Impact of Different Oak Chips’ Aging on the Volatile Compounds and Sensory Characteristics of Vitis amurensis Wines

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Abstract: In this work, different oak chips were used to age Vitis amurensis wine, and the effects on sensory properties were observed. Twenty-one different oak chips were added to a one-year-old wine made by a traditional technique. The wine was aged for 6 months before analysis by CIELab for color parameters, GC–MS for volatile compounds, and electronic tongue and a tasting panel for sensory properties. The results showed that the addition of any tested oak chip could significantly strengthen the wine’s red color. Among 61 volatile compounds, alcohols presented the highest concentrations (873 to 1401 mg/L), followed by esters (568 to 1039 mg/L) and organic acids (157 to 435 mg/L), while aldehydes and volatile phenols occurred at low concentrations. Different oak species with different toasting levels could affect, to varying degrees, the concentrations of esters, alcohols, and volatile phenols, but to a lesser extent those of aldehydes. Sensory analysis by a tasting panel indicated that non- and moderately roasted oak chips gave the wines higher scores than those with heavy toasting levels. The major mouthfeel descriptors determined by electronic tongue were in good agreement with those from the tasting panel.

Keywords: Vitis amurensis wine; oak chips; aging; sensory characteristics

1. Introduction

Vitis amurensis Rupr. is an East Asian member of the Vitaceae family. It originates from China and is distributed mainly in China, Russia, and Korea [1]. Because it is one of the most cold-tolerant grape varieties, it has been studied extensively [2–4]. Berries of V. amurensis have been used in the wine industry in Northeastern China for more than 70 years. Studies have found that the active constituents, i.e., the polyphenols, and the antioxidant properties of V. amurensis wine are 2 to 16 times and 5 to 15 times higher, respectively, than those of V. vinifera wine [5,6]. In addition, V. amurensis grape berries contain a wide range of nutrients, suggesting that this species could provide excellent raw materials for wine-making [3]. However, berry skins from V. amurensis grapevines have a high tannin content, resulting in wines with a strongly astringent mouthfeel [7]. It is, therefore, important to establish a method for improving the quality of V. amurensis wine. The wine quality is predominantly affected by its sensory properties (color, aroma, and taste). In order to make quality dry red wine, producers use various methods, for
example, delaying the picking time, girdling at different periods, root restriction, malolactic fermentation, low-temperature treatment, and aging processes [8–10].

Aging in oak barrels is a traditional winemaking practice, providing the wine with volatile oak aroma compounds and oak polyphenols, thus improving its quality [11–13]. After barrel aging, the wine usually shows fewer vegetal notes and higher complexity with a new aroma profile [14,15]. At the same time, wood pores can gently oxidize some compounds, resulting in a reduction in astringency and changes in color [14]. Since wine aging in barrels is slow and expensive, the use of oak chips has been proposed as a valid alternative for accelerating and reducing the cost of producing wood-flavored wine. Wine aging in the presence of oak chips has exhibited a higher production of aroma compounds and hydrolyzed tannins, increasing the quality of the wine [14,16]. Puech et al. found that oak contains 40~45% cellulose, 20~25% hemicellulose, 25~30% lignin, and 8~15% tannin [17–19].

Oak chips of different origins with different toasting levels have different effects on the sensory characteristics of the wine. If the features of the wine do not integrate well with the oak elements, the wine will lose its specific characteristics. There is no clear stipulation on what kind of oak treatment is suitable for a particular type of wine, so the oak treatment must be carefully selected.

The objective of this work was to improve the quality of *V. amurensis* wine by aging it with oak chips. For this purpose, different kinds of oak chips, namely, non-toasted French oak (NFr), moderately roasted French oak (MFr), heavily roasted French oak (HFr), moderately roasted Chinese oak (MCh), heavily roasted Chinese oak (HCh), moderately roasted American oak (MAm), as well as the combination of any two of these, were tested. The CIELab method was used for the analysis of the color parameters, GC–MS analysis for quantification of the volatile compounds, and electronic tongue and a tasting panel analysis for the evaluation of the sensory properties of the tested wines.

### 2. Materials and Methods

#### 2.1. Materials

Chips of heavily toasted French oak, moderately toasted French oak, non-toasted French oak, and moderately toasted American oak were purchased from Enartis (Beijing, China). The chips of moderately toasted Chinese oak (*Quercus mongolica*) and heavily toasted Chinese oak (*Quercus mongolica*) were provided by Fisch. ex Ledeb (Jilin, China).

Ethyl octanoate, 1-pentanol, propane-1, 1,3-triethoxy, 3-ethoxypropanol, 1-octene-3-ol, phenylethyl alcohol, pentadecanoic acid, 3-methyl butyl ester, and n-decanoic acid were obtained from Chengdu Chroma-Biotechnology Co., Ltd. (Chengdu, China). The pure hydrocarbon mixture (C10-C23) standard was obtained from Chengdu Chroma-Biotechnology Co., Ltd. All chemicals and reagents were obtained from Tianjin Chemical Company, Ltd. (Tianjin, China).

#### 2.2. Preparation of *Vitis amurensis* Wine

*Vitis amurensis* grapes (Shuang Hong variety) cultivated on the Zijinggege estate (Jian, Jilin, China) were harvested in the technological ripeness stage during the vintage period (September–October) of 2016. The *Vitis amurensis* wine was made by the winery of the same estate on an industrial scale using traditional vinification technology. The harvested grape clusters were crushed and destemmed using a destemmer-crusher. The must was collected in stainless steel tanks and treated with sulfur dioxide (50 mg/L) before undergoing alcoholic fermentation at 25 °C. The cap was punched down twice a day until it remained submerged. After six days of maceration, when alcoholic fermentation was finished, the wine was pressed. Free-run and press wines were combined and stored in a stainless steel tank at 25 °C. The racking treatments were performed at the end of three, six, and twelve months of wine storage. After each racking, sulfur dioxide (30 mg/L) was added. The wine stored for one year was then divided into various 2 L micro-stainless-steel tanks for further aging with oak chips. The *Vitis amurensis* wine before the oak-chip aging
experiments presented the following physico-chemical characteristics: alcohol content 10.68 (% vol), total sugar 3.63 g/L, dry extract 31.60 g/L, total acidity 16.17 g/L (expressed as tartaric acid), volatile acidity 0.50 g/L (expressed as acetic acid), free SO$_2$ 30 mg/L, and total SO$_2$ 130 mg/L.

2.3. Oak-Chip Aging

The experimental oak-chip aging conditions are reported in Table 1. The tested chips include those of heavily, moderately, and non-toasted French oak, moderately toasted American oak, heavily toasted Chinese oak, moderately toasted Chinese oak, as well as the combination of any two of these. Prior to further analysis, a total of 21 different oak chips were added individually in different micro-stainless steel tanks and aged at 15 °C for six months.

Table 1. Addition of the 21 oak chips to wines of *Vitis amurensis*, including different single oak chips and combined oak chips, dosage (4 g/L).

| No. | Samples | Sample Abbreviation | Total Additive Amounts (g/L) | Sample Proportion |
|-----|---------|---------------------|------------------------------|------------------|
| 1   | Control | Control             | 4                            | 4:1              |
| 2   | Non-toasted French oak | NFr                 | 4                            | 1:1              |
| 3   | Moderately toasted French oak | MFr             | 4                            | 1:1              |
| 4   | Heavily toasted French oak | HFr               | 4                            | 1:1              |
| 5   | Moderately toasted Chinese oak | MCh            | 4                            | 1:1              |
| 6   | Heavily toasted Chinese oak | HCh               | 4                            | 1:1              |
| 7   | Moderately toasted American oak | MAm           | 4                            | 1:1              |
| 8   | Non-toasted French oak:Moderately toasted French oak | NFr:MFr         | 4                            | 1:1              |
| 9   | Non-toasted French oak:Heavily toasted French oak | NFr:HFr         | 4                            | 1:1              |
| 10  | Non-toasted French oak:Moderately toasted Chinese oak | NFr:MCh         | 4                            | 1:1              |
| 11  | Non-toasted French oak:Heavily toasted Chinese oak | NFr:HCh         | 4                            | 1:1              |
| 12  | Non-toasted French oak:Moderately toasted American oak | NFr:MAm         | 4                            | 1:1              |
| 13  | Moderately toasted French oak:Heavily toasted French oak | MFr:HFr         | 4                            | 1:1              |
| 14  | Moderately toasted French oak:Heavily toasted Chinese oak | MFr:MCh         | 4                            | 1:1              |
| 15  | Moderately toasted French oak:Heavily toasted American oak | MFr:MAm         | 4                            | 1:1              |
| 16  | Heavily toasted French oak:Moderately toasted Chinese oak | HFr:MCh         | 4                            | 1:1              |
| 17  | Heavily toasted French oak:Heavily toasted American oak | HFr:MAm         | 4                            | 1:1              |
| 18  | Heavily toasted French oak:Heavily toasted American oak | HFr:MAm         | 4                            | 1:1              |
| 19  | Heavily toasted French oak:Heavily toasted American oak | HFr:MAm         | 4                            | 1:1              |
| 20  | Heavily toasted Chinese oak:Heavily toasted Chinese oak | MCh:HCh         | 4                            | 1:1              |
| 21  | Heavily toasted Chinese oak:Heavily toasted American oak | MCh:MAm         | 4                            | 1:1              |
| 22  | Heavily toasted Chinese oak:Heavily toasted American oak | HCh:MAm         | 4                            | 1:1              |
2.4. Determination of Polyphenols

2.4.1. Determination of Total Phenolic Compounds

The total phenolic content (TP) was measured using the modified Folin–Ciocalteau method [20,21]; 0.2 mL of samples were diluted 5 times and mixed with 8 mL of 7.5% sodium carbonate. After 5 min, 0.5 mL of 2 N Folin–Ciocalteau reagent was added, and the volume was adjusted to 10 mL using water. Next, the color (absorbance) generated after about 120 min at 25 °C was measured at 760 nm. Gallic acid was used to construct a calibration and expressed as gallic acid equivalent (GAE).

2.4.2. Determination of Total Tannins

In this study, the total tannin content (TTA) of the *V. amurensis* wines was examined following the previously reported phenanthroline spectrophotometry method with appropriate modifications [22]. The TTA was measured spectrophotometrically using tannic acid as reference. Then, the standard solution with different concentration gradients was diluted 5 times with 10% ethanol. Ammonium ferric sulfate was added to the standard solution and allowed to react at 80 °C for 25 min. Then, buffer solution, 1,10-phenanthroline monohydrate, and EDTA were added one after the other. Lastly, the absorbance was measured at 442 nm.

2.5. Color Evaluation

The WSC-3B CIELab (Shanghai Inesa Optical Instrument Co., LTD., Shanghai, China) tristimulus colorimeter was used to record the wine color values, such as L* (lightness), a* (red/green values), b* (yellow/blue values), c* (chroma), and h* (hue angle). ∆E* (color difference) was used for a comprehensive measurement of color. The L* axis represented the wine lightness scale, which ranged from 0 to 100; L* = 0 means black, while L* = 100 means white. The a* value represents the degree of red and green, and the higher the value of a*, the more the color tends toward red. Similarly, the higher the value of b*, the more it tends toward yellow. The c* value represents the color saturation. The larger the value of c*, the higher the color saturation. The value of the hue angle (h*) ranged from 0° to 360°, with red wine generally being between 0° and 90°. Lower values of h* lead to purple or ruby red, while higher values lead to brick red or reddish-brown. ∆E* represents the difference in the comprehensive color of the sample.

2.6. Extraction and GC–MS Analysis of Aroma Components

The aroma components of each wine sample were extracted by liquid–liquid extraction in accordance with Yin et al. [23]. Briefly, 5 mL of wine samples were extracted three times with dichloromethane at a ratio of 1:1. The extracts were combined and concentrated to 5 mL, then filtered and analyzed by GC–MS (Thermo Trace 1300-ISQ; Thermo Technology Co., Ltd., Maltham, MA, USA). The oven temperature was programmed at an initial temperature of 40 °C for 10 min, increased at a rate of 3 °C/min to 160 °C, further increased up to 240 °C at a rate of 6 °C/min, and maintained at this temperature for 25 min. The carrier gas was helium (99.996%) at a flow rate of 1 mL/min followed by a 1:75 split ratio. The temperature of the injection port was 260 °C. Mass spectrometry detection was performed by electronic impact ionization (70 eV). The temperatures used were 260 °C for the trap and 255 °C for the transfer line, and the scan range was from 50 to 650 amu.

The internal standard was prepared by dissolving the accurate transfer reference standard of 2-octanol in dichloromethane to yield concentrations of 8.3 mg/mL. Standard solutions were prepared by dissolving the accurate transfer reference standard of 1-pentanol, propane-1,1,3-triethoxy, 3-ethoxypropanol, ethyloctanoate, 1-octene-3-ol, phenylethyl alcohol, pentadecanoic acid,3-methyl butyl ester, and n-decanoic acid in dichloromethane to yield concentrations of 816, 900, 904, 878, 837, 1020, 865, and 886 µg/mL of the stock solution. An appropriate amount of stock solution was taken at the concentrations of 244.8, 135.0, 135.6, 52.7, 251.1, 306.0, 259.5, and 265.7 µg/mL and diluted step by step to concentrations of 7.650, 4.219, 4.238, 1.646, 7.847, 9.562, 8.109, and 8.304 µg/mL to obtain the mixed
standard solution. Quantitative standards and calibration curves for the quantification of volatile compounds are presented in Supplementary Tables S1 and S2.

The identification of the volatile compounds was confirmed by comparing their mass spectra (HP MSD chemical workstation and NIST08 spectrum library) and their retention times with those of the pure compounds. The compounds of existing standards were quantified by the internal standard method, and the compounds without standards were quantified by reference materials with similar chemical structures and functional groups.

2.7. Sensory Analysis by Electronic Tongue

Electronic tongue (e-tongue) (SA402B multi-channel bionic lipid membrane electronic tongue, Intelligent Sensor Technology, Inc., Kanagawa, Japan) was used for taste measurement, according to previous reports [24]. The detection system consists of six electrochemical sensors (AAE, CTO, Cao, AE1, COO, and GL1) and a reference electrode (Ag/AgCl). The main taste attributes of each sensor are: AAE sensor (umami), CTO sensor (saltiness), Cao sensor (sourness), AE1 sensor (astringency), COO sensor (bitterness), and GL1 sensor (sweetness). In addition to the above five taste senses, the electronic tongue system can also detect the aftertaste of bitterness and astringency through the potential difference. The electrodes were connected to a multi-frequency and large-amplitude pulse scanner controlled by a computer. The e-tongue analysis was conducted immediately after opening the wine bottle, and 15 mL of each sample was poured into the measuring cup for testing. The working electrode was cleaned between each measurement to prevent any cumulative effects. The results were subjected to principal component analysis and radar graph analysis.

2.7.1. Principal Component Analysis (PCA)

Principal component analysis (PCA), as a commonly used method of data dimensionality reduction, can transform multiple indexes representing multiple characteristics of samples into 2–3 comprehensive indexes. There is no relationship between these comprehensive indicators, but it can reflect the information of the original multiple indicators.

These indicators are then transformed into a new coordinate system, and the PCA diagram is obtained. The smaller the distance between the samples on the PCA diagram, the closer the sample; the larger the distance on the PCA diagram, the greater the characteristic difference. The distance can characterize the difference between the samples.

2.7.2. Radar Graph Analysis

_Vitis amurensis_ wines with different oak chips have different tastes. The radar graph can clearly reflect the taste values of all kinds of _V. amurensis_ wines, which is convenient for comparison and analysis. In this study, the effect of oak-chip aging on the richness, astringent aftertaste (After-A), bitter aftertaste (After-B), sourness, sweetness, bitterness, astringency, umami, and saltiness of the wines were analyzed.

2.8. Sensory Evaluation by Tasting Panel

Sensory evaluation of the 6-month-aged wines with oak chips was performed by a tasting panel composed of 12 trained judges who had Wine & Spirit Education Trust (WSET) Level 3 Award in Wines qualifications and participated regularly in wine-tasting sessions. Standard glasses of wine for tasting (NFV09-110) were used. Other tasting conditions were as follows: room temperature, 20 °C; wine temperature, 16 °C–18 °C; amount of wine, a quarter to a third of the volume of the glass. The process of the sensory evaluation included observing the appearance under suitable light. To judge the aroma, the taster sniffs the wine at rest for 5–8 s, then shakes the glass to smell the aroma for 5–10 s, with an interval of 1–2 min between the two sniffs. The taster then sips 6–10 mL of wine. The amount should be the same each time so that the wine covers the tongue. While inhaling a small mouthful of air, the taster closes the lips, stirs the tongue, feels for 12–15 s, spits out the wine sample, feels the wine taste for 5–8 s, and the sample tasting is over. The taster then gurgles with
distilled water and continues to the next wine after the feeling disappears completely. The wine is scored using the Wine Tasting Table (AWS) of the Wine Institute of America as the evaluation index, and several specific descriptors for the aromatic profile of wine are referred to on the Wine Aroma Wheel (U.C. Davis Aroma Wheel). It is scored from five aspects: appearance, aroma, taste and structure, aftertaste, and overall impression, while the total score is calculated after averaging each evaluation index. The 20-point method was used in Table 2 [25].

Table 2. Evaluation of sensory qualities.

| Grades       | Appearance, 3 Max | Aroma, 6 Max | Taste and Texture, 6 Max | Aftetaste, 3 Max | Overall Impression, 2 Max | Total Scores |
|--------------|-------------------|--------------|--------------------------|-----------------|--------------------------|--------------|
| 3—Excellent  | 6—Extraordinary   | 6—Extraordinary | 3—Excellent | 3—Excellent | 18–20 Extraordinary      |
| 2—Good       | -Unmistakable,    | -Unmistakable, | -Lingering,       | -Good          | 15–17 Excellent          |
| -Clear with  | characteristic    | characteristic | outstanding         | -Pleasant      | 12–14 Good               |
| characteristic| color.            | aroma of grape | aftertaste.        | aftertaste.    | 9–11 Commercially        |
| color.       | 5—Excellent       | variety or wine| 3—Excellent | 1-Poor         | Acceptable               |
| 4—Good       | -Excellent        | type.        | -Good            | 0—Poor         | 6–8 Deficient            |
| -Characteristic| aroma.            | balance.     | -Excellent        | 0—Poor         | 0–5 Poor and             |
| 3—Acceptable | -Complex bouquet. | Well           | -Extraordinary   | objectionable  | objectionable            |
| 2—Deficient  | -Distinguishable  | balanced.    | -Unmistakable     | -Unmistakable  | -Objectionable           |
| -No perceptible| wine but pleasant. | -unsatisfactory| flavors.         | -Unmistakable  | -Disagreeable            |
| -Slight aroma and| May have minor| -Faults than above.| -Disagreeable flavors, poorly balanced, and/or unpleasant. | -Disagreeable flavors, poorly balanced, and/or unpleasant. | -Disagreeable flavors, poorly balanced, and/or unpleasant. |
| 1—Poor       | 1—Poor            | 2—Deficient  | 1—Poor           | 0—Objectionable| -Disagreeable            |
| -Off odors.  | -Off              | -Deficient   | -Disagreeable    | -Objectionable | -Objectionable           |
| 0—Objectionable | -Deficient      | -Deficient   | -Objectionable    | -Disagreeable | -Objectionable           |
| -off-color.  | -Deficient        | -Deficient   | -Objectionable    | -Disagreeable | -Objectionable           |

2.9. Statistical Analysis

Vinification and oak-chip aging experiments were performed in replicate and sample analysis in triplicate. The average values and standard deviations were calculated using Excel 2010 software. The SPSS 17.0 software was used for statistical analysis, and analysis of variance was used to assess significance. The heat map was made using the R studio 3.6.3 software. The PCA plot was made using the matlab 7.0 software.
3. Results

3.1. Polyphenol Content of V. amurensis Wines

The total polyphenol and tannin contents in V. amurensis wines before and after aging are shown in Table 3. Based on the analysis of the content of polyphenol compounds in the wine samples, the tannin contents of the wine increased significantly after oak aging. The total polyphenol content of the wine ranged from 7.89 to 9.43 g/L, and the total tannin content to be tested was between 4.57 g/L and 6.18 g/L. It can be seen from Table 3 that the total polyphenol and tannin contents in the wine increased after aging, which may be due to increased hydrolyzed tannins [26,27]. There was no significant difference in the total polyphenol and tannin contents of samples treated with French oak with different roasting levels, and the same was true for Chinese oak. The MCh:HCh sample had the highest polyphenol content. In addition, the total polyphenol content in wines aged with Chinese oaks was higher than that of wines treated with American and French oaks.

Table 3. Total polyphenols and total tannins of wines of Vitis amurensis.

| No. | Sample Abbreviation | TP (g/L) | TTA(g/L) |
|-----|---------------------|----------|----------|
| 1   | Control             | 7.91 ± 0.03d | 4.57 ± 0.12c |
| 2   | NFr                 | 8.26 ± 0.04cd | 4.92 ± 0.06bc |
| 3   | MFr                 | 8.44 ± 0.08cd | 5.09 ± 0.10bc |
| 4   | HFr                 | 8.17 ± 0.09cd | 4.82 ± 0.42bc |
| 5   | MCh                 | 8.80 ± 0.04bc | 5.45 ± 0.06b  |
| 6   | HCh                 | 8.95 ± 0.03b  | 5.61 ± 0.05ab |
| 7   | Mam                 | 8.54 ± 0.09c  | 5.20 ± 0.14bc |
| 8   | NFr:MFr             | 7.89 ± 0.07d | 4.67 ± 0.11c  |
| 9   | NFr:HFr             | 8.46 ± 0.18c  | 5.11 ± 0.37bc |
| 10  | NFr:MCh             | 8.66 ± 0.11bc | 5.32 ± 0.17bc |
| 11  | NFr:HCh             | 8.91 ± 0.09bc | 5.56 ± 0.14ab |
| 12  | NFr:Mam             | 8.36 ± 0.16cd | 5.01 ± 0.23bc |
| 13  | MFr:HFr             | 8.43 ± 0.13cd | 5.09 ± 0.19bc |
| 14  | MFr:MCh             | 8.35 ± 0.11cd | 5.00 ± 0.17bc |
| 15  | MFr:HCh             | 9.36 ± 0.19a  | 6.02 ± 0.26ab |
| 16  | MFr:Mam             | 8.49 ± 0.13c  | 5.15 ± 0.18bc |
| 17  | HFr:MCh             | 7.92 ± 0.09d | 4.63 ± 0.31c  |
| 18  | HFr:HCh             | 8.05 ± 0.04d | 4.70 ± 0.04c  |
| 19  | HFr:Mam             | 8.07 ± 0.03d | 4.72 ± 0.05c  |
| 20  | MCh:HCh             | 9.43 ± 0.13a  | 6.18 ± 0.17a  |
| 21  | MCh:Mam             | 8.51 ± 0.05cd | 5.17 ± 0.06bc |
| 22  | HCh:Mam             | 8.89 ± 0.09bc | 5.54 ± 0.11bc |

Different letters in a column indicate significant differences at p < 0.05; statistically, a, b, c, and d following the values indicate significant differences among these values. Total polyphenols are expressed as TP. Total tannins are expressed as TTA.

3.2. Color Evaluation

Table 4 shows that there were significant differences in the color parameters among the V. amurensis wines before and after oak-chip aging. It was observed that the wines darkened (lower L*) after aging, which would be due to their higher phenolic content. The a* value and h* value had significant differences, while the b* and ∆E* value had no significant differences before and after aging. The addition of oak chips increased the red hue of the wine. The more colorful the red wine, the better its appearance. Except for the V. amurensis wine with oak chips MFr:MCh, which changed to a yellow hue, the b* of the other aged wines did not change significantly. The results show that the color saturation of oak-chip-aged wines was improved. In addition, the h* value of the red wine was between 0° and 90°, and the color changed to ruby red. There were significant differences in the color intensity between the aged V. amurensis wine and the control group.
Table 4. Color parameters of wines of *Vitis amurensis* (n = 3).

| Sample   | L*     | a*     | b*     | c*     | h*     | ΔE*   |
|----------|--------|--------|--------|--------|--------|-------|
| Control  | 25.63 ± 0.07h | 6.11 ± 0.35a | 2.27 ± 0.03b | 2.31 ± 0.04h | 45.81 ± 0.23a | 31.91 ± 0.36ab |
| NFr     | 28.15 ± 0.02a | 4.64 ± 0.01b | 1.83 ± 0.01b | 4.98 ± 0.01cd | 41.84 ± 0.01b | 30.33 ± 0.02ab |
| MFr     | 26.32 ± 0.11ef | 1.60 ± 0.21ef | 2.24 ± 0.00b | 2.76 ± 0.12gh | 40.86 ± 0.42d | 32.97 ± 0.24ab |
| HFr     | 26.5 ± 0.03ef | 3.20 ± 0.03cd | 2.18 ± 0.03b | 3.89 ± 0.00ef | 39.66 ± 0.07ef | 32.18 ± 0.05ab |
| MCh     | 26.41 ± 0.08f | 2.06 ± 0.14ef | 2.20 ± 0.02b | 2.81 ± 0.20lg | 39.91 ± 0.28e | 32.71 ± 0.16ab |
| HCh     | 24.74 ± 0.02l | 3.80 ± 0.06bc | 2.54 ± 0.02ab | 2.57 ± 0.04gh | 45.98 ± 0.18a | 33.38 ± 0.07ab |
| MAm     | 26.84 ± 0.02de | 4.63 ± 0.02b | 2.47 ± 0.01ab | 5.24 ± 0.01c | 40.75 ± 0.01d | 31.26 ± 0.05ab |
| NFr:MFr | 25.43 ± 0.17h | 3.25 ± 0.56ced | 2.63 ± 0.01ab | 4.19 ± 0.45ef | 39.36 ± 0.06ef | 32.95 ± 0.59ab |
| NFr:HFr | 26.6 ± 0.01ef | 2.64 ± 0.06de | 2.09 ± 0.06b | 3.37 ± 0.07f | 39.06 ± 0.07f | 32.34 ± 0.09ab |
| NFr:MCh | 27.45 ± 0.04c | 4.25 ± 0.15bc | 2.10 ± 0.01b | 4.74 ± 0.13d | 40.96 ± 0.15cd | 30.99 ± 0.16ab |
| NFr:HCh | 26.76 ± 0.02e | 5.42 ± 0.03a | 2.34 ± 0.01b | 5.90 ± 0.03b | 41.76 ± 0.01c | 31.1 ± 0.04ab |
| NFr:MAm | 25.57 ± 0.09h | 1.38 ± 0.08f | 2.27 ± 0.08b | 2.66 ± 0.03gh | 41.37 ± 0.32cd | 33.69 ± 0.14a |
| MFr:HFr | 25.90 ± 0.18gh | 3.52 ± 0.04cd | 2.39 ± 0.05ab | 4.26 ± 0.06e | 39.59 ± 0.05ef | 32.51 ± 0.19ab |
| MFr:MCh | 28.01 ± 0.12ab | 5.42 ± 0.27a | 6.97 ± 6.54a | 5.90 ± 0.23b | 41.80 ± 0.32bc | 28.74 ± 6.55b |
| MFr:HCh | 27.86 ± 0.02b | 6.00 ± 0.04a | 2.12 ± 0.33b | 6.37 ± 0.07a | 42.63 ± 0.55b | 30.02 ± 0.33ab |
| MFr:MAm | 26.83 ± 0.06de | 3.00 ± 0.16d | 2.13 ± 0.08b | 3.69 ± 0.08f | 39.37 ± 0.33ef | 31.99 ± 0.19ab |
| HFr:MCh | 26.54 ± 0.11ef | 4.11 ± 0.13bc | 2.32 ± 0.00b | 4.72 ± 0.11de | 40.41 ± 0.14de | 31.76 ± 0.17ab |
| HFr:HCh | 26.11 ± 0.13g | 2.88 ± 0.47de | 2.22 ± 0.28b | 3.65 ± 0.21f | 39.68 ± 0.23ef | 32.63 ± 0.56ab |
| HFr:MAm | 27.06 ± 0.03d | 2.87 ± 0.22de | 1.91 ± 0.14b | 3.45 ± 0.11f | 39.57 ± 0.59ef | 31.92 ± 0.26ab |
| MCh:HCh | 25.68 ± 0.08h | 2.42 ± 0.04de | 2.39 ± 0.03ab | 3.41 ± 0.01f | 39.70 ± 0.07ef | 33.13 ± 0.09ab |
| MCh:MAm | 28.00 ± 0.11ab | 2.23 ± 0.21e | 1.83 ± 0.16b | 2.89 ± 0.06g | 39.31 ± 0.10ef | 31.42 ± 0.29ab |
| HCh:MAm | 26.64 ± 0.04ef | 2.95 ± 0.11d | 2.36 ± 0.02b | 3.78 ± 0.09f | 38.94 ± 0.01f | 32.10 ± 0.12ab |

Different letters in a column indicate significant differences at *p* < 0.05; statistically, a, b, c, and d following the values indicate significant differences among these values.

3.3. Aroma Components

The compounds for which the standards were available were quantified by the internal standard method, and the compounds without standards were quantified using compounds with similar chemical structures and functional groups as references. The contents of the quantified aromatic compounds are presented in Table 5. According to Table 5, a total of 24 esters, 21 alcohols, 6 acids, 2 aldehydes, and 8 volatile phenols were detected in nearly all tested wines aged with different oak chips. However, the quantified aroma-component contents were varied among the different oak-chip-aged wines. Figure 1 presents a heat map representing the aroma composition data of different oak chips and combinations. Through the heat map, the content of the aroma components can be expressed by color, and the change in contents can be clearly seen. We can observe that after aging, the main components of aroma components, i.e., esters and alcohols, have increased. It seems that the effect of single aging was not as good significant as that of mixed aging, and the increasing quality trend of NFr:HFr, NFr:MCh, NFr:MFr, MFr:MAm, HFr:MCh is more obvious.
| Compounds            | CON     | NFr      | MFr      | HFr      | MCh     | MAm     | NFr     | MFr     | HFr      | MCh     | MAm     | HFr      | MCh     | MCh      | HCh     |
|----------------------|---------|----------|----------|----------|---------|---------|---------|---------|----------|---------|---------|----------|---------|----------|---------|
| Ethyl acetate        | 1.638   | 3.798    | 1.723    | 1.828    | 1.427   | 1.968   | 1.173   | 1.545   | 2.738    | 2.927   | 1.980   | 1.878    | 1.968   | 1.928    | 1.928   |
| Isopropyl acetate    | 0.15c   | 0.54ab   | 0.92c    | 1.427g   | 1.828g  | 1.427f  | 1.828f  | 1.173f  | 1.545f   | 2.738f  | 2.927f  | 1.980f   | 1.878f  | 1.968f   | 1.928f  |
| 3-Methylbutyrate     | 5.635   | 3.534    | 3.05f    | 0.84f    | 1.427f  | 1.828f  | 1.173f  | 1.545f   | 2.738f  | 2.927f  | 1.980f   | 1.878f   | 1.968f   | 1.928f  |
| Butanoic acid, ethyl ester | 0.2271 | 0.2691   | 0.2648   | 0.2691   | 0.2648  | 0.2648  | 0.2648  | 0.2648  | 0.2648   | 0.2648  | 0.2648  | 0.2648   | 0.2648  | 0.2648   | 0.2648  |
| Acetic acid, ethyl ester | 0.83b   | 0.61b    | 0.47b    | 0.47b    | 0.47b   | 0.47b   | 0.47b   | 0.47b   | 0.47b    | 0.47b   | 0.47b   | 0.47b    | 0.47b   | 0.47b    | 0.47b   |
| Propanoic acid, 2-hydroxy-ethyl ester | 2.264   | 3.134    | 0.33bc   | 0.33bc   | 0.33bc  | 0.33bc  | 0.33bc  | 0.33bc  | 0.33bc   | 0.33bc  | 0.33bc  | 0.33bc   | 0.33bc  | 0.33bc   | 0.33bc  |
| Propanoic acid, 2-hydroxy-ethyl ester | 0.33bc  | 0.12bc   | 0.12bc   | 0.12bc   | 0.12bc  | 0.12bc  | 0.12bc  | 0.12bc  | 0.12bc   | 0.12bc  | 0.12bc  | 0.12bc   | 0.12bc  | 0.12bc   | 0.12bc  |
| Ethyl(-)-lactate     | 53.14   | 56.36    | 56.36    | 56.36    | 56.36   | 56.36   | 56.36   | 56.36   | 56.36    | 56.36   | 56.36   | 56.36    | 56.36   | 56.36    | 56.36   |
| N-nonanoic acid, ethyl ester | 3.547   | 0.43b    | 1.70c    | 1.70c    | 1.70c   | 1.70c   | 1.70c   | 1.70c   | 1.70c    | 1.70c   | 1.70c   | 1.70c    | 1.70c   | 1.70c    | 1.70c   |
| Trans-2-hexenal-acetate | 3.14c   | 0.64b    | 0.97bc   | 1.13c    | 1.25c   | 1.09c   | 1.09c   | 1.09c   | 1.09c    | 1.09c   | 1.09c   | 1.09c    | 1.09c   | 1.09c    | 1.09c   |
| Propanoic acid, 3-methyl, ethyl ester | 3.411   | 0.27bc   | 0.30d    | 0.30d    | 0.30d   | 0.30d   | 0.30d   | 0.30d   | 0.30d    | 0.30d   | 0.30d   | 0.30d    | 0.30d   | 0.30d    | 0.30d   |
| Pentadecanoic acid, 3-methylbutyl ester | 80.3    | 119.1    | 105.8    | 121.8    | 118.5   | 149.1   | 149.1   | 149.1   | 149.1    | 149.1   | 149.1   | 149.1    | 149.1   | 149.1    | 149.1   |
| HFr                  | 1.09d   | 1.09d    | 1.09d    | 1.09d    | 1.09d   | 1.09d   | 1.09d   | 1.09d   | 1.09d    | 1.09d   | 1.09d   | 1.09d    | 1.09d   | 1.09d    | 1.09d   |

Table 5. Quantitative analysis of the aroma compounds of wines aged with oak chips. All values are expressed as means (µg/mL) ± standard deviation (SD).
| Compounds                          | CON | NFr | MFr | HFr | MCh | MAm | NFr | MFr | HFr | MCh | MAm | NFr | MFr | HFr | MCh | MAm | HFr: MCh | HFr: MAm | HFr: HCh | HFr: MAm |
|-----------------------------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-------|--------|--------|--------|--------|
| Butanoic acid, diethyl ester      | 1.406 | 1.632 | 1.458 | 1.451 | 1.427 | 1.311 | 1.735 | 1.675 | 1.853 | 1.763 | 1.550 | 1.093 | 1.832 | 1.521 | 1.879 | ±      | ±      | ±      | ±      |
| Benzoic acid, ethyl ester         | 0.22a | 0.30a | 0.44a | 0.33a | 0.57a | 0.22a | 0.41a | 0.32a | 0.64a | 0.94a | 0.41a | 0.77a | 0.63a | 0.83a | 0.98a | 0.22c | 0.48bc | 0.63bc | 0.98c  | 0.98c  |
| Propanoic acid, 2-hydroxy-ethyl ester | 0.16b | 0.29b | 0.42a | 0.21b | 0.43a | 0.39a | 0.35a | 0.48a | 0.88a | 0.58a | 0.72c | 0.34bc | 0.87a | 0.92a | 0.79a | 0.37a | 0.89bc | 0.22c | 0.48bc | 0.63bc | 0.98c  |
| Butanedioic acid, diethyl ester    | 0.947 | 10.20 | 53.03 | 1.871 | 8.171 | 6.365 | 11.12 | 75.16 | 22.79 | 25.84 | 12.4 | 3.61 | 21.84 | 3.87 | 13.53 | 2.08b | 15.84 | ±      | ±      | ±      | ±      |
| 2-Phenyl acetate                   | 0.64a | 0.94d | 0.90d | 0.36f | 0.17f | 0.72f | 0.65d | 0.54c | 0.53b | 0.47e | 0.33a | 0.13f | 0.57de | 0.51f | 0.32d | 0.34e | 0.05d | 0.77f | 0.48f | 0.62f  |
| Methyl dihydro-isonicotinate       | 0.03d | 0.20d | 0.27c | 0.99d | 0.73f | 0.84d | 0.74f | 0.67d | 0.11f | 0.94f | 0.76f | 0.49f | 0.16de | 0.94b | 0.89de | 0.32e | 0.77f | 0.48f | 0.62f  |
| Butanoic acid, hydroxy-ethyl ester | 90.04 | 1.591 | 1.741 | 90.1 | 92.7 | 92.4 | 126.5 | 110.1 | 243.1 | 124.1 | 81.9 | 128.2 | 111.2 | 109.9 | 6.183 | 120.9 | 1.701 | 110.5 | 108.1 | 124.0 |
| Ethyl succinate                   | 4.45e | 3.92g | 2.09g | 4.18h | 4.26e | 0.47e | 1.79b | 2.50c | 0.89g | 1.43b | 2.63a | 3.86c | 4.14d | 1.54c | 1.15g | 3.77b | 1.04g | 1.66c | 1.74d | 0.75b  |
| 3-methylbutyl succinate           | 38.05 | 50.13 | 43.06 | 37.99 | 43.51 | 42.77 | 32.99 | 52.62 | 59.45 | 54.76 | 43.56 | 38.53 | 58.63 | 55.05 | 46.85 | 50.68 | 55.49 | 48.18 | 54.43 | 51.01 | 43.56 |
| Tetradecanoic acid, ethyl ester    | 1.04c | 1.54b | 1.06c | 2.99c | 2.16c | 3.22c | 1.00c | 1.61b | 1.82c | 2.09b | 2.10c | 1.23c | 1.13c | 2.07ab | 1.82bc | 1.22bc | 2.27ab | 3.02ab | 0.94bc | 3.05ab | 3.44b  | 3.45a  |
| Hexadecanoic acid, methyl ester   | 1.19a | 2.04b | 1.82b | 1.42b | 2.28b | 3.08b | 3.01b | 1.73b | 1.96ab | 4.23b | 2.45b | 1.65b | 1.54ab | 2.77b | 0.91b | 3.82b | 2.40ab | 1.80a | 1.23b | 1.67b | 3.02ab | 1.92b  |
| Ethyl hydrogen succinate          | 0.62a | 0.69a | 0.89a | 0.10a | 0.41a | 0.12a | 0.36a | 0.61a | 0.45a | 0.18a | 0.41a | 0.59a | 0.88a | 0.38a | 0.42a | 0.98a | 0.69a | 0.18a | 0.51a | 0.41a | 0.59a  |
| Hexadecanoic acid                 | 118.0 | 161.8 | 157.1 | 172.8 | 151.7 | 109.4 | 4.033 | 251.6d | 1.98a | 1.06d | 3.89d | 2.46b | 1.43e | 1.28bc | 3.85e | 3.23d | 3.13c | 2.33gh | 2.62c | 4.17c  | 1.76d  |
| 2-hydroxyethyl ester Total        | 614.7 | 666.7 | 631.4 | 624.2 | 722.9 | 646.4 | 652.2 | 831 | 915 | 973 | 676.8 | 624.6 | 1039 | 852 | 759.1 | 817 | 785.3 | 892 | 568.5 | 852 | 913 | 801  |
| Compounds      | CON | NFr | MFr | HFr | MCh | HCh | MAm |
|---------------|-----|-----|-----|-----|-----|-----|-----|
| 1-Propanol    |     |     |     |     |     |     |     |
| 1-Butanol     |     |     |     |     |     |     |     |
| 1-Butanol, 3- |     |     |     |     |     |     |     |
| Methylpentanol|     |     |     |     |     |     |     |
| 1-Propanol, 3- |     |     |     |     |     |     |     |
| 2-Octen-1-ol |     |     |     |     |     |     |     |
| 2-Hexen-1-ol  |     |     |     |     |     |     |     |
| 1-Octen-3-ol  |     |     |     |     |     |     |     |
| 2-Octen-1-ol, (E)- |     |     |     |     |     |     |     |
| 3-Octen-1-ol, (Z)- |     |     |     |     |     |     |     |
| 1-Heptanol    |     |     |     |     |     |     |     |
| 2,3-Butanediol, [R-(R*, R*)]- |     |     |     |     |     |     |     |
| 2-Octen-1-ol, (E) |     |     |     |     |     |     |     |

| Alcohols      |     |     |     |     |     |     |     |
|---------------|-----|-----|-----|-----|-----|-----|-----|
| NFr:          |     |     |     |     |     |     |     |
| MCh:          |     |     |     |     |     |     |     |
| MAm:          |     |     |     |     |     |     |     |
Table 5. Cont.

| Compounds                        | CON         | NFr | MFr | HFr | MCh | HCh | MAm | NFr: MFr | HFr: HCh | MCh: HCh | MAm: HCh |
|----------------------------------|-------------|-----|-----|-----|-----|-----|-----|---------|---------|----------|----------|
| A-terpineol                      | 25.86       | 26.14 | 25.31 | 19.82 | 29.97 | 24.27 | 23.64 | 30.19 | 29.87 | 28.28 | 22.33 | 15.86 | 37.82 | 34.55 | 31.52 | 24.91 | 20.58 | 21.03 | 12.19 | 26.12 | 20.17 | 22.33 |
| 3-(Methylthio) propanol          | 1.48a       | 2.29ab | 1.93ab | 4.44ab | 2.47ab | 1.04ab | 1.62ab | 1.26ab | 2.42ab | 1.50ab | 2.24ab | 3.62ab | 1.28ab | 1.20b | 3.57b | 4.39b | 1.01b | 1.29b | 2.24bc | 0.96bc | 2.57c | 1.31c |
| Benzyalcohol                     | 0.82a       | 0.62ab | 0.53ab | 0.88ab | 0.63ab | 0.37ab | 0.2ab | 0.41ab | 0.72b | 0.31b | 0.73b | 0.39b | 0.67b | - | - | 0.38b | 0.68b | 1.33b | 0.52b | b | 0.94b | 0.66b | 0.26b |
| Pheny lethyl alcohol             | 11.4c       | 3.99e | 3.76c | 2.84c | 1.32c | 1.12bc | 1.35c | 2.35bc | 3.55a | 2.37bc | 3.55a | 1.10c | 1.32bc | 0.96bc | 1.73bc | 3.87bc | 0.71bc | 2.67b | 1.39d | 2.55bc | 2.68bc | 1.81c |
| Benzenethanol, 4-hydroxy-        | 65.33       | 94.5 | 78.53 | 80.0 | 84.6 | ± | ± | 92.4 | ± | ± | 5.29 | ± | ± | ± | ± | ± | ± | ± | ± | ± | ± |
| Total                            | 886.5       | 1035.7 | 1043.6 | 873 | 1096.2 | 1054.4 | 1116.9 | 938.0 | 938.0 | 891.2 | 1041.0 | 1056.0 | 1217.4 | 1308.9 | 1186.7 | 998.4 | 1144.8 | 1134.9 | 998.3 |

| Acids                            |             |     |     |     |     |     |     |     |     |     |     |
|----------------------------------|-------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Acetic acid                      | 345.0       | 234.8 | 256.9 | 205.1 | 260.8 | 252.7 | 159.7 | 213.3 | 170.5 | 223.8 | 201.4 | 244.1 | 264.4 | 218.2 | 204.8 | 218.1 | 4243 | ± | ± | ± |
| Butyric acid                     | 7.647       | 9.61 | 10.25 | 7.573 | 8.50 | ± | ± | 6.408 | ± | ± | 10.89 | 14.66 | 21.40 | 7.861 | 21.91 | 9.27 | ± | ± | ± | ± | ± |
| Pentanoic acid                   | 0.16d       | 0.68d | 0.25cd | 0.95e | 0.66d | 0.26e | 0.50cd | 0.49b | 0.89a | 0.44de | ± | ± | ± | ± | - | 0.10a | - | - | - | ± | ± |
| Hexanoic acid                    | 3.22d       | 3.98e | 2.83c | 1.31c | 2.13c | 3.37c | 4.39b | 3.46ab | 2.78ab | 4.14c | 1.37c | 4.20bc | - | - | 12.55 | 14.27 | 13.31 | 2.030 | - | - | - |
| n-Decanoic acid                  | 7.675       | 7.952 | 5.402 | 5.748 | 6.456 | 5.681 | 4.151 | 4.837 | 11.24 | 7.849 | 2.982 | 3.771 | 11.69 | 4.746 | 5.641 | 7.841 | 5.178 | 10.43 | 4.642 | ± | ± | ± |
| n-Hexadecanoic acid              | 0.16c       | 0.51b | 0.76cd | 0.84cd | 0.19cd | 0.71cd | 0.41d | 0.64cd | 0.31b | 0.66bc | 0.13cd | 0.76d | 0.17a | 0.35cd | 0.33bc | 0.33bc | 0.46cd | 0.88b | 0.77d | ± | ± | ± |
| Total                            | 11.77       | 12.53 | 10.14 | 12.37 | 13.27 | 6.534 | 13.92 | 21.30 | 22.26 | 13.32 | 12.05 | ± | ± | ± | ± | - | 19.08 | - | 11.83 | 15.27 | 22.26 | - | - | - |
| Aldehydes                        |             |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |     |
| Furfural                         | 1.092       | 0.915 | 0.885 | 0.930 | 0.845 | 1.088 | 0.880 | 1.312 | 1.816 | 0.7282 | 0.7009 | 1.007 | 1.097 | 0.7858 | 1.016 | 1.829 | 1.108 | 0.872 | 0.7372 | 1.004 | 0.7822 |
| 2- Furanaldehyde, 5-methyl-       | 2.101       | 2.342 | 1.829 | 2.030 | 1.906 | 1.898 | 1.773 | 2.496 | 2.745 | 2.931 | 2.221 | 1.778 | 2.571 | 2.398 | 2.188 | 2.445 | 3.033 | 2.779 | 2.158 | 2.471 | 2.343 | 2.221 |
| Total                            | 2.101       | 3.376 | 2.744 | 2.836 | 2.751 | 2.743 | 2.681 | 3.376 | 4.057 | 4.747 | 2.949 | 2.479 | 3.599 | 3.495 | 2.974 | 3.461 | 3.033 | 3.887 | 2.096 | 3.208 | 3.347 | 2.949 |
**Table 5. Cont.**

| Compounds                  | CON  | NFr | MFr | HFr | MCh | MCh | MAm  |
|----------------------------|------|-----|-----|-----|-----|-----|------|
|                           |      |     |     |     |     |     |      |
| Phenol, 2-methoxy-phenol   | 2.788| 3.426| 2.548| 2.848| 3.118| 2.057| 3.323| 4.326|
|                           | 0.28c| 0.25bc| 0.81c| 0.17c| 0.78bc| 0.29c| 0.20bc| 0.14b|
| Phenol, 4-ethyl-phenol     | 2.792| 4.586| 2.962| 4.550| 2.351| 5.648| 4.415| 7.429|
|                           | 0.88c| 0.67bc| 0.54c| 0.53bc| 0.47c| 0.93bc| 0.48bc| 0.40ab|
| Phenol, 4-ethyl-2-methoxy- | 8.36  | 20.17 | 8.75 | 12.99 | 8.33  | 3.97 | 22.99 | 21.93 |
|                           | 3.02c| 2.02bc| 0.63c| 3.40c| 2.83c| 2.63ab| 3.28bc| 0.97a|
| 4-Vinyl phenol            | 2.548| 1.363| 1.701| 1.584| 2.198| 1.162| 1.132| 1.337|
|                           | 0.86a| 0.93a | 0.84a| 0.89a| 0.19a| 0.28a | 0.18a | 0.37a|
| Phenol, 2,6- | 13.58 | 6.170| 5.574| 8.65 | 6.320| 5.513| 5.460| 4.410|
| dimethoxy-phenol          |     |     |     |     |     |     |     |     |
|                           | 0.99a| 0.29c | 0.34c| 0.04b| 0.76c| 0.34cd| 0.75cd| 0.14cd|
| DL-a-Phenylactic acid     | 5.826| 6.587| 5.144| 6.666| 5.206| 5.375| 4.529| 5.809|
|                           | 0.13c| 0.56bc| 0.16c| 0.82bc| 0.87c| 0.14c | 0.14c | 0.19c|
| 2,4-Di-tert-butyl/phenol  | 45.72 | 47.52 | 45.86 | 38.57 | 42.33 | 40.36 | 34.91 | 52.61 |
|                           | 3.50cd| 1.31cd| 4.12cd| 2.45cd| 2.51cd| 2.85cd| 1.45cd| 3.42bc|
| 2,5-Dimethylphenol        | 1.63ab| 1.51ab| 0.66ab| 3.09a | 2.74ab| 1.49ab| 0.51ab| 0.54ab|
|                           | 93.7 | 96.7 | 83.1 | 89.9 | 81.0 | 91.9 | 74.43 | 113.2 |
|                           |     |     |     |     |     |     |     |     |
| Volatile phenols          |      |     |     |     |     |     |     |     |

Different letters in a column indicate significant differences at p < 0.05; statistically, a, b, c, and d following the values indicate significant differences among these values.
Figure 1. Heatmap representation of the GC–MS-determined aroma content of compounds in wines aged with different oak chips. In normalized mapping, the negative value is lower than the average value of all numbers, and the aroma content increases from red to green.

3.3.1. Esters

Esters give wines their primary fruit and floral aromas and contribute substantially to the flavor of wine [28]. Ester molecules are compounds formed by the condensation of a hydroxyl group of a phenol or alcohol and a carboxyl group from an organic acid. As one of the most important volatile constituents in grape wine, esters also directly influence the aromatic profiles and sensory perception of wines. In this study, a total of 24 esters were detected, most of which were acetate esters and ethyl esters of fatty acids. It was notable that the ester contents increased significantly after aging. In 21 oak-chip-treated samples, the ester compounds presenting high content were isopropyl acetate, ethyl lactate, pentadecanoic acid, 3-methylbutyl ester, butanoic acid, hydroxy-, diethyl ester, and ethyl hydrogen succinate. The isopropyl acetate content in the wine aged with MFr:HFr increased by more than two times, while the ethyl lactate content increased between 3.42% and 42.55% after aging. Butanoic acid and diethyl ester increased by between 11.85% and 71.30%. The change in ester contents due to aging may provide rich flower and fruit fragrances for the wines.

3.3.2. Alcohols

Alcohols are generally considered to be the aromatic compounds with the greatest impact on the aroma of wine [29,30]. Excessive concentrations of alcohols can result in a strong, pungent smell and taste, whereas optimal levels impart fruity characteristics [31].
strong, pungent smell and taste, whereas optimal levels impart fruity characteristics [31]. A total of 21 alcohol compounds were detected in this study, and there was an overall increase in the alcohol contents of the V. amurensis wine after oak-chip aging (Table 5). The increase in alcohol content was more pronounced for the wines aged with NFr:MFm, NFr:HFr, NFr:MCh, MFm:HFr, and MFm:MAm. It was found that 3-methyl-1-butanol and phenylethyl alcohol were abundant in the wine, and studies have shown that they have cheese, honey, and rose aromas, respectively [32]. In addition, 1-pentanol and 1-hexanol were also detected. While 1-pentanol is known to have a mellow flavor, 1-hexanol is said to taste of grass and toast [33]. When these compounds are combined, the flavors in the wine may change, making the aroma of V. amurensis wine more complex and layered.

3.3.3. Acids

The organic acids in wine come primarily from the berries (grapes) and are precursors for the synthesis of esters, which can increase the mellowness of wine. Moreover, organic acids have preservative properties and increase the physical and chemical stability of wine. It has been reported that, at appropriate levels, the organic acids play an important role in the aromatic equilibrium of wine, mainly because they restrict the hydrolysis of the relevant esters and maintain a high content of aromatic esters [34]. Having an appropriate amount of organic acid is important in V. amurensis wine. The acetic acid content is high and usually constitutes about 90% of the volatile acids in wine [35]. Six organic acids were identified in this study. After aging with oak chips, the organic acid content in V. amurensis wine decreased significantly, thus alleviating the high acidity of the product. A previous study considered that decanoic acid (fatty and unpleasant notes) negatively affected the overall wine aroma [34]. We found small amounts of decanoic acid in V. amurensis wine. In addition, the hexanoic acid content, which smells of cat urine and sweat, decreased significantly with oak-chip aging. A low concentration of hexanoic acid was only detected in the wines aged with HFr, NFr:MFm, MFm:MAm, HFr:MCh, HFr:HCh, and HFr:MAm.

The overall content of the eight volatile phenols detected in this study was not high and increasing or decreasing trends were not apparent. We found that 4-viny phenol showed no significant change after aging, consistent with previous research results [36]. Another important volatile compound detected in oak-chip-aged V. amurensis wine was furfural, which might result from the decomposition of pentose, mainly from hemi-cellulose in oak chips. The increased furfural might add fragrance, fruit, and flower aromas to the V. amurensis wine [32].

In summary, after oak-chip aging, the total aroma component contents in 21 kinds of aged V. amurensis wine increased. Specifically, the content of alcohols and esters increased significantly, while the content of organic acid compounds decreased, which may have been due to the esterification reaction. The highest concentrations of volatile compounds were found in the wines aged with MFm:HFr, NFr:HFm, and NFr:MCh up to 3.011 g/L, 2.863 g/L, and 2.905 g/L. However, some of the aroma components occurred at low levels, and, combined with other minor compounds, may provide delicate background aromas that contribute to the complexity and equilibrium of the overall varietal aroma. At the same time, it can be seen from Figure 1 that, NFr:HFr, NFr:MCh, MFm:MAm, and HFr:MCh have an overall aging effect, and the effect of mixed aging is more pronounced than that of single aging.

3.4. Sensory Evaluation by Panelists

Table 6 shows the sensory evaluation results for the V. amurensis wines aged with different kinds of oak chips. After the treatment with oak chips, the total scores of the sensory evaluation were higher than those of the control, which meant a total sensory quality promotion. As well as the control, the wine treated with HFm, HCh, and MAm oak chips received a lower sensory evaluation score than others due to the poor taste and structure as well as an inadequate aftertaste. The wines treated with NFr:HFm, NFr:MCh, and MFm:MAm oak chips obtained the highest scores, with a clear, shiny body, typical
varietal aromas, fresh fruity flavors, and a good, balanced aftertaste. According to the test panel, the wines aged in contact with MAm added “vanilla” and “toast” aromas. Moreover, the wines aged with NFr, MFr, and MCh also had rich aromas, with some “vanilla”, “toast”, and “smoky” aromas added, but they were not as obvious as those of MAm-treated wine. It was found that HFr and HCh were too heavy to cover their fruit aromas. At the same time, there were some pleasant, toasted-nut aromas in the heavily toasted group. In addition, the addition of mixed oak chips enriched the aroma but also produced some adverse effects. Notably, MAm:NFr had a better aftertaste, and MAm:HCh had a longer aftertaste. Among the four groups of MAm, the MCh:MAm and HCh:MAm produced some less-pleasant smells of overripe fruit, with MAm:HCh being slightly more astringent. Among the five groups treated with HFr, MAm, MCh, and MFr had a better performance. The wine samples treated with HFr:MFr had rich chocolate and fruit flavors.

Table 6. Sensory evaluation of wines after oak chip additions.

| Sample    | Appearance | Aroma | Taste | Typicality | Clarity | Total Scores |
|-----------|------------|-------|-------|------------|---------|--------------|
| Control   | 3          | 6     | 3     | 1.5        | 1.6     | 13.7         |
| NFr       | 2.4        | 4.8   | 4.6   | 1.4        | 1.6     | 14.8         |
| MFr       | 2.3        | 4.8   | 4.5   | 1.7        | 1.5     | 14.8         |
| HFr       | 2.4        | 4.5   | 4.4   | 1.5        | 1.6     | 14.4         |
| MCh       | 2.5        | 4.5   | 4.5   | 1.8        | 1.7     | 15.0         |
| HCh       | 2.5        | 4.2   | 4.3   | 1.9        | 1.5     | 14.4         |
| MAm       | 2.3        | 4.6   | 4.4   | 1.5        | 1.6     | 14.4         |
| NFr:MFr   | 2.4        | 4.7   | 4.6   | 1.4        | 1.6     | 14.9         |
| NFr:HFr   | 2.5        | 4.8   | 4.4   | 2.0        | 1.4     | 15.1         |
| NFr:MCh   | 2.6        | 4.8   | 4.4   | 1.8        | 1.5     | 15.1         |
| NFr:HCh   | 2.3        | 4.7   | 4.2   | 1.7        | 1.6     | 14.5         |
| NFr:MAm   | 2.3        | 4.6   | 4.7   | 1.8        | 1.5     | 14.9         |
| MFr:HFr   | 2.2        | 4.9   | 4.5   | 1.9        | 1.5     | 15.0         |
| MFr:MCh   | 2.5        | 4.4   | 4.3   | 1.7        | 1.8     | 14.7         |
| MFr:HCh   | 2.3        | 4.6   | 4.5   | 1.6        | 1.5     | 14.5         |
| MFr:MAm   | 2.6        | 4.5   | 4.5   | 1.9        | 1.6     | 15.1         |
| HFr:MCh   | 2.2        | 4.8   | 4.4   | 1.8        | 1.4     | 14.6         |
| HFr:HCh   | 2.6        | 5     | 4.2   | 1.4        | 1.4     | 14.6         |
| HFr:MAm   | 2.4        | 4.8   | 4.1   | 1.8        | 1.5     | 14.6         |
| MCh:HCh   | 2.5        | 4.7   | 4.3   | 1.7        | 1.5     | 14.7         |
| MCh:MAm   | 2.2        | 4.7   | 4.8   | 1.7        | 1.6     | 15.0         |
| HCh:MAm   | 2.4        | 4.6   | 4.3   | 1.6        | 1.7     | 14.6         |

3.5. Electronic Tongue (E-Tongue) Evaluation

3.5.1. Principal Component Analysis (PCA)

The principal component analysis clearly distinguished 21 kinds of aged V. amurensis wines, indicating that the e-tongue could evaluate the taste differences of the oak-chip aged wines to some extent (Figure 2). The first two principal components possessed 82.2% of the total variance (71.9% and 10.3% for PC1 and PC2), which showed that these factors were sufficiently important to warrant further discussion. As can be seen from Figure 2, 22 kinds of V. amurensis wines aged with different oak chips can be divided into three groups. Among them, the control and V. amurensis wines with NFr, MFr, HFr, MCh, and MAm oak chips as the first group; V. amurensis wines with HCh, NFr:MFr, NFr:HFr, NFr:MAm, MFr:HFr, MFr:MCh, MFr:HCh, MFr:MAm, HFr:MCh, HFr:HCh, HFr:MAm, and MCh:MAm oak chips as the second group; and the rest as the third group. The distinction between these three groups of V. amurensis wines was obvious. Among the six kinds of V. amurensis wines aged with just one type of oak chip, except for the V. amurensis grapes with heavily roasted Chinese oak chips (HCh), the rest of the V. amurensis wines were in the same group as the control wine. Although these wines were quite similar to the control, they were also slightly
different. The HCh and mixed-oak-chip aging tended to converge, which may be because high-temperature toasting changes the polyphenol composition and affects the flavor. The group comprising NFr:MCh, NFr:HCh, MCh:HCh, and HCh:MAm were probably Chinese oak with a strong flavor. In addition to these groups, other oak chip combinations with Chinese oak may mask some of the oak and toasty flavors of the Chinese oak.

Figure 2. PCA plot of the electronic tongue detection results of wines of *Vitis amurensis*.

3.5.2. Radar Graph of *V. amurensis* Wines

The e-tongue taste radar graph of *V. amurensis* wines with different oak chips is shown in Figure 3. The obtained data from the electronic tongue evaluation are presented in Supplementary Table S3. There was no significant difference in saltiness, astringent aftertaste (After-A), and bitter aftertaste (After-B) between the control and the other 21 oak-chip-aged wines. Compared with the control wine, it was found that the acidity of oak-chip-aged *V. amurensis* wines decreased. The sweet taste decreased in single-oak-chip-aged wines but increased in mixed-oak-chip-aged ones. The same was true for the umami taste. The e-tongue results generally supported the wine panel’s results. In the sensory evaluation by the panel, sweet and strong tastes were also detected. The mixed-oak-chip aging increased the complexity of the wines, covered up some bad smells, and also covered up some of the fruit aromas. However, the wines aged with single oak chips had a fruity aroma, which was not as layered as that of the wines aged with mixed oak chips.
4. Discussion

As shown in Table 3, the total polyphenol and tannin contents in all tested wines increased after oak-chip aging. Liu et al. studied the effect of oak chips on wine quality and found that the content of polyphenols increased after oak aging [37], which may be due to the increase in hydrolyzed tannins from the oak chips [26,27]. In the evaluation of color parameters (Table 4), the values of a* showed a downward trend, which was consistent with a study by Perez-Magarino et al., who found that red tones fell (values of a*) with aging. They explained that the loss of red tones is mainly due to the loss of free anthocyanins [38]. Regarding the values of b*, except for the fact that MFr:MCh tends toward yellow, there were no significant differences. Mateus et al. pointed out that a small amount of oxygen in the wine body can oxidize ethanol to form acetaldehyde, and acetaldehyde can be involved in the formation of polymeric pigments. Then, because polymeric pigments are mostly yellow, red wines often become lighter in color during aging [39]. This result is consistent with that of Li et al. [40].

In the GC–MS analysis (Table 5), the types and concentrations of esters and alcohols in *V. amurensis* wine accounted for a large proportion of the constituents. They are among the most important volatile components, and their contribution to the flavor cannot be ignored. We noticed that ethyl acetate was not detected after some oak aging, which was inconsistent with the research of Georgiana et al. [41]. Considering the low content of other detected components, we speculated that it had reacted with other aromatic constituents. As for 4-vinyl phenol, which is formed by the enzymatic hydrolysis or thermal decarboxylation of cinnamic acid, it exerts a smoky aroma, which is also an indicator of the relative degree of the roasting of the oak chips because it is mainly formed by the degradation of lignin during the roasting process [41,42]. Aldehydes and acids also play important roles in supplementing and modifying the flavor of wines. An appropriate amount of acid increases the taste of wine, participates in the esterification reaction, and gives the wine a fruity aroma. Six kinds of acids were detected, and the content of acetic acid was the highest. Overall, the aroma components and contents of the wine aged with
different oak chips varied, and esters and alcohols were the main aroma components. This result is generally consistent with the findings of most studies [36,41].

In the sensory evaluation by the panel, it was found that the addition of any tested oak chip could significantly strengthen the wine’s red color, i.e., from violet to ruby or garnet red, particularly when using non-toasted French oak and moderately toasted American oak. Gordillo et al. showed that the addition of oak chips promoted color enhancement and stability [43]. The oak-chip-aged wines had high sweet-taste intensity and were full-bodied. They differed from the control in that they were not as acidic and astringent, possibly due to the high sweetness intensity, which reduced the perception of acidity [44]. High levels of sweetness were also detected in the e-tongue results. Tannins gradually become softer during the aging process, and the astringency is gradually reduced. According to the test panel evaluation, the vanilla flavor of the wines might be related to a higher 1-hexanol content (Table 5), which has been reported to be responsible for the perception of a vanilla odor. In addition, 3-hexen-1-ol and trans-2-hexenyl-acetate are known to contribute to vanilla odor. Their contents were greater in the wine treated with oak chips than in the control group (Table 5). The fruit aroma in the wine is also more obvious in the aged wines than in the control. It can be seen from Table 5 that the contents of ethyl lactate and isopropyl acetate are higher in oak-chip-treated wines, providing elegant fruity and creamy flavors [36].

In summary, the mixed-oak-chip aging treatment increased the complexity of the wines, masked some bitter and astringent tastes, and also covered up some fruit aroma. However, the wine aged with single oak chips had more fruit aroma, the astringency was more obvious, and the aroma was not as layered as that in wines aged with mixed oak chips. On the basis of the above, in order to make wines of different styles, different oak chips and mixed oak chips can be selectively added, and the sensory complexity and layering can be altered by changing the oak chip treatment. If more of the fruit taste of the grape is preferred, the wine can be aged by adding single oak chips with lower toasting levels, rendering the wine softer and smoother in texture.

5. Conclusions

In this study, taking the color, aroma components, and taste as the main evaluation indexes, the effects of different oak-chip aging treatments on the sensory properties of V. amurensis wines were comprehensively analyzed. The type of oak chips should be selected according to the characteristics of V. amurensis wine. The aging process enhanced the organoleptic properties of the wine. A CIELab analysis showed that, after oak-chip aging, V. amurensis wine increased in brightness, and its color changed to ruby red. Moreover, the types of aroma components increased, with the alcohol and ester content increasing and the acid content decreasing. A combination of various aroma components gave the V. amurensis wine a unique flavor, taste, and aroma. The e-tongue technical analysis showed that the sour taste of V. amurensis wine decreased slightly with oak-chip aging, while the sweetness, astringency, freshness, and bitterness increased, and the increase in sweetness was the most obvious. After oak-chip aging, the color, aroma structure, and taste of V. amurensis wines were significantly improved, and mixed oak chips were observed to have the most satisfactory effects. Furthermore, the V. amurensis wines aged with mixed oak chips had a better appearance, aroma, and taste, with a clear and shiny body, ruby-red color, rich fruit aroma, good wood flavor, mellowness, harmoniousness, a long taste, and a rich personality. The wines aged with mixed oak chips exhibited specific characteristics and appeared to have long-aging potential.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/foods11081126/s1, Table S1: Calibration curves for quantification in this study; Table S2: Quantitative standards and calibration curves for quantification of volatile compounds in this study; Table S3: Electronic tongue data of wine made from Vitis amurensis (n = 3).
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