c	148,344.89 ± 4190.07c	139,066.55 ± 3769.08c	134,223.71 ± 50.87c
11	(*E*)-3-Hexenol	467.80 ± 54.51a	259.35 ± 13.81 cd	366.95 ± 29.91b	205.86 ± 11.90d	198.90 ± 33.26d	353.70 ± 0.40b	227.13 ± 72.91d	229.28 ± 20.04d
12	Isoamyl acetate	11,926.03 ± 256.54a	11,177.34 ± 447.86b	11,175.21 ± 473.01b	13,221.85 ± 248.91a	12,240.36 ± 991.98a	10,722.43 ± 243.74b	12,005.82 ± 232.12a	12,631.56 ± 463.32a
13	Isobutyl acetate	tr	716.89 ± 11.94c	717.22 ± 40.88c	926.87 ± 30.52a	790.60 ± 68.58b	659.40 ± 58.85c	819.23 ± 6.52b	848.39 ± 17.60b
14	Phenylethyl acetate	177.26 ± 21.84d	213.51 ± 13.19c	273.74 ± 10.30b	319.15 ± 6.87a	324.39 ± 20.25a	295.98 ± 1.35a	303.54 ± 11.59a	315.41 ± 28.51a
15	Ethyl acetate	1,015,555.75 ± 81,572.36a	651,198.63 ± 2750.08d	677,111.24 ± 44,200.32d	875,066.60 ± 25,650.67b	723,964.28 ± 10,899.59c	612,561.73 ± 17,877.96d	764,281.85 ± 20,926.73c	803,603.20 ± 17,426.80b
16	Ethyl butanoatec	336.94 ± 15.66a	156.42 ± 2.08bc	167.52 ± 5.51b	182.04 ± 5.67b	173.41 ± 24.04b	147.26 ± 0.71c	162.79 ± 4.13b	186.52 ± 8.47b
17	Ethyl decanoate	tr	20,465.81 ± 1044.51a	17,983.95 ± 870.13b	15,568.26 ± 1901.89b	13,247.65 ± 729.99c	11,075.51 ± 80.48d	9886.06 ± 1435.60d	9261.27 ± 264.52d
18	Ethyl hexanoate	1978.66 ± 159.74b	3494.91 ± 199.07a	3651.08 ± 79.11a	3570.20 ± 94.80a	3315.79 ± 220.96a	3520.24 ± 373.51a	3692.70 ± 71.80a	3548.71 ± 91.60a
19	Ethyl nonanoate	2.32 ± 0.40c	8.44 ± 0.40a	9.59 ± 0.39a	10.01 ± 0.85a	6.97 ± 1.72b	6.53 ± 0.06b	5.81 ± 0.68b	5.56 ± 0.28b
20	Ethyl octanoate	18,041.24 ± 458.62a	13,653.84 ± 486.61c	14,445.30 ± 24.42b	11,664.74 ± 28.54d	11,652.82 ± 1147.30d	12,501.25 ± 355.18d	11,787.72 ± 356.48d	11,377.91 ± 35.61d
21	Ethyl laurate	72.24 ± 35.98d	360.68 ± 74.46b	434.98 ± 45.13b	744.27 ± 156.53a	356.35 ± 141.67b	195.25 ± 1.41c	170.22 ± 39.75c	129.63 ± 6.37c
22	Ethyl lactate	13,385.62 ± 894.62b	21,951.78 ± 379.68a	22,046.12 ± 1504.72a	23,955.44 ± 491.68a	23,445.67 ± 3202.26a	18,699.44 ± 1456.81a	21,887.75 ± 1323.13a	25,549.06 ± 3583.32a
23	Methyl salicylate	1.30 ± 0.46c	3.02 ± 0.44a	2.15 ± 0.10b	1.75 ± 0.15c	1.37 ± 0.12c	1.33 ± 0.01c	1.18 ± 0.14c	1.21 ± 0.17c
24	Methyl octanoate	6.84 ± 0.72d	11.40 ± 0.64b	12.73 ± 0.26a	11.50 ± 0.40b	9.86 ± 0.02c	11.76 ± 0.51b	11.29 ± 0.06b	10.83 ± 0.09b
25	Diethyl butanedioate	376.56 ± 14.72c	535.00 ± 16.25b	611.67 ± 10.25a	666.95 ± 30.12a	694.73 ± 76.33a	630.81 ± 44.90a	627.92 ± 44.10a	785.18 ± 168.46a
26	Ethyl 2-methylbutanoate	10.00 ± 0.76b	15.38 ± 0.08a	14.99 ± 0.19a	13.05 ± 0.01a	10.95 ± 1.85a	14.09 ± 3.86a	12.78 ± 0.29a	14.21 ± 1.15a
27	α-Terpineol	39.13 ± 6.64a	4.72 ± 0.70b	3.47 ± 0.42b	5.33 ± 0.21b	5.09 ± 0.14b	4.10 ± 0.25b	5.24 ± 0.24b	5.55 ± 1.48b
28	Linalool	4.26 ± 1.94b	10.80 ± 0.99a	10.08 ± 1.06a	11.42 ± 0.07a	12.47 ± 0.32a	9.62 ± 0.13a	11.95 ± 0.18a	12.58 ± 0.89a
29	Acetic acid	2707.37 ± 233.07a	1095.79 ± 6.09 cd	1074.68 ± 69.17 cd	908.83 ± 28.03d	970.46 ± 77.15d	1114.54 ± 78.63 cd	1441.42 ± 263.59c	2176.47 ± 131.87b
30	Decanoic acid	1020.52 ± 25.44a	1155.62 ± 316.34a	993.12 ± 109.05a	466.48 ± 22.29b	592.24 ± 46.66b	1115.51 ± 373.49a	813.53 ± 157.60a	726.02 ± 53.65a
31	Hexanoic acid	890.66 ± 28.78ab	961.21 ± 46.40a	776.36 ± 16.79b	850.77 ± 75.03b	957.61 ± 72.65a	866.32 ± 41.86b	926.00 ± 194.66a	1286.83 ± 173.35a
32	Octanoic acid	891.00 ± 79.14b	1100.74 ± 108.22a	1269.37 ± 94.59a	909.82 ± 58.62b	1320.25 ± 1.83a	1427.48 ± 288.45a	1419.64 ± 231.68a	1870.17 ± 332.62a
33	Propanoic acid	tr	22,079.23 ± 1456.67a	15,416.94 ± 110.50b	13,725.39 ± 449.87c	18,459.15 ± 1052.59b	18,416.19 ± 365.60b	16,943.49 ± 1852.34b	18,741.35 ± 1541.05b
34	Benzaldehyde	162.84 ± 34.95a	59.20 ± 0.45d	80.85 ± 1.52b	76.15 ± 1.68c	77.30 ± 2.67c	79.02 ± 0.65c	83.70 ± 0.51b	84.93 ± 1.07b
35	Furfural	1933.00 ± 159.78e	18,978.76 ± 383.13a	22,914.09 ± 375.31a	6528.30 ± 246.56d	5350.37 ± 168.45d	14,291.14 ± 192.53b	7873.77 ± 283.23c	7641.65 ± 159.11c
36	Nonanal	26.92 ± 3.27a	3.19 ± 0.11b	2.48 ± 0.31b	2.00 ± 0.27b	2.82 ± 0.14b	3.02 ± 0.25b	2.59 ± 0.29b	1.93 ± 0.25b
37	Octanal	82.96 ± 25.02a	12.81 ± 3.91b	7.70 ± 1.10b	8.16 ± 2.32b	5.27 ± 0.73c	3.55 ± 0.05c	4.71 ± 0.34c	9.26 ± 0.80b
38	Phenol	2.48 ± 1.44d	66.88 ± 11.44a	43.71 ± 4.02b	35.49 ± 0.74c	34.90 ± 0.38c	30.27 ± 0.76c	31.96 ± 0.91c	30.40 ± 2.67c

FO, French oak; AO, American oak; MO, Mongolian oak; JO, Japanese blue oak. ‘tr’ means trace. Different letters in the same row means significant differences among the spine grape brandy samples according to Duncan’s multiple range tests ($p < 0.05$).

Table 3
Odor activity values (OAVs) of volatile compounds in the spine grape brandies after aging with wood chips.

Number	Volatile compounds	Class	Aroma characteristics	Aroma series	Threshold (μg L^{-1})	OAVs							
						Control	FO	AO	MO	JO	Chestnut	Catalpa	Cherry
1	Isobutanol	HA	fusel, solvent	1	28300^{5}	31.98	47.52	51.36	53.29	51.09	47.45	50.93	54.70
2	Isoamyl alcohol	HA	fusel, solvent, pungent	1	179000^{5}	8.43	13.63	14.11	13.48	13.57	13.64	14.32	14.53
3	1-Octanol	HA	alcoholic, fruity	1, 3	900^{3}	0.08	0.19	0.17	0.16	0.18	0.18	0.17	0.17
4	1-Propanol	HA	fusel, solvent	1	54000^{5}	9.97	13.62	14.30	15.89	14.84	13.19	14.92	16.04
5	3-Methyl-1-pentanol	HA	alcoholic, harsh	1, 2	500^{5}	1.02	1.87	1.82	1.72	1.77	1.83	1.82	1.88
6	4-Methyl-1-pentanol	HA	alcoholic, almond, roasted	1, 2, 5	1000^{3}	0.22	0.37	0.38	0.35	0.36	0.35	0.38	0.37
7	Phenylethanol	HA	floral	7	2600^{5}	–	7.38	8.51	8.56	10.34	11.04	9.42	13.62
8	1-Butanol	C6	alcoholic, solvent	1	2730^{2}	1.47	2.09	2.54	2.21	3.49	1.99	2.13	3.43
9	1-Hexanol	C6	green, grass	2	8000^{5}	0.02	0.26	0.26	0.23	0.24	0.26	0.26	0.26
10	(*Z*)-2-Hexenol	C6	grass, green	2	1257^{5}	95.07	128.95	136.61	119.84	109.73	118.02	110.63	106.78
11	(*E*)-3-Hexenol	C6	grass, green	2	1000^{5}	0.47	0.26	0.37	0.21	0.20	0.35	0.23	0.23
12	Isoamyl acetate	AE	fruity, sweet, honey	3, 4	245^{5}	38.68	45.62	45.61	53.97	49.96	43.77	49.00	51.56
13	Isobutyl acetate	AE	fruity, pear, banana	3	1600^{3}	–	0.45	0.45	0.58	0.49	0.41	0.51	0.53
14	Phenylethyl acetate	AE	rose, honey	3, 4, 7	250^{3}	0.71	0.85	1.09	1.28	1.30	1.18	1.21	1.26
15	Ethyl acetate	EE	pineapple, fruity, sweet	1, 3	32600^{2}	31.15	19.98	20.77	26.84	22.21	18.79	23.44	24.65
16	Ethyl butanoate	EE	fruity, sweet	3	9.51^{5}	35.43	16.45	18.62	19.14	18.23	15.48	17.12	19.61
17	Ethyl decanoate	EE	floral	7	1120^{2}	–	18.27	16.06	13.90	11.83	9.89	8.83	8.27
18	Ethyl hexanoate	EE	fruity	3	30^{5}	65.96	116.50	121.70	120.01	110.53	117.34	123.09	118.29
19	Ethyl nonanoate	EE	fruity	3	3150^{2}	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
20	Ethyl octanoate	EE	fruity	3	12.90^{2}	1398.55	1058.44	1119.79	904.24	903.32	969.09	913.78	882.01
21	Ethyl laurate	OE	fruity	3	–	–	–	–	–	–	–	–	–
22	Ethyl lactate	OE	fruity	3	128000^{2}	0.10	0.17	0.17	0.19	0.18	0.15	0.17	0.20
23	Methyl salicylate	OE	mint	–	–	–	–	–	–	–	–	–	–
24	Methyl octanoate	OE	waxy, fruity	3	100^{4}	0.07	0.11	0.13	0.12	0.10	0.12	0.11	0.11
25	Diethyl butanedioate	OE	fruity	3	6000^{4}	0.06	0.09	0.10	0.11	0.12	0.11	0.10	0.13
26	Ethyl 2-methylbutanoate	OE	fruity	3	57.50^{2}	0.17	0.27	0.26	0.23	0.19	0.25	0.22	0.25
27	α-Terpineol	Ter	floral, lilac	7	250^{3}	0.16	0.02	0.01	0.02	0.02	0.02	0.02	0.02
28	Linalool	Ter	rose, floral	7	23^{5}	0.14	0.36	0.34	0.38	0.42	0.32	0.41	0.43
29	Acetic acid	FA	vinegar	6	75521^{5}	0.04	0.01	0.01	0.01	0.01	0.01	0.02	0.03
30	Decanoic acid	FA	fatty, sweaty	6	2800^{5}	0.36	0.41	0.35	0.17	0.21	0.40	0.29	0.26
31	Hexanoic acid	FA	sweaty, pungent	6	2520^{2}	0.35	0.38	0.31	0.34	0.38	0.34	0.37	0.51
32	Octanoic acid	FA	fatty, sweaty	6	$2700^{3,5}$	0.33	0.41	0.47	0.34	0.49	0.53	0.53	0.69
33	Propanoic acid	FA	fatty, sweaty	6	8100^{5}	–	2.73	1.90	1.69	2.28	2.27	2.09	2.31
34	Benzaldehyde	Car	sharp, cherry	7	4200^{5}	0.04	0.01	0.02	0.02	0.02	0.02	0.02	0.02
35	Furfural	Car	roasted, smoky, sweet, almond	4, 5	5800^{2}	0.30	2.27	3.95	1.38	0.93	2.43	1.37	1.30
36	Nonanal	Car	green, floral, fruity	2, 3, 7	15^{3}	1.79	0.21	0.17	0.13	0.19	0.20	0.17	0.13
37	Octanal	Car	fatty	6	39.60^{1}	2.09	0.32	0.19	0.21	0.13	0.09	0.12	0.23
38	Phenol	VP	phenol, medicinal		18900^{2}	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01

The categories for volatile compounds: HA, higher alcohols; C6, C6 alcohols; AE, acetate esters; EE, ethyl esters; OE, other esters; Ben, benzenes; Ter, terpenes; FA, fatty acids; Car, carbonyl compounds; VP, volatile phenols. The classification standard of aroma series referenced according to Xiang et al., 2020. 1, fusel/solvent; 2, green/plant; 3, fruity; 4, sweet; 5, roasted/smoky; 6, sweaty/fatty; 7, floral. The references for odor thresholds: 1, Fan et al. (2015); 2, Gao et al. (2014); 3, Kong et al. (2019); 4, Peng, Wen, Tao, and Lan (2013); 5, Xiang et al. (2020). OAVs, Odor activity values. FO, French oak; AO, American oak; MO, Mongolian oak; JO, Japanese blue oak.

26.84). However, the OAVs of ethyl octanoate, ethyl butanoate, ethyl acetate, nonanal, and octanal were greater in the control samples than in the wood-aged brandies. These results were consistent with previous observations that ethyl butanoate (OAV: 1894) had the highest OAV in the head of distilled spirits from spine grape wines, followed by ethyl hexanoate (OAV: 1086), ethyl octanoate (OAV: 425), and isoamyl acetate (OAV: 106) (Xiang et al., 2020), although some of these compounds were present at relatively low concentrations in spine grape brandies after wood aging. Several different groups of volatile compounds, such as higher alcohols, esters, terpenes, fatty acids, carbonyl compounds, and volatile phenols, have significant effects on the final aroma characteristics of the resulting brandy products (Tao et al., 2014; Xiang et al., 2020).

Alcohols are produced primarily through the process of ethanolic fermentation and provide alcohol products with alcoholic odors and some grass/green attributes. C6 compounds (especially 2-hexenal) are characteristic flavor components of various spine grape (*V. davidii* Foex) clones (Meng et al., 2013). In this study, the OAVs of higher alcohols and C6 alcohols, including (*Z*)-2-hexenol (from 106.78 to 136.61), isobutanol (from 47.45 to 54.70), isoamyl alcohol (from 13.48 to 14.53), 1-propanol (from 13.19 to 16.04), phenylethanol (from 7.38 to 13.62), 1-butanol (from 1.99 to 3.49), and 3-methyl-1-pentanol (from 1.72 to 1.88), markedly increased in brandies aged with wood chips. These alcohols have also been identified as key odor-active compounds in many wines, such as brandy (Coldea et al., 2020), baijiu (Fan, Fan, & Xu, 2015; Gao, Fan, & Xu, 2014), and spine grape spirits (Xiang et al., 2020). Compared to those of the control samples, the OAVs of isoamyl alcohol and 3-methyl-1-pentanol were greater in the brandies aged with FO and AO, chestnut, catalpa, and cherry chips, whereas the OAVs of isobutanol and 1-propanol were greater in the brandies aged with MO and cherry chips. Phenylethanol and 1-butanol were relatively high OAVs in the brandies aged with cherry and JO chips. (*Z*)-2-Hexenol had the highest OAVs in the brandies aged with AO chips, followed by those aged with FO chips. These results indicated that the concentration and proportion of odor-active compounds in alcohol products are affected not only by fermentation and distillation technologies but also by the type of oak wood used in the aging process. Previous studies have shown that higher alcohols, such as 1-propanol, 1-butanol, and 2-methyl-1-propanol, positively influence the aroma complexity of wines or distillates in a specific proportion, whereas at relatively high concentrations, these compounds are characterized by penetrating odors that mask the aromatic finesse (Tsakiris, Kallithraka, & Kourkoutas, 2014; Xiang et al., 2020).

Ester compounds are also particularly important since they mainly provide fruity and floral attributes. The OAVs of most acetate esters, ethyl esters, and other esters, such as ethyl hexanoate (from 110.53 to 123.09), isoamyl acetate (from 43.77 to 53.97), ethyl decanoate (from 8.27 to 18.27), and phenylethyl acetate (from 0.85 to 1.30), markedly increased after the brandies were aged with wood chips. The OAVs of isoamyl acetate, phenylethyl acetate, and ethyl acetate were greater in the brandies aged with MO, cherry, catalpa, and JO chips than in those aged with other chips. However, ethyl decanoate, ethyl octanoate, and ethyl 2-methylbutanoate were more abundant in the brandies aged with FO and AO chips. Moreover, the OAVs of ethyl hexanoate were the highest in the brandies aged with catalpa chips, followed by those aged with AO, MO, and cherry chips. The esters contributed to a pleasant fruity and floral aroma at a specific quantity in brandies aged with French and American oak chips (Bozalongo, Carrillo, Torroba, & Tena, 2007; Yan et al., 2024). In apple brandies, ethyl acetate significantly decreased in cherry and mulberry aged brandies, but a 2.3 % increase was observed in sessile aged brandies (Coldea et al., 2020), indicating that the type of wood also has a decisive effect on ester compounds.

In addition, fatty acids, such as octanoic acid, propanoic acid, and decanoic acid, provide fatty and sweaty attributes (Tsakiris et al., 2014; Xiang et al., 2020). In this study, the OAVs of propanoic acid were the highest in spine grape brandies aged with FO chips, followed by those aged with JO, chestnut, and cherry chips. Although fatty acids are often thought to confer an undesirable aroma, these compounds produce these aroma attributes only at concentrations above 20 mg L^{-1} (Zhao et al., 2012). In the spine grape brandies, the OAVs of linalool were greater in the brandies aged with JO, chestnut, and cherry chips than in the other brandy groups, and the OAVs of furfural were greater in the brandies aged with AO, FO, and chestnut chips. Previous studies have shown that the extraction of furfural from wood is greater than the conversion of furfural to form the corresponding alcohols during short aging periods; thus, furfural tends to accumulate in wine and confers a pleasant wood, almond, caramel and vanilla-like aroma (Bautista-Ortín et al., 2008; Flamini et al., 2021). In addition, the volatile profiles of apple brandies aged with different wood chips revealed that cherry wood caused an ~15 % decrease in furfural content, but sessile oak caused an ~5 % increase, suggesting that the effects of different woods on the concentration of furfural differ depending mainly on the wood species (Coldea et al., 2020).

Collectively, these findings establish the aroma potential of oaks or substitute woods in spine grape brandy during the aging process.

3.3. Effect of wood chip addition on volatile compounds in spine grape brandies

Clustered heatmap and principal component analysis (PCA) were constructed and independently performed to obtain an overall view of the effects of various wood chips on volatile compounds in spine grape brandies (Fig. 1 and Fig. 2). Hierarchical clustering analysis classified the five sets of samples into two major groups (Fig. 1). The first group included ethyl esters (ethyl acetate, ethyl butanoate, and ethyl octanoate), carbonyl compounds (nonanal, octanal, and benzaldehyde), terpenes (α-terpineol), fatty acids (acetic acid), and alcohols ((*E*)-3-hexenol), which corresponded to high levels in the control samples and low levels in the wood-aged brandy samples. The second group was subdivided into three subgroups due to the relatively high and different contents of most volatile compounds in the wood-aged brandy samples as a result of aging with different species of wood chips. Specifically, high levels of ethyl esters (ethyl laurate, ethyl decanoate, and ethyl nonanoate), other esters (ethyl 2-methylbutanoate and methyl salicylate), carbonyl compounds (furfural), C6 alcohols ((*Z*)-2-hexenol), and phenol were observed in the brandies aged with FO and AO chips, followed by those aged with MO chips. High levels of higher alcohols (1-octanol, 3-methyl-1-pentanol, isoamyl alcohol, 4-methyl-1-pentanol, isobutanol, 1-propanol, and phenylethanol), fatty acids (propanoic acid), C6 alcohols (1-hexanol and 1-butanol), ethyl esters (ethyl hexanoate, isobutyl acetate, and phenylethyl acetate), other esters (methyl octanoate, ethyl lactate, and diethyl butanedioate), and terpenes (linalool) were detected in the brandies aged with FO, AO, MO, JO, chestnut, catalpa, and cherry chips, although the concentrations of these compounds varied in these brandy samples. Additionally, high levels of acetate esters (isoamyl acetate) and fatty acids (hexanoic acid and octanoic acid) were observed in the brandies aged with cherry chips; and their levels were relatively low after aging with FO, AO, and chestnut chips, indicating that the volatile composition of different species of wood chips differentiated the chemical characteristics of spine grape brandies, in agreement with previous studies (Coldea et al., 2020; Tao et al., 2014; Yan et al., 2024).

Unsupervised PCA was also performed on those volatile compounds that presented significant differences in wood-aged brandy samples to determine which compounds produced the greatest variability among the brandies aged with various species of wood chips (Fig. 2). The first

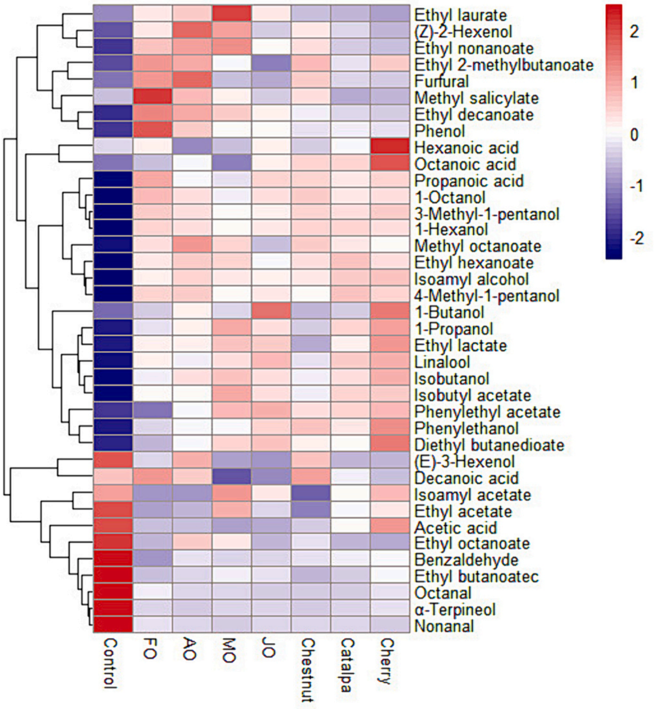

Fig. 1. Hierarchical clustering analysis of the volatile compounds obtained from spine grape brandies after aging with different wood chips.

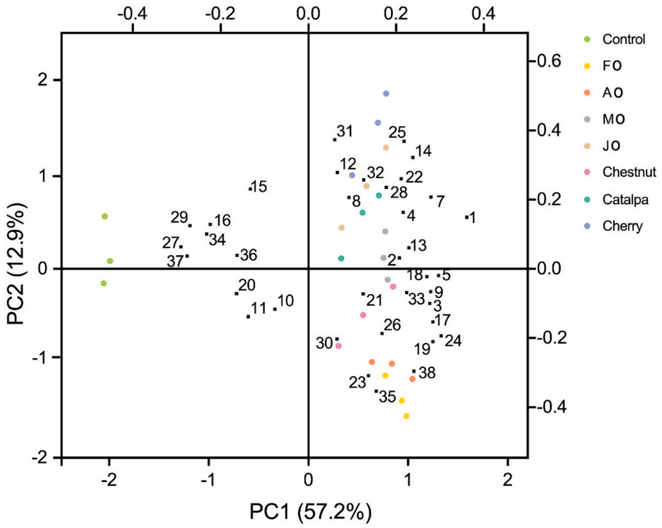

Fig. 2. Principal component analysis of the volatile compounds (represented by the numbers presented in Table 2) from the spine grape brandies aged with different wood chips.

two components (PC1 and PC2) explained for 70.1 % of the data variance (PC1: 57.2 % and PC2: 12.9 %), representing the largest fraction of variability. The wood-aged brandy samples were distributed in the positive direction of PC1 despite these brandies being aged with different types of wood, while the control samples were distributed in the negative direction of PC1. The high loading values obtained for phenylethyl acetate, isoamyl acetate, octanoic acid, 1-butanol, ethyl lactate, isobutanol, phenylethanol, and 1-propanol in the positive directions of both PC1 and PC2 were the main contributors to the brandies aged with cherry, JO, and chestnut samples, with more pronounced fruity, honey, and floral attributes. In contrast, furfural, methyl salicylate, phenol, ethyl decanoate, ethyl 2-methylbutanoate, ethyl decanoate, 3-methyl-1-pentanol, and decanoic acid in the positive direction of PC1 and the negative direction of PC2 corresponded to the brandies aged with FO, AO, and chestnut chips with more pronounced roasted/smoky, floral, and fruity attributes. In addition, isobutanol, 1-propanol, isobutyl acetate, and isoamyl alcohol in the positive direction of PC1 and the origin of PC2 corresponded to the brandies aged with MO chips, resulting in a greater fusel, solvent, and fruity aroma.

As mentioned above, previous studies have shown that due to differences in the volatile components of different wood species, the concentrations and compositions of aromatic compounds extracted from wood during the aging process differ among brandies (Caldeira et al., 2010; Coldea et al., 2020; Tao et al., 2014). Compared to the brandies aged with Portuguese chestnut wood (*C. sativa*), the wines-brandies aged with French oak (*Q. robur*) chips or staves extracted significant quantities of ethyl 2-methylpropanoate, ethyl butyrate and ethyl octanoate and lower levels of butanoic acid, syringol and *cis*-β-methyl-γ-octalactone during the aging period (Caldeira et al., 2010). In apple brandies, some researchers found that the main ester contributors, such as ethyl acetate, isobutyl acetate, and isoamyl-2-methylbutyrate, significantly increased in content with age in the presence of mulberry (*M. alba*), chestnut (*C. sativa*), and cherry (*P. avium*) chips; hexanal, ethyl-4-decenoate, and homovanilic acid were solubilized only in fir (*A. alba*) wood-aged brandy (Coldea et al., 2020).

3.4. Sensory properties of spine grape brandies

The transfer of compounds from wood chips to beverages through wood utilization modifies not only the chemical composition but also the sensory properties of alcoholic beverages (Tao et al., 2014). To identify the discrimination power of the sensory attributes of spine grape brandies aged with wood chips, a descriptive sensory analysis was additionally applied for the characterization test. Spine grape brandies aged with different species of wood chips presented distinct sensory attributes compared to those of the control samples (Fig. 3). In this study, the color of all wood-aged brandies was observed noticeably yellow or amber color (Fig. 3A). The wood-aged brandies were perceived to have significantly greater color intensity, aroma quality (purity, intensity, and persistence), and taste quality (purity, intensity, and persistence) than the control samples (Fig. 3B). Among these wood-aged brandy samples, the brandies aged with MO (8.18), JO (8.00), and catalpa (8.54) chips presented lower color intensities and differed from the brandies aged with FO (9.64), AO (9.46), chestnut (9.62), and cherry (9.10) chips, which presented higher color intensities, indicating that the wood chip additives could enhance the color of the spine grape brandies during the aging period. In addition, the aroma and taste intensity of the brandies aged with AO (9.10 and 8.30) and FO (9.15 and 8.80) chips were the perceived high average scores, followed by those of the samples aged with chestnut (9.05 and 8.10), catalpa (8.95 and 7.90), MO (8.80 and 8.05), and cherry (8.70 and 7.75) chips, whereas the aroma and taste intensity of the brandies aged with JO chips were the lowest, with values of 7.80 and 7.00, respectively. These results are consistent with those of Coldea et al. (2020), who reported that volatile and phenolic compounds extracted from different types of wood could enhance the aroma and taste characteristics of wood-aged apple brandies during a short aging period. The toasted chip-treated wine produced more taste sensation and had more grassy/vegetal and roasted/smoky odors in the wines because of the extraction of wood-derived volatile and phenolic compounds during the exchange of oak/substitute wood and wine or its liquor (Coldea et al., 2020; García-Carpintero, Gallego, Sánchez-Palomo, & Viñas, 2012; Ortega-Heras et al., 2010). Moreover, the evolution of the redox potential, which reflects the oxidation–reduction reaction in red wines during the aging process, has also been shown to be different in different aging systems, such as oak chips, oak staves, and barrels (del Alamo, Nevares, & Cárcel, 2006), resulting in notable differences in wine style and characteristics. Previous studies have shown that wood botanical species are more discriminant than aging systems (Caldeira et al., 2010; Canas et al., 2019). Among the wood species, the brandies aged with chestnut wood presented higher intensities of topaz color and lower intensities of yellow-straw color than did the brandies aged with Limousin oak (Caldeira et al., 2010). Generally, the sensory attributes of aged wines and brandies, such as color, aroma, and taste, depend upon a combination of several factors, such as the wood species, type of oak toasting, and wine–wood contact time (Cadahía, De Simón, & Jalocha, 2003; Cerdán & Ancín-Azpilicueta, 2006; Tao et al., 2014).

Compared with the absence of wine chips, the addition of oak chips improved aroma complexity and modified sensory descriptors (Alencar et al., 2019; Coldea et al., 2020). According to aroma descriptors of the main volatile compounds, including fusel/solvent, green/plant, fruity, sweet, roasted/smoky, sweaty/fatty, and floral attributes, the total OAVs logarithmic values of the different aromatic series were calculated separately, standardized, and showed significant differences between the brandies aged with different species of wood chips (Fig. 3C). Compared with the control samples, the brandies aged with AO, FO, and chestnut chips, as expected, presented the three highest intensities of fruity, floral, and roasted/smoky characters, which might be ascribed to the high levels of (*Z*)-2-hexenol, ethyl decanoate, ethyl 2-methylbutanoate, ethyl hexanoate, and ethyl octanoate, and furfural, according to the OAV results (Table 3); the brandies aged with JO and cherry chips with more fruity, honey, and floral attributes might be ascribed to the high levels of 1-butanol, 1-propanol, phenylethanol, phenylethyl acetate, isoamyl acetate, and linalool. However, the control samples showed the lowest intensity of roasted/smoky, sweet, and floral aromas but were still dominated by fruity aroma characters, which might be attributed to the high levels of ethyl esters (ethyl acetate, ethyl butanoate, ethyl octanoate) and carbonyl compounds (nonana and octanal).

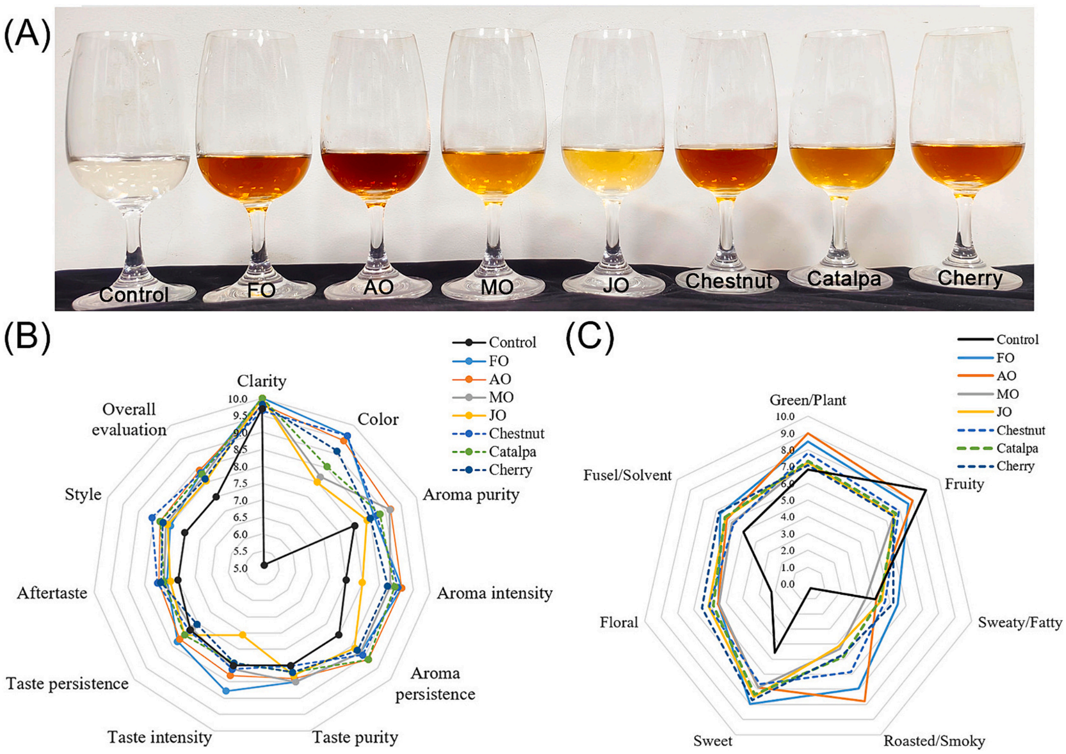

Fig. 3. Sensory properties of spine grape brandies after aging with different wood chips. (A) Samples of spine grape brandies. FO, French oak; AO, American oak; MO, Mongolian oak; JO, Japanese blue oak. (B) Sensory evaluation. (C) Aroma attributes. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

These findings are in agreement with those of Alencar et al. (2019), who reported that the addition of oak chips to wine also imparts the sensations of a vegetal and spicy aroma, sweetness/caramelization, taste persistence, and an alcoholic flavor. Syrah wines aged with medium-toasted American oak chips seem to have relatively exotic aromas of coffee and woody attributes, whereas French oak chips impart more of a perception of sweetness and provide a more elegant and balanced aroma with vanilla, nut, and spice notes (Alencar et al., 2019). In addition, previous studies have shown that chestnut chips significantly increase the vanillin content in wood-aged apple brandy and showed excellent qualities for this type of oenological product (Canas et al., 2019; Coldea et al., 2020).

4. Conclusions

Aging with seven species of wood chips, including FO, AO, MO, JO, chestnut, catalpa, and cherry, markedly increased the levels of tannins, polyphenols and most volatile compounds in spine grape brandies. The chemical and sensory profiles of brandies aged with chestnut chips were more similar to those of brandies aged with FO and AO chips than to those of brandies aged with other wood species. The brandies aged with FO, AO and chestnut chips presented high color intensities (low L* values and high b*, C* and ΔE values), tannin and polyphenol contents, and aroma and taste qualities. Concerning the volatile composition of the spine grape brandies, the volatile compounds represented by ethyl decanoate, ethyl 2-methylbutanoate, ethyl octanoate, methyl salicylate, (*Z*)-2-hexenol, and furfural contributed to the brandies aged with FO, AO, and chestnut chips with more floral, fruity, and roasted/smoky attributes, while 1-butanol, 1-propanol, phenylethanol, phenylethyl acetate, isoamyl acetate, and linalool contributed to the brandies aged with JO and cherry chips with more fruity, honey, and floral attributes. Therefore, these findings could provide potential technology for choosing a range of wood chips to produce differentiated and high-quality spine grape brandies.

CRediT authorship contribution statement

Bingbing Duan: Writing – original draft, Visualization, Investigation, Data curation. **Wei Chang:** Methodology, Investigation, Data curation. **Leqi Zhang:** Investigation, Data curation. **Mingyuan Zheng:** Visualization, Formal analysis. **Chenxing Su-Zhou:** Software. **Hasmik Merkeryan:** Writing – review & editing. **Meilong Xu:** Validation, Supervision. **Xu Liu:** Writing – review & editing, Resources, Project administration, Funding acquisition, Conceptualization.

Declaration of competing interest

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

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fochx.2024.101777.

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