



# Geographical characterization of wines from seven regions of China by chemical composition combined with chemometrics: Quality characteristics of Chinese ‘Marselan’ wines

Yue Wang<sup>a,b,1</sup>, Caihong Li<sup>c,1</sup>, Qian Ge<sup>a,b,c</sup>, Xingsan Huo<sup>a</sup>, Tingting Ma<sup>b</sup>, Yulin Fang<sup>b</sup>, Xiangyu Sun<sup>a,\*</sup>

<sup>a</sup> College of Enology, Shaanxi Provincial Key Laboratory of Viti-Viniculture, Viti-viniculture Engineering Technology Center of State Forestry and Grassland Administration, Shaanxi Engineering Research Center for Viti-Viniculture, Heyang Viti-viniculture Station, Ningxia Eastern Foot of Helan Mountain Wine Station, Northwest A&F University, Yangling, 712100, China

<sup>b</sup> College of Food Science and Engineering, Northwest A&F University, Yangling, 712100, China

<sup>c</sup> Institute of Quality Standard and Testing Technology for Agro-products of Ningxia, Yinchuan, 750002, China

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## ABSTRACT

This study investigated the basic and functional compositions, volatile compounds, intelligent sensory characteristics and antioxidant capacity of the commercial ‘Marselan’ wines from seven Chinese regions. The Nei Mongol wines featured high total reducing sugar, fructose, ammonia nitrogen, 17 monomeric phenolic acids contents and elevated antioxidant capacity. Malic acid was the only organic acid that significantly different in all seven regions. Malvidin-3-O-glucoside and *trans*-peonidin-3-O-(6-O-*p*-coumaroyl)-glucoside showed the highest and lowest contents. A total of 102 volatiles was detected and Hebei wines had the most (91). Hexanoic acid and  $\beta$ -damascenone were considered to have high potential sensory effects (OAV  $\geq 1$ ) as compounds detected in all regions. Floral, sweet, and fruity were the most important aroma series. *E*-eye analysis revealed the colors of the samples tended to yellowish with aging. PCA and OPLS-DA based on the basic wine composition, monomeric organic acids and anthocyanins allowed achieving a discrimination of the seven regions, respectively.

## 1. Introduction

The ‘grape variety’ and ‘geographical identification’ are two crucial considerations for consumers when purchasing wines. Varieties have different adaptability in specific regions thus further affecting the basic wine chemical composition and style of the wine produced (Costa et al., 2015). Therefore, it is important to find a suitable variety for a specific region. Despite having some distinctive and world-famous regions, such as eastern foothills of Helan Mountain in Ningxia, China still lacks a variety that can represent the style of Chinese regions and this may make it challenging to market globally. Currently, China is the 12th largest wine-producing country with negligible wine exportation. In view of this, it is necessary to investigate the differences of Chinese wine regions and explore a representative grape variety for Chinese regions.

‘Marselan’ (*Vitis vinifera* L.) is a red wine variety from France. It exhibited excellent resistance to diseases such as *Botrytis cinerea*

(Rahman et al., 2019) and was considered to have high fruit quality (Alcalde-Eon et al., 2006; Dong et al., 2022; Lan, Liu, et al., 2022). Moreover, ‘Marselan’ wines were indicated as having elegant fruity aromas (Lyu et al., 2019). ‘Marselan’ have demonstrated good adaptability in China. Chinese ‘Marselan’ wine has won numerous awards in various international and domestic competitions. A number of ‘Marselan’ wines from the Yantai, Xinjiang, Ningxia and Hebei regions of China have been awarded Gold medals at the Berliner Wein Trophy. For three years, 2018, 2019 and 2021, Chinese ‘Marselan’ wines have won Gold medals at the Decanter World Wine Awards. In the first edition of the First Ningxia Marselan selection by concours Mondial De Bruxells (CMB), two ‘Marselan’ wines from Ningxia and Xinjiang won the Grand Gold prize among 259 ‘Marselan’ wines from 18 regions of 11 countries. The burgeoning interest of scholars and wine enthusiasts in Chinese ‘Marselan’ wine prompts contemplation regarding its potential as a representative variety of China’s different regions. A study investigated

\* Corresponding author.

E-mail address: [sunxiangyu@nwfufu.edu.cn](mailto:sunxiangyu@nwfufu.edu.cn) (X. Sun).

<sup>1</sup> These authors contributed equally to this work.

the aroma characteristics of ‘Marselan’ wines from Xinjiang but was constrained by the inadequate sample size (Lyu et al., 2019). ‘Marselan’ wines from some regions of China had been geographically distinguished successfully by volatile compounds (Lan, Wang, et al., 2022). Li et al. (2022) had confirmed the differences among the microbial terroirs of major ‘Marselan’ wine regions in China. The preceding studies suggest the potential possibility of characteristic differences among ‘Marselan’ wines from diverse regions in China. However, there remains scope for further investigation into the comprehensive characteristics of Chinese ‘Marselan’ wines from varying regions. Commercial wine is commonly employed to investigate regional characteristics, while the utilization of commercial wines from various wineries within the same region could mitigate interference from diverse oenological techniques (Duley et al., 2021; Ranaweera et al., 2021). Overall, the lack of representative varieties adapted to regions and the insufficient exploration of regional styles have hindered the development of Chinese wine industry. Comprehensive investigation of Chinese ‘Marselan’ wines along side comparative analysis across different regions is imperative. It will also contribute to developing the quality characteristics of different Chinese regions.

In this study, 50 commercial ‘Marselan’ wines from seven regions (Ningxia, Hebei, Shandong, Xinjian, Nei Mongol, Gansu, and Beijing) of China with considerably different terroir characteristics were selected. The seven regions include the major and famous grape producing sub-regions in China such as *eastern foothills of Helan Mountain* in Ningxia, *Changli* in Hebei, *Yantai* in Shandong, *Yili River Valley* in Xinjiang, *Wuhai* in Nei Mongol, and *Wuwei* in Gansu, etc. Despite deviating from conventional large-scale viticulture and mass production methods, Beijing retains its distinctiveness within the Chinese wine industry, warranting its inclusion in the sample selection, albeit in limited numbers due to its small-scale characteristic. In addition, the climate of selected regions covers the major climatic conditions for ‘Marselan’ cultivation in China. All the selected samples of ‘Marselan’ wines were free from quality defects and were evaluated as typical by experts. They were analyzed for basic wine composition, phenol compounds, organic acids, volatile compounds, and antioxidant capacity to investigate the characteristics

of the seven regions. Meanwhile, multivariate analysis was performed to distinguish the geographical characteristics of different regions.

## 2. Materials and methods

### 2.1. Samples and chemicals

Fifty commercially available typical ‘Marselan’ red wines from seven regions in China (Ningxia, Hebei, Shandong, Xinjian, Nei Mongol, Gansu, and Beijing; Fig. 1) were selected by a panel of wine experts and practitioners. Each wine sample had two replicates. The basic information was shown in Table S1.

High-performance liquid chromatography (HPLC)-grade methyl alcohol, acetic acid, acetonitrile, and ethyl acetate were purchased from Merck (Darmstadt, Germany). Organic acid standards, anthocyanin standards, monomeric phenolic acid standards, and 6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox) were purchased from Sigma-Aldrich (St. Louis, MO, USA). All analytical-reagent grade chemicals were purchased from Xilong Chemical Industry Co. Ltd. (Chengdu, China).

### 2.2. Basic wine composition

The basic chemical composition (including reducing sugar, fructose, glucose, total acidity, total sulfite, free sulfite, ammonia nitrogen and alcohol content) as well as total phenol and total anthocyanin were determined by an Enology Y15 automatic analyzer (Biosystems, Spain).

### 2.3. Monomeric organic acid

Organic acids were quantified by Waters 2699 high-performance liquid chromatography (HPLC) equipped with a C18 chromatographic column (4.6 × 250 mm; 5 μm; CAPCELL PAK C18; Shiseido, Japan) as described by Gao et al. (2004). Mobile phase A consisted of 0.02 M dipotassium hydrogen phosphate (pH 2.3) and mobile phase B was methanol. Elution was conducted with A/B in a ratio of 99:1, at a flow

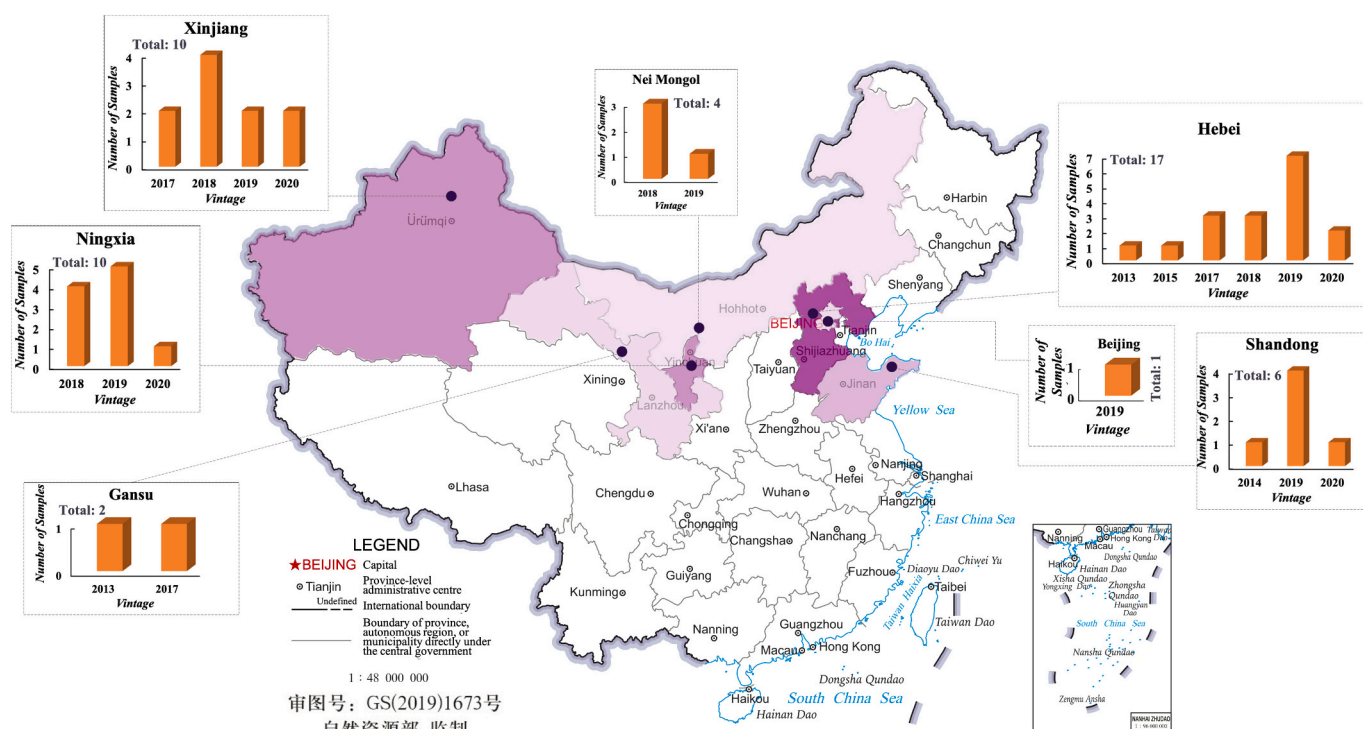


Fig. 1. Locations of the wine samples collected from China seven regions.

rate of 0.5 mL/min. Identification was based on retention times of standards. Each sample was assessed in triplicate.

#### 2.4. Monomeric anthocyanin

Monomeric anthocyanins were determined by Shimadzu LC-20AT (Shimadzu, Suzhou, China) HPLC system equipped with a photodiode array detector as previously described by Yang et al. (2018). Separations were performed with a Synergi Hydro-RP C18 column (250 mm × 4.6 mm, 4 μm, Phenomenex, Torrance, CA, USA) at a flow rate of 1 mL/min. The mobile phase A consisted of 2.5% formic acid (v/v) in solution (water/acetonitrile = 8:1, v/v) and mobile phase B was 2.5% formic acid (v/v) in solution (water/acetonitrile = 4:5, v/v). The gradient was as follows: 0–45 min, 0–35% B; 45–46 min, 35%–100% B; 46–50 min, 100% B isocratic; 50–51 min, 100%–0 B; and 51–55 min, 100% A isocratic. The photodiode array detector was set at 520 nm. Malvidin-3-O-glucoside was used as an external anthocyanin standard for the quantitation of all the anthocyanins in the wine. Each sample was assessed in triplicate.

#### 2.5. Monomeric phenolic acid

The sample pretreatment was carried out as follows: 30 mL wine sample was added to 60 mL ethyl acetate for extraction, and the experiment repeated three times. The samples were rotary-evaporated to dryness at 35 °C under reduced pressure and were adjusted to 10 mL by HPLC-grade methanol. Filtration was through a 0.22 μm organic filter and then stored at –40 °C until analysis.

Monomeric phenolic acids were characterized by Shimadzu LC-15C (Shimadzu, Suzhou, China) high performance liquid chromatograph with a Waters × Terra MS C18 column (250 mm × 4.6 mm, 5 μm) at 30 °C. The mobile phase A consisted of 2% aqueous acetic acid (v/v) and mobile phase B was 0.5% acetic acid (v/v) in aqueous acetonitrile (water/acetonitrile = 1:1, v/v). The elution was conducted with A/B in a ratio of 1:1, using the following gradient: 0–50 min, 10%–55% A; 50–60 min, 55%–100% A; 60–65 min, 100%–10% A; 65–75 min, 10% A. 20 μL sample was injected and the flow rate was 0.8 mL/min. The detection wavelength was 280 nm for flavan-3-alcohols and dihydrochalcones, 320 nm for hydroxycinnamic acids, and 360 nm for flavonols. Identification was based on retention times of standards and external standards were used for quantitation. Each sample was assessed in triplicate.

#### 2.6. Antioxidant capacity

Antioxidant capacity was evaluated using the following three methods. The free radical scavenging of 1,1-diphenyl-2-picrylhydrazyl (DPPH) and ferric reducing ability of plasma (FRAP) were determined according to Ma et al. (2020), with slight modifications. The method of cupric ion reducing antioxidant capacity (CUPRAC) assay was modified as described by Cheng et al. (2020). All results were expressed in mmol Trolox equivalents/L (mM TE/g). Each sample was assessed in triplicate.

#### 2.7. Volatile compounds

Volatile compounds were analyzed by Headspace–solid phase microextraction–gas chromatography–mass spectrometry (HS-SPME-GC-MS) using GC2030-TQ8050 NX (Shimadzu, Kyoto, Japan) triple quadrupole gas chromatography-mass spectrometer, equipped with InertCap WAX polar column (60 m × 0.25 mm, 0.25 μm, Shimadzu, Kyoto, Japan) and an AOC-6000 (Shimadzu, Kyoto, Japan) according to Ge et al. (2021). The wine sample was placed into a 20 mL vial capped with a PTFE-silicon septum. After incubation at 45 °C for 5 min, the sample was extracted by a DVB/CAR/PDMS 50/30 μm SPME fiber (Supelco, Bellefonte, PA) for 30 min with agitating at 250 rpm. The fiber was then thermally desorbed at the injection port for 3 min. The flow of the carrier gas (He) was splitless at a flow rate of 0.8 mL/min. The ion

source temperature was 230 °C. The oven temperature procedure was as follows: the initial temperature of 40 °C was held for 5 min, then increased to 120 °C at 3 °C/min, and increased by 8 °C/min to 230 °C holding for 10 min. Each sample was assessed in triplicate.

The odor activity value (OAV) was calculated as the ratio between the concentration of the volatile compound and its odor threshold, as described in the literature. Volatile compounds with OAVs ≥ 1 were considered to be compounds that may have important effects on aroma profiles (Marcq & Schieberle, 2021), and therefore further analysis was performed on these compounds.

#### 2.8. Intelligent sensory characterization

##### 2.8.1. E-eye color analysis

The lightness ( $L^*$ ), green/red component ( $a^*$ ), yellow/blue component ( $b^*$ ), chroma ( $C^*$ ) and hue ( $h^\circ$ ) of samples were determined by W100 wine color analyzer (Hanon Advanced Technology Group Co., Ltd., China). Each sample was assessed in triplicate.

##### 2.8.2. E-nose analysis

PEN 3 (Airsense Analytics, Schwerin, Germany) electronic nose was used to evaluate the overall odor characteristics of the 'Marselan' wine samples as described by Lan, Liu, et al. (2022), with slight modifications.

#### 2.9. Statistics analysis

Date organization and processing was carried out by Excel 16.54 (Microsoft, Redmond, WA, USA) and SPSS 26.0 (IBM, Armonk, NY, USA). GraphPad Prism 9.3.1 (GraphPad Software, CA, USA), TBtools (<https://github.com/CJ-Chen/TBtools/releases> accessed on: September 2, 2021), SIMCA 14.1 (Sartorius Corporate, Germany), and R (version 4.0.3) were used for analysis. Color simulation was conducted on Adobe Photoshop CC 2019 (Adobe Systems Incorporated, CA, USA).

### 3. Results and discussion

#### 3.1. Basic wine composition

As previously described by Parr et al. (2013), the chemical properties of wine were closely related to many factors, such as vineyard and oenological decisions. Moreover, the differences between wineries in the same region were even greater than between different regions (Li et al., 2022). Therefore, the values of the same region were averaged respectively to focus as much as possible on the characteristics of the region rather than the differences between the wineries.

##### 3.1.1. Basic wine composition of the seven regions

The reducing sugar content of wine is largely affected by the oenological strategy, which is mainly related to the ripening status and weather conditions (Godden et al., 2015). The reducing sugar content was significantly different between all seven regions ( $P < 0.05$ ). The reducing sugar (3.25 g/L) and alcohol content (15.83%) of Nei Mongol was highest among all regions (Fig. S1). The northwest region of China, such as Nei Mongol, is generally abundant in photothermal resources and it contributes to the accumulation of sugar in grapes which might lead to higher residual sugar and alcohol content in wines. Nei Mongol had the largest reducing sugar interquartile range (IQR) among all regions (Fig. S1). Although Hebei had the largest amount of samples, its reducing sugar IQR was the smallest. Additionally, the average reducing sugar of Hebei was the lowest, which is 81.2% lower than Nei Mongol. Among the basic chemical composition studied, only reducing sugar had significant differences among all seven regions ( $P < 0.05$ ), which partly represented the regional characteristics. As for glucose and fructose, the two important monosaccharides in grapes and wines (Berthels et al., 2008), Gansu and Nei Mongol were the two highest regions of glucose contents, which were also high in reducing sugar content. Similar to the

reducing sugar content, Nei Mongol was also the region with the largest IQR in glucose and fructose contents. Although the reducing sugar of Xinjiang was significantly higher than Hebei, there was no significant difference in glucose between the two regions. Nei Mongol had the highest fructose content (2.59 g/L).

The total acid could directly affect the overall sensory characteristics of wines (Mato et al., 2005). Beijing wines had the highest total acid (2.10 g/L), followed by Ningxia (2.06 g/L). There was no significant difference in the total acid content between Hebei and Xinjiang samples. The range in total acid content of Xinjiang samples was large. Shandong and Gansu had the lowest total acid contents with no significant differences between them. The variability and range of total acid content for Shandong and Xinjiang were large. This indicated the characteristics of samples from the two regions.

Sulfur dioxide is the most common wine additive and acts as an antibacterial and antioxidant agent. Beijing samples had the highest contents of total sulfite (146.50 mg/L) and free sulfite (14.33 mg/L) among the seven regions. The total sulfite of Hebei (80.88 mg/L) was second to Beijing, with the largest range (Fig. S1). All regions had significant differences in total sulfite content, except for Gansu and Xinjiang. Beijing wines had the highest free sulfite (14.33 mg/L) followed by Nei Mongol (13.02 mg/L). Free sulfite was not detected in Gansu wines, which may be due to the relatively old vintage of samples collected. The sulfur dioxide content in wine was more likely to change over time (Godden et al., 2015). Except for Gansu, Hebei had the lowest sulfur content (6.55 mg/L).

Ammonia nitrogen in wines could be utilized by microorganisms including *Saccharomyces cerevisiae* and affected wine flavor (Ivit & Kemp, 2018). The ammonia nitrogen contents of Ningxia (43.95 mg/L) and Nei Mongol (43.44 mg/L) were significantly higher than the other regions. Also, the variability of the ammonia nitrogen contents of these two regions were large. The ammonia nitrogen contents of Hebei and Beijing wines had no significant difference. Gansu wines had the lowest ammonia nitrogen (24.98 mg/L).

The total polyphenol contents of the seven regions were significantly different ( $P < 0.05$ ). Nei Mongol (2371.13 mg/L) was the region had the highest total polyphenol average and Gansu (1585.50 mg/L) wines had the lowest average (Fig. S1). Nei Mongol and Xinjiang, located in the arid and semi-arid region of China, had higher total phenol average than most regions. This may be due to the accumulation of phenols in fruits which could be greatly affected by the climate. Moreover, the water status of the grapevine was indicated as a vital parameter of the phenol content (Gutierrez-Escobar et al., 2021). Gansu have a similar geographical location as Nei Mongol and Xinjiang. However, Gansu wines had the lowest total phenol. It was similar to the results of monomeric organic acids and anthocyanins described later. Particularly, Hebei, located in eastern China, was second to the highest Nei Mongol in the total phenolic average. Factors except climate and water status could play important roles in affecting the phenolic content (Fischer et al., 1999). In conclusion, total phenol contents of wines from arid and semi-arid regions in northwest China were generally higher than the other regions in this study.

Significant differences were observed between the total anthocyanin contents of seven regions ( $P < 0.05$ ). The highest content was found in Ningxia wines (348.98 mg/L), which was about 4.2 times higher than Gansu (Fig. S1). Gutierrez-Escobar et al. (2021) observed that climate had the greatest influence on the grape composition, followed by soil and cultivar. The sufficient photothermal resources of Ningxia could contribute to the accumulation of anthocyanin in grapes, potentially explaining the highest total anthocyanin of Ningxia wines. Nei Mongol and Xinjiang, the other two geographically similar regions, were second to Ningxia. However, Gansu had the lowest total anthocyanin even though located in the arid and semi-arid region. In this study, Gansu samples were surprisingly characterized by low anthocyanins, acids and phenols, which may be due to the relatively older vintage of the Gansu samples compared to the other regions. The content of various types of

anthocyanins in the fruit was affected by many factors during processing (Lv et al., 2024). It suggested a severe aging problem (Cheng et al., 2023; He et al., 2012) but could also result from the limitation of the sample collected. In general, wines from the arid and semi-arid regions with sufficient light and heat had relatively higher total anthocyanin as expected (Gutierrez-Escobar et al., 2021).

### 3.1.2. Regional differentiation based on basic wine composition

The principal component analysis (PCA) based on the above compositions demonstrated the clustering of the same region samples and those of different regions could be clearly distinguished (Fig. S1). The glucose largely contributed to PC1, while the total acid and ammonia nitrogen largely contributed to PC2. The seven regions could be roughly clustered into two groups: Hebei, Xinjiang, Ningxia and Nei Mongol the first group, and the remaining three regions the second group, as the hierarchical cluster analysis (HCA) showed. The successful differentiation suggested the possibility that basic chemical composition could be used as differentiation criterion for Chinese 'Marselan' wines from different regions.

## 3.2. Organic acids

### 3.2.1. Organic acid characteristics of the seven regions

Organic acids were considered to have a close relation to the total acidity of wines (Chidi et al., 2018). Cultivar, climate, vineyard management and oenological strategy were indicated as important factors that could affect the acidity of wines (Ferreira & Mendes-Faia, 2020). The average of shikimic acid (0.06 g/L) was the lowest compared to the eight organic acids studied (Fig. 2a). Quinic acid, malic acid, and succinic acid were 51, 49, and 40 times higher than shikimic acid, respectively. Oxalic acid, synthesized using ascorbic acid (Asc) as a precursor, is a natural organic acid in grapes. Ningxia wines had the highest oxalic acid average (0.29 g/L), and was about 87% higher than the lowest Gansu. Tartaric acid is a natural organic acid found in grapes and is commonly used to adjust the acidity of low-acid wines (Boulton et al., 1996). Shandong (1.21 g/L) and Xinjiang (1.20 g/L) had the highest tartaric acid average with no significant differences and Gansu had the lowest (0.70 g/L). Malic acid is also a grape-derived organic acid. Unlike tartaric acid, malic acid is susceptible to degradation by microorganisms in wines. Hebei wines had the highest malic acid average than the other regions with significant differences, which was about 2.4 times higher than the lowest Beijing. Malic acid was the only one had significant differences in all the seven regions among the organic acids studied.

### 3.2.2. Regional differentiation based on organic acid characteristics

The HCA result based on the organic acids demonstrated three clusters of the seven regions (Fig. 2b). Hebei, Xinjiang, Ningxia and Nei Mongol were clustered into the first group and all of them are arid and semi-arid regions, except Hebei. Shandong and Gansu were clustered and Beijing formed the third group alone. Hence, it suggested that there was no obvious trend of regional aggregation based on the eight organic acids in commercial 'Marselan' wines. As shown in Fig. 2c, most of the Shandong and Hebei samples had higher organic acid content than other regions. The HCA results of the heatmap showed a scattered distribution of 50 samples with no obvious trend of regional clustering. A reliable OPLS-DA model was established based on the content of eight organic acids ( $R^2X = 0.801$ ,  $R^2Y = 0.865$ ,  $Q^2 = 0.788$ ). As Fig. 2d showed, the seven regions could be distinguished clearly. In the scatter plot, Hebei and Xinjiang were close in the direction of the first component, so did Ningxia and Nei Mongol. This result was consistent with the HCA results (Fig. 2b) suggested the similar characteristics of these close regions for organic acids. Gansu and Beijing were the most distinctive regions from the other regions. Beijing was distant from the other six regions in the direction of the first component. Tartaric acid, malic acid and shikimic acid were the key compounds (variable influence on projection (VIP) value  $> 1.1$ ). This demonstrated distinguishable organic acid profiles of

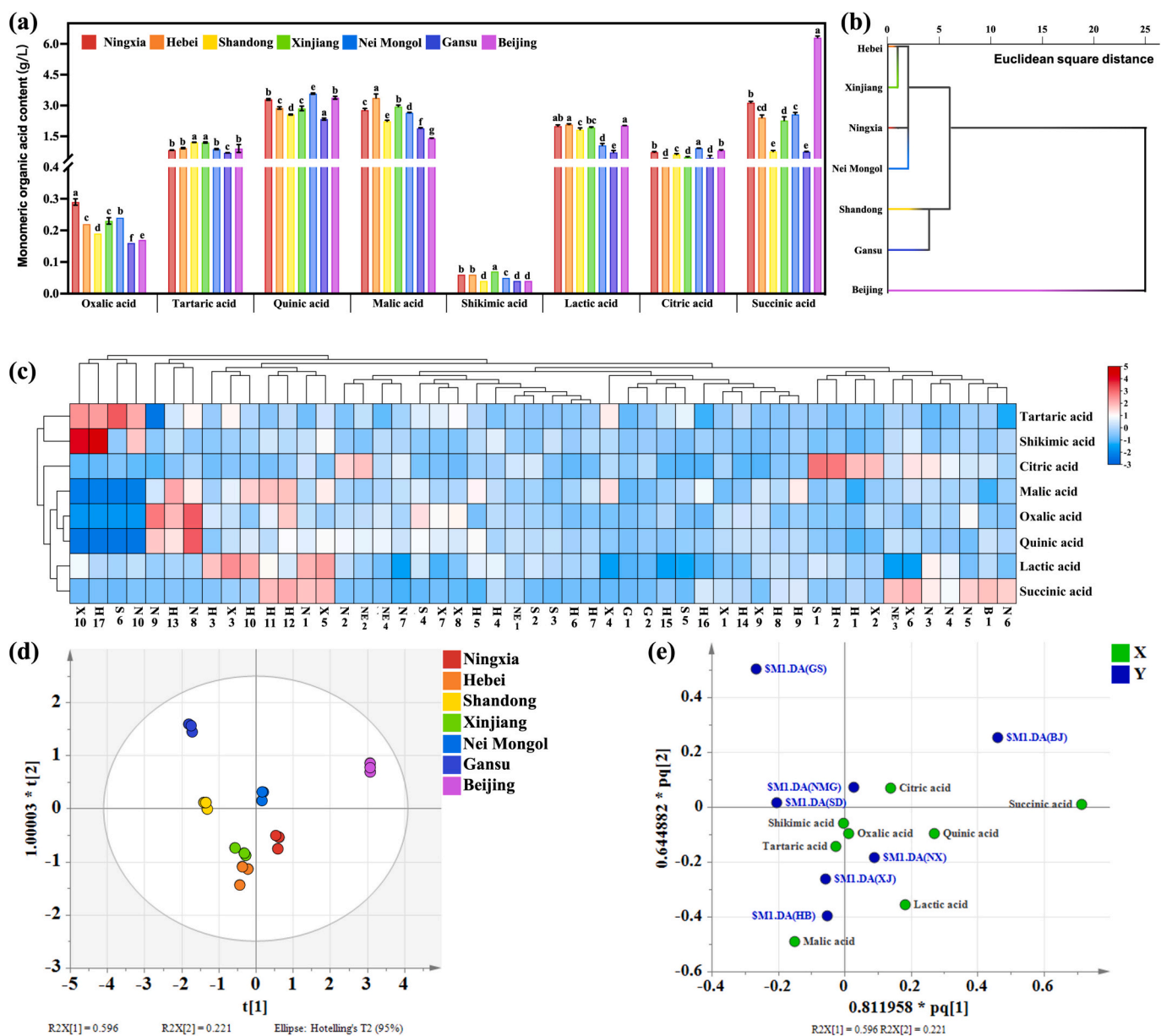


Fig. 2. Organic acid content (a, c); Hierarchical cluster analysis cluster plot (b) and OPLS-DA scatter plot (d) and loading plot (e) based on organic acid.

'Marselan' wines from seven Chinese regions and remained to be further studied.

### 3.3. Phenolic substances and antioxidant capacity

#### 3.3.1. Monomeric anthocyanin

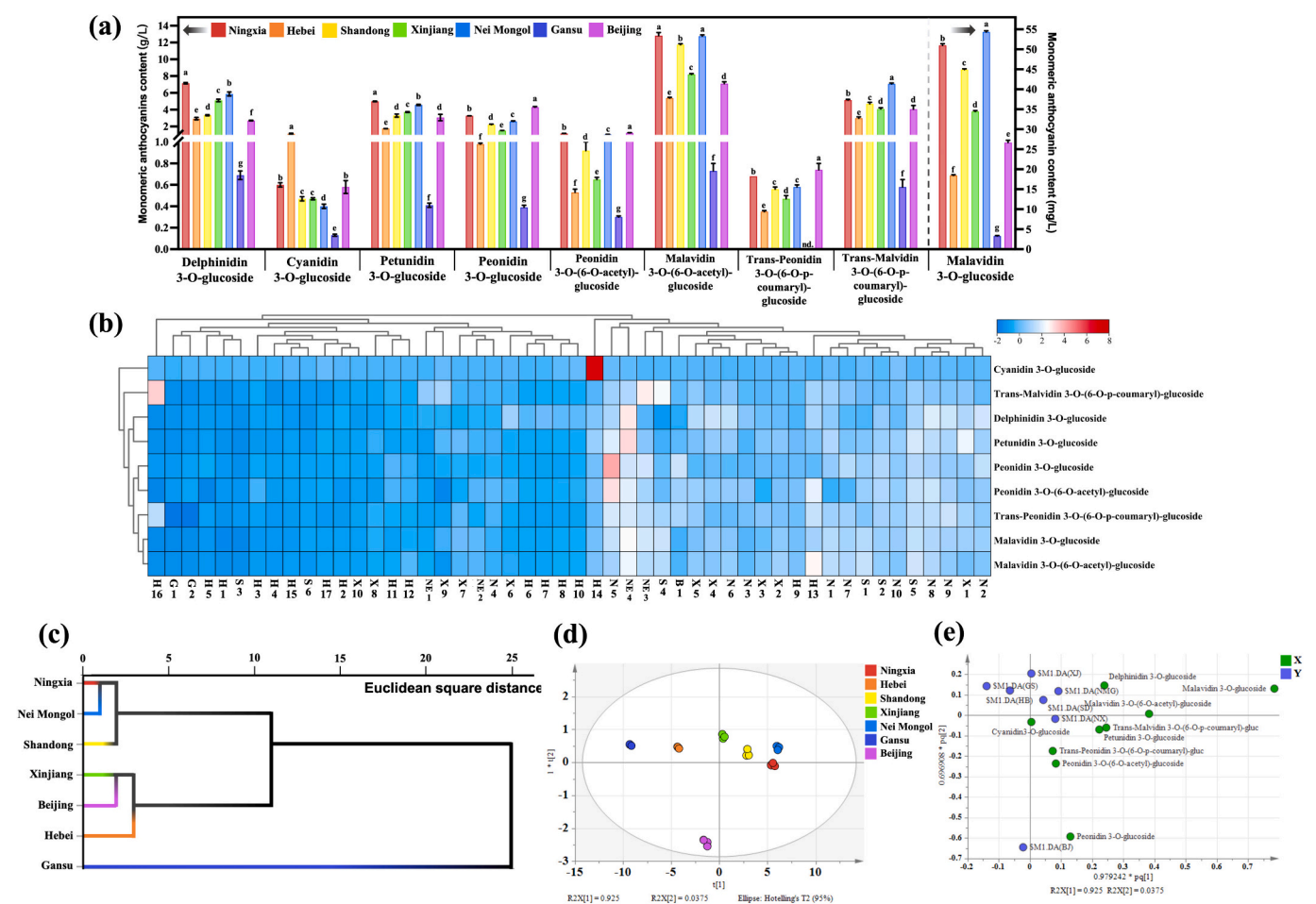
##### (1) Monomeric anthocyanin characteristics.

The content of malvidin-3-O-glucoside was the highest among the monomeric anthocyanins studied (average, 33.3 mg/L), 69 times that of *trans*-peonidin-3-O-(6-O-*p*-coumaryl)-glucoside (0.48 mg/L). The levels of nine monomeric anthocyanins in Gansu samples were significantly lower than other regions, which was similar to the result of total anthocyanin (Fig. 3a). *Trans*-peonidin-3-O-(6-O-*p*-coumaryl)-glucoside was not detected in Gansu samples. Ningxia wines had the highest content of delphinidin-3-O-glucoside, petunidin-3-O-glucoside and malvidin-3-O-(6-O-acetyl)-glucoside. Hebei demonstrated the highest level of cyanidin-3-O-glucoside among the seven regions. Nei Mongol was characterized by the highest contents of malvidin-3-O-glucoside, malvidin-3-O-(6-O-acetyl)-glucoside and *trans*-malvidin-3-O-(6-O-*p*-

coumaryl)-glucoside. Beijing surprisingly exhibited the highest levels of peonidin-3-O-glucoside, peonidin-3-O-(6-O-acetyl)-glucoside and *trans*-peonidin-3-O-(6-O-*p*-coumaryl)-glucoside despite its relatively low content of total anthocyanin.

##### (2) Regional differentiation based on monomeric anthocyanin.

The heatmap results of monomeric anthocyanin showed that Nei Mongol samples could be divided into two clusters by wineries (Fig. 3b). The wine samples from the same winery were relatively close and were belong to the same cluster. Gansu, Hebei, and Ningxia were also consistent with this phenomenon. Winemaking techniques of the same winery tends to be more consistent compared to different winery even from the same region which could be inflected in the composition of wines. Most of wine samples from Hebei and Ningxia were clustered by region, which could imply the regional characteristics of monomeric anthocyanin. Region and specific winemaking strategies could be important factors that affected the type and content of anthocyanins in 'Marselan' wines. The clustering trend of monomeric anthocyanin was stronger than the organic acids analyzed. Cyanidin-3-O-glucoside was distinct from the other eight monomeric anthocyanins and formed a



**Fig. 3.** Monomeric anthocyanin content (a-b); Hierarchical cluster analysis cluster plot (c) and OPLS-DA scatter plot (d) and loading plot (e) based on monomeric anthocyanin.

single cluster (Fig. 3b). The seven regions could be clustered into three groups as the HCA results showed (Fig. 3c). Ningxia, Nei Mongol and Shandong were clustered into the first group, Xinjiang, Beijing, and Hebei were clustered into the second, and Gansu formed the third group alone. Except for Gansu and Xinjiang, all the northwest regions were clustered into the same group.

The reliable OPLS-DA model ( $R^2X = 0.997$ ,  $R^2Y = 0.959$ ,  $Q^2 = 0.926$ ) was established based on the monomeric anthocyanin and each of the seven region was distinguished clearly (Fig. 3d). The seven regions could be divided into two groups by the second component. The first group included Beijing and the second group included the other six regions. The two northwestern regions (Ningxia and Nei Mongol) were close according to the first component. Delphinidin-3-O-glucoside, malvidin-3-O-glucoside, petunidin-3-O-glucoside, malvidin-3-O-6-O-acetyl-glucoside and *trans*-malvidin-3-O-6-O-p-coumaryl-glucoside were the key monomeric anthocyanins played a key role in the establishment of the model according to VIP value (Table S3).

### 3.3.2. Non-anthocyanin monomeric phenolic acid

#### (1) Non-anthocyanin monomeric phenolic acid characteristics.

Nei Mongol had a significantly higher contents of 17 monomeric phenolic acids than other regions (Fig. 4a). However, protocatechuic acid (Comp 24, 1.88 mg/L) and 3-hydroxycinnamic acid (Comp 20, 1.18 mg/L) of Nei Mongol wines were significantly lower than the other regions except Beijing, which had no significant difference with Nei Mongol. Comp 24 and Comp 20 were 78.5% and 67.5% lower than Gansu and Xinjiang, respectively. Similarly, there was no significant

difference in methyl vanillate (Comp 29) between Nei Mongol, Shandong, Xinjiang, and Ningxia. These regions significantly lower in Comp 29 than the other three regions. In general, Nei Mongol had the highest contents of total phenol and most monomeric phenolic acids among the seven regions. Methyl 3,4-dihydroxybenzoate (Comp 28) and vanillic acid diethyl ester (Comp 31) contents of Ningxia wines were the highest among all regions. Comp 28 was 5.5 times higher than Gansu with the lowest content while Comp 31 was not found in Gansu and Beijing. Hebei wines had the highest content of 2, 5-dihydroxybenzoic acid (Comp 25) and ethyl 4-hydroxybenzoate (Comp 30) among all regions. The *trans*-monoferuloyl tartrate (Comp 15) content of Shandong was the highest. Xinjiang wines had the highest content of (–)-epigallocatechin gallate (Comp 3), 3-hydroxycinnamic acid (Comp 20), and sinapic acid (Comp 21). Unlike grape, wine is a result of a series of processes. Therefore, the phenolic compounds in the wine were not only affected by the natural conditions but also by some artificial factors such as oenological strategies (Godden et al., 2015).

#### (2) Regional differentiation based on non-anthocyanin monomeric phenolic acid characteristics.

The results of heatmap clustering indicated that all Nei Mongol wines of the same vintage were clustered into one group (Fig. 4b). All regions were divided into three groups according to the clustering results. Hebei, Shandong, and Ningxia were belong to the first group; Xinjiang, Gansu, and Beijing formed the second; and Nei Mongol formed the third group alone. As shown in the HCA results, all regions could be divided into two categories, among which Nei Mongol region can be distinguished separately (Fig. 4c).

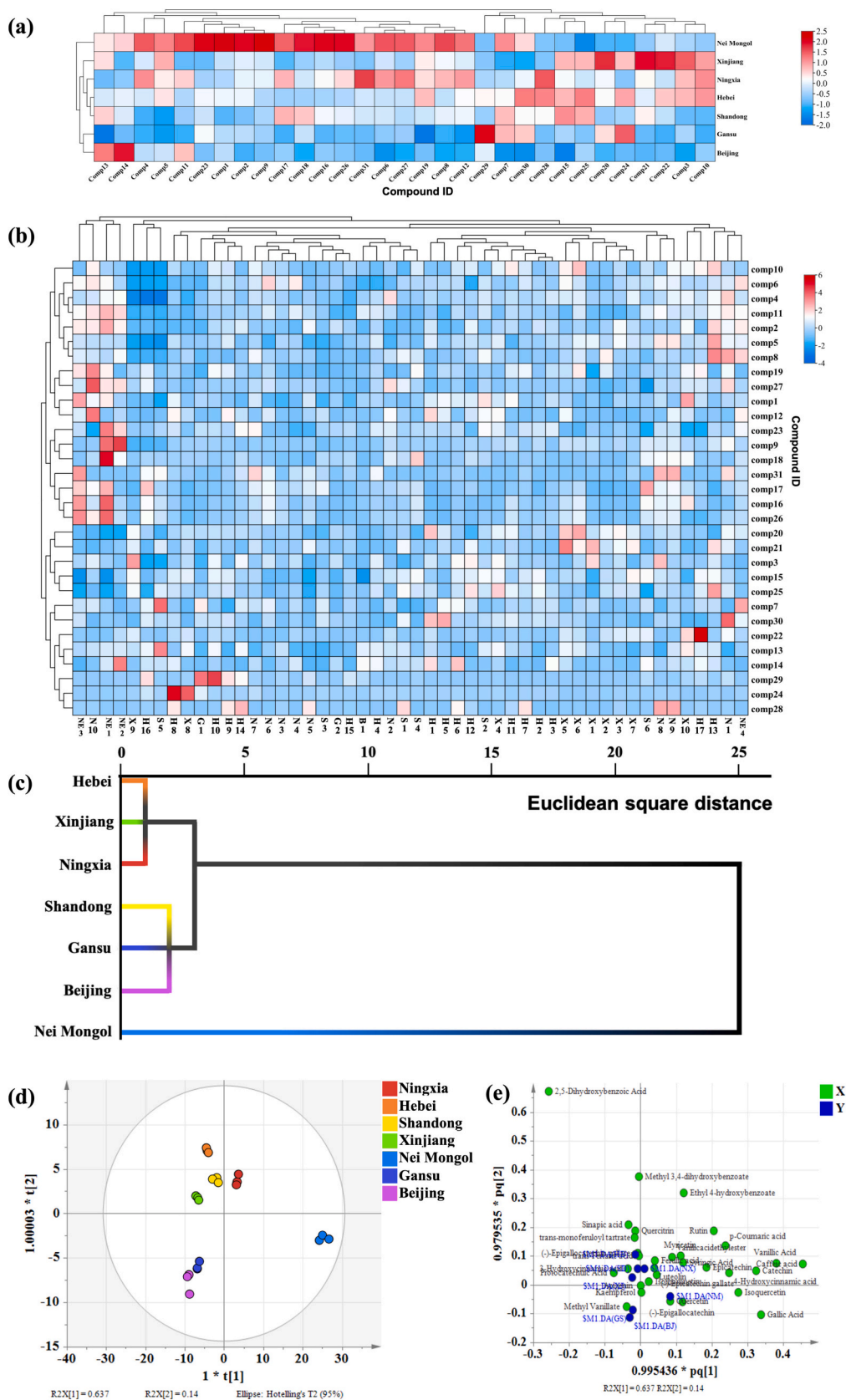


Fig. 4. Non-anthocyanin monomeric phenolic acid content (a-b); Hierarchical cluster analysis cluster plot (c) and OPLS-DA scatter plot (d) and loading plot (e) based on non-anthocyanin monomeric phenolic acid. The compound codes in the figure were annotated in tables S2.

A reliable OPLS-DA model was established based on the contents of 31 monomeric phenolic acids ( $R^2X = 0.859$ ,  $R^2Y = 0.992$ ,  $Q^2 = 0.986$ ). The other regions except Gansu could be clearly distinguished in the scatter plot (Fig. 4d). It was concluded that Nei Mongol had distinctive phenolic characteristics from other regions (Fig. 4a, c) which was also reflected in the OPLS-DA model in Fig. 4d. The regions were divided into two categories in the direction of the first component. As shown in the scatter plot, Nei Mongol could be clearly distinguished and is far away from the other regions. It was slightly different from a previous study of young 'Marselan' wines, which indicated that it was difficult to differentiate regions from the OPLS-DA model based on the phenolic compounds (Lan, Wang, et al., 2022). Sinapic acid (Comp 21), *trans*-ferric acid (Comp 22), vanillic acid diethyl ester (Comp 31), and methyl vanillate (Comp 29) were critical in the establishment of the model (VIP value >1.5).

### 3.3.3. Antioxidant capacity

#### (1) Characteristics of antioxidant capacity.

The antioxidant capacities of DPPH, FRAP and CUPRAC of Nei Mongol were significantly higher than the other six regions ( $P < 0.05$ ; Fig. S2). The antioxidant capacities of DPPH for Beijing and Gansu samples were significantly lower than the other regions, which were only 57% and 56% of the highest Nei Mongol samples respectively. The FRAP antioxidant capacity of Beijing was the lowest and was 44.3% lower than Nei Mongol. The CUPRAC antioxidant capacity of Gansu wines (13.34 mM Trolox/L) was significantly lower than the other six regions. Overall, the results of regional antioxidant capacity characterized by the three methods showed a similar trend.

#### (2) Correlation analysis.

The correlation analysis was based on organic acids, anthocyanins, and non-anthocyanin phenolic acids and antioxidant capacity. There was no significant correlation between the organic acids studied and the antioxidant capacity was found, except for malic acid and shikimic acid. Malic acid showed a significant positive correlation with the antioxidant capacity of DPPH and shikimic acid had a significant positive correlation with CUPRAC. Petunidin-3-O-glucoside and malvidin-3-O-(6-O-acetyl)-glucoside demonstrated a significant positive correlation with DPPH respectively. There were significant positive correlations between delphinidin-3-O-glucoside and the antioxidant capacities of FRAP and DPPH. Moreover, total anthocyanin was positively correlated with all antioxidant parameters. Correlation clustering results showed that delphinidin-3-O-glucoside was distant from the other monomeric anthocyanins studied (Fig. S2). Anthocyanins were considered as natural antioxidants and indicated as the most effective safe and natural water-soluble free radical scavengers (Fernandes et al., 2014). It is reported that the antioxidant activities of them was related to their chemical structures (Qi et al., 2022).

The total phenol was positively correlated with the three antioxidant parameters with highly significant differences. Most of monomeric phenolic acids were significantly correlated with antioxidant capacity as expected. Foods with higher phenol contents tended to have relatively higher antioxidant capacity (Perez-Jimenez et al., 2015). Particularly, quercitrin, quercetin, (-)-epigallocatechin, epicatechin, (-)-epicatechin gallate, and catechin showed significant positive correlations with all antioxidant parameters. It suggested the six monomeric phenolic acids played important roles in the antioxidant capacity of 'Marselan' wines.

### 3.4. Volatile compounds

#### 3.4.1. Volatile compounds characteristics of the seven regions

Previous studies reported an amount of 64 and 65 volatile compounds in 'Marselan' commercial wines, respectively (Lan et al., 2022; Song et al., 2023). With a larger amount of wine samples and a wider range of wine regions, a total of 102 volatile compounds were detected (Table S5). Ester was the largest chemical category consisted of 47

esters, followed by alcohols (30), ketones (6), acids (6), volatile phenols (5), alkanes (5), and aldehydes (3). There were 91 kinds of volatile compounds determined in the samples of Hebei, followed by Xinjiang (90) and Ningxia (88), and the least was Beijing (53) (Fig. 5a). Forty compounds were common in the seven regions, including 25 esters and 11 alcohols. Hexanoic acid, *n*-decanoic acid, acetic acid, and  $\beta$ -damascenone were some important compounds found in all the seven regions. Particularly, hexanoic acid and  $\beta$ -damascenone were also compounds with OAVs  $\geq 1$ . It was worth noting that Ningxia (benzaldehyde, 3-furanmethanol), Hebei (4-*tert*-amylphenol, 3-ethyl-4-methylpentan-1-ol), Xinjiang (butyl butyrate), and Gansu (2-phenylethyl isovalerate) had specific compounds detected in these regions only. This suggested the typical regional characteristics of the seven regions in the volatile compound profiles. However, except for 3-furanmethanol, 3-ethyl-4-methylpentan-1-ol and 2-phenylethyl isovalerate (thresholds not found in the literature), the OAVs of all the above region-specific volatile compounds were under one.

Fig. 5c showed the sum of the contents of each chemical category of the seven regions, respectively. Ester was the largest chemical category in 'Marselan' samples, and Xinjiang wines had the highest ester content, while Shandong was the lowest. The alcohol content was second to ester, and the Ningxia wines had the highest alcohol content and the lowest was Beijing. Linalool and  $\alpha$ -terpineol were two important terpenoid volatile compounds detected in this study, which were considered to bring floral and fruity aroma attributes (Table S5). The content of linalool varied greatly among regions. Linalool was not detected in Beijing, while the average of Nei Mongol was as high as 209.21  $\mu\text{g/L}$  (Table S6). The average of  $\alpha$ -terpineol was the highest in the Hebei wines (26.68  $\mu\text{g/L}$ ). Xinjiang (11.22  $\mu\text{g/L}$ ) had the lowest content of  $\alpha$ -terpineol among the six regions except Beijing (not detected). Some studies suggested that linalool and  $\alpha$ -terpineol were the two compounds with significant differences between the 'Marselan' wine and 'Cabernet Sauvignon' wine. The content of terpenes in the 'Marselan' wine seemed to be genetically related to its parent Grenache (Song et al., 2023). Song et al. also suggested that ethyl hexadecanoate was an important volatile compound that differed in 'Cabernet Sauvignon' and 'Marselan' wines. In this study, ethyl hexadecanoate was relatively high and its OAV  $\geq 1$  in all seven regions suggested its importance in the aroma profiles of 'Marselan' wines from the seven regions. The seven regions could be clustered into two groups: one group was Gansu and Beijing, and the remaining five regions formed another group (Fig. 5b). In addition, Ningxia and Hebei were close to each other, so did Xinjiang and Shandong.

#### 3.4.2. OAV analysis

Compounds (OAVs  $\geq 1$ ) were considered to have an important influence on aroma perception (Guth, 1997). A total of 26 compounds (OAVs  $\geq 1$ ) were found (Table S7). Among them,  $\beta$ -damascenone, ionone, ethyl decanoate, 2,2,4-trimethyl-1,3-pentanediol diisobutyrate, ethyl isovalerate, ethyl octanoate, methyl decanoate, and ethyl hexanoate were the key compounds detected in 'Marselan' wines with relatively high OAVs (average, 10.2–1511.7); similar results were reported in several studies on 'Marselan' wines (Lu et al., 2020; Song et al., 2023). The above compounds mentioned were considered to be associated with flowers, fruits, and plant aroma attributes, except octanoic acid generally conferred fat-related odors (Table S5). Particularly,  $\beta$ -damascenone had the highest OAVs in the seven regions consistent with the results of Lan et al. (2022). Therefore,  $\beta$ -damascenone was considered to be an important compound affecting the aroma characteristics of Chinese 'Marselan' wines.  $\beta$ -damascenone is a C-13 nor-isoprenoid compound that was reported to bring sweet, exotic flower, and stewed odor characteristics (Noguerol-Pato et al., 2015).

As shown in Fig. 5d and Table S7, "Floral", "Sweet" and "Fruity" were the most important aromatic series of 'Marselan' wines and Ningxia wines had the highest intensities (measured by OAVs) of the three aromatic series. Conversely, The aromatic series "Roasty"



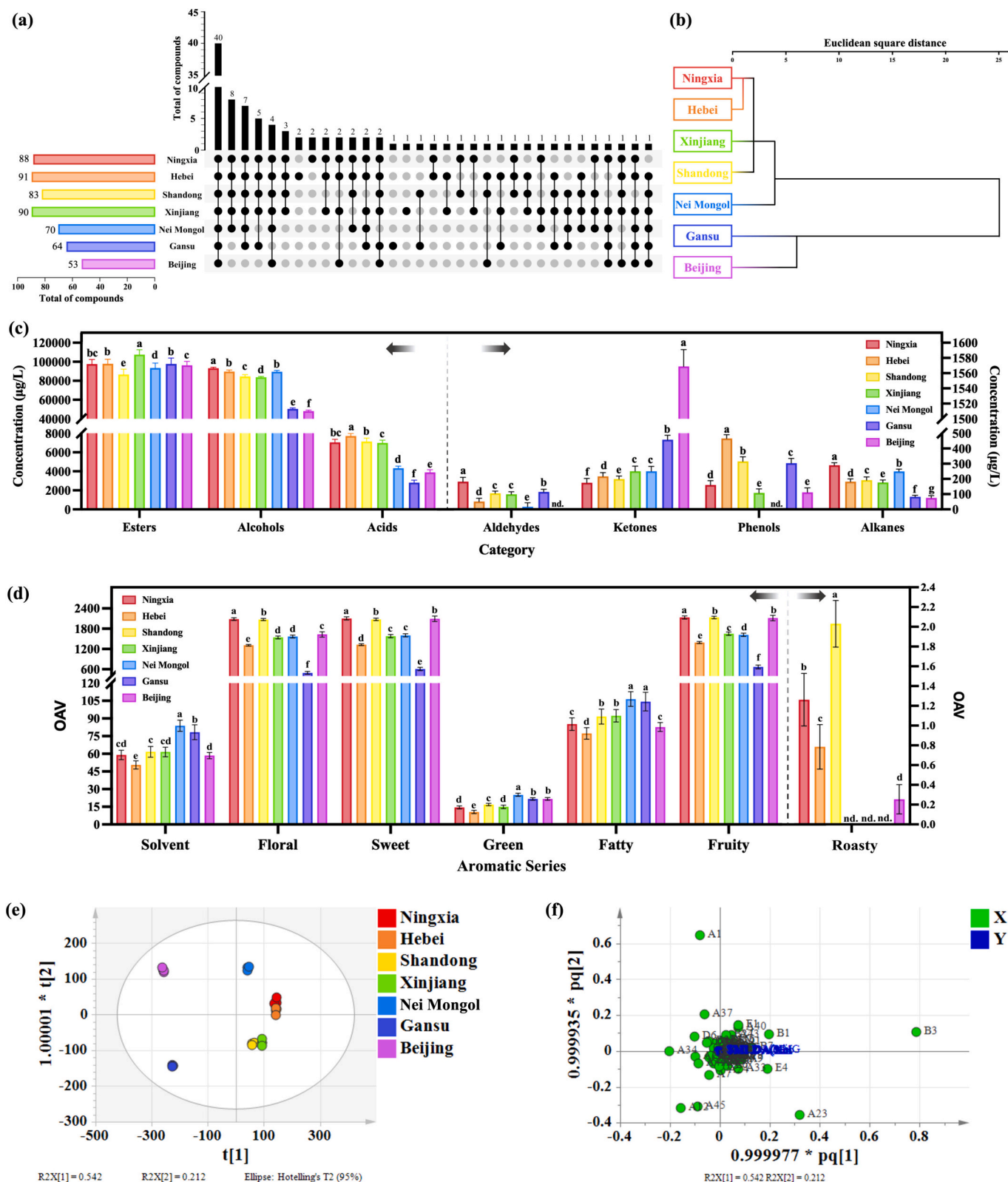


Fig. 5. Venn diagram of volatile compounds (a); Hierarchical cluster analysis cluster plot (b); The contents of different chemical categories (c); Aromatic series intensities (d); OPLS-DA Score plot (e) and load plot (f) based on volatile compounds.

exhibited the lowest intensity among all the aromatic series and was plotted separately on the right Y-axis. The intensity of “Roasty” was considerably lower than other aromatic series. Shandong demonstrated the highest “Roasty” intensity whereas Xinjiang, Nei Mongol, and Gansu exhibited the intensity of 0 in “Roasty” aroma series. The intensity of the

three aromatic series (“Solvent”, “Green”, and “Fatty”) of Nei Mongol was highest in the seven regions. It suggested that the ‘Marselan’ aroma characteristics were mainly characterized by fresh floral and fruity notes.

### 3.4.3. Regional differentiation based on volatile compounds

A reliable OPLS-DA model was established based on the content of volatile compounds in 'Marselan' wines ( $R^2X = 0.599$ ,  $R^2Y = 0.999$ ,  $Q^2 = 0.997$ ). Nei Mongol, Gansu and Beijing could be clearly distinguished (Fig. 5e). Ningxia and Hebei were relatively close to each other and were difficult to distinguish consequently, so did Shandong and Xinjiang. It was also reflected in HCA result (Fig. 5b). As shown in the scatter plot (Fig. 5e), Gansu and Beijing were distant from other regions.

### 3.5. Intelligent sensory features

The sensory evaluation of wine is usually carried out by a combination of methods. Human panel evaluation was considered as time-consuming and complex, with strong subjectivity and low repeatability (Shu et al., 2022). Moreover, the sensory fatigue problem of evaluators limits the amount of samples in the experiment. On the contrary, intelligent sensory evaluations, such as e-nose and e-eye, are more objective and can analyze a large number of samples in a limited time.

#### 3.5.1. Color analysis

There is a close relationship between the spectrum data of the wine color and the actual color perception. The lowest  $L^*$  value was found in the sample N2 (from Ningxia, vintage 2019) and the sample S3 (from

Shandong, vintage 2014) had highest  $L^*$  value. It was reflected in the color simulation diagram (Fig. 6a). There were discernible differences in the color simulation based on color parameters of the wine samples (Table S8). The sample N10 and N8 had the largest difference in  $\Delta E^*$  values. The  $L^*$  value of Ningxia was significantly lower than other regions. Ningxia exhibited the highest  $a^*$  and  $C^*$  values and it was reflected a dark red tone compared with other regions in color simulation (Table S9). It was evident the  $h^\circ$  values were mainly distributed in the range of  $20^\circ$ – $40^\circ$  (Fig. 6f). The Shandong exhibited the lowest  $b^*$  value and  $h^\circ$  values suggested that the colors of Shandong wines were more inclined to purplish red. The  $L^*$  value of Gansu was the highest, followed by Beijing and the  $a^*$  and  $C^*$  values of them were relatively low. Consequently, the color simulation diagram of the two regions showed relatively bright colors and weak red tones. Distinguishable color characteristics of different regions could be recognized.

The vintage 2014 had the highest  $L^*$  value, followed by the 2013 and 2015 (Table S10). Particularly, the  $L^*$  value was found to increase with the aging of the vintage. The trends of  $a^*$  value and  $L^*$  value were mostly opposite. As the vintage grew older, the  $a^*$  values showed a decreasing tendency. The vintage 2014 had the lowest  $a^*$  value, followed by the 2013 and the vintage 2020 had the highest  $a^*$  value. Moreover, vintage 2015 had the second highest  $a^*$  value despite the relatively older vintage compared to most samples. The  $b^*$  values and  $L^*$  values had similar tendencies that values increased with vintages. The vintage 2013 had

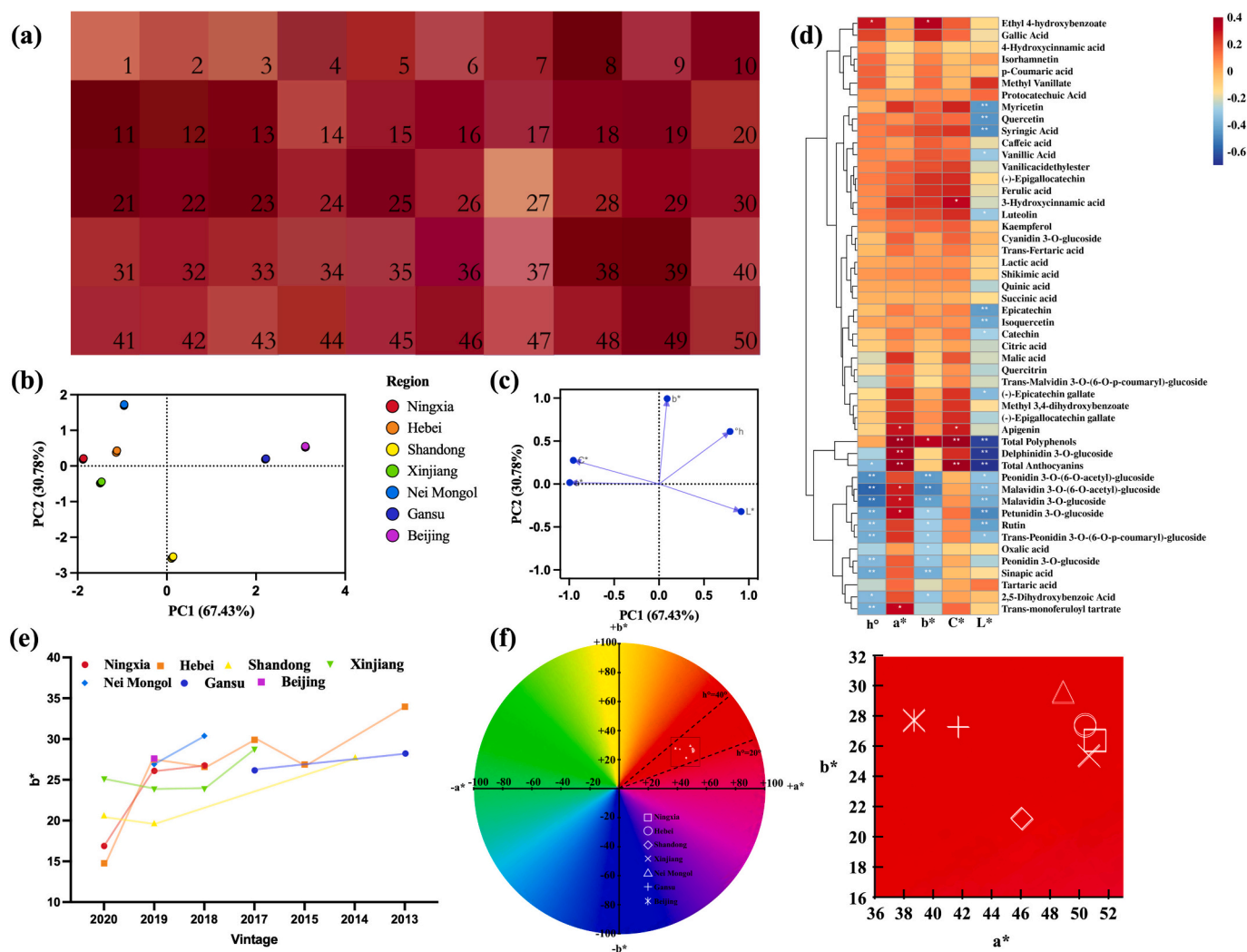


Fig. 6. Color simulation diagram (a); PCA score plot (b) and loading plot (c) based on color parameters; Pearson correlation analysis heatmap based on color parameters (d);  $b^*$  values of different vintages (e); Color characterization of different regions and enlarged partial view (f).

the highest  $b^*$  value while the 2020 vintage had the lowest. As shown in Fig. 6e, ‘Marselan’ wines of different regions showed an general increase of  $b^*$  values with vintage. Except for the above three color parameters, the relations between other color parameters and the vintage were not obvious.

It was obvious that Ningxia, Hebei, Xinjiang, and Nei Mongol regions were close on the figure, so did Gansu and Beijing (Fig. 6f). However, the Shandong region was relatively isolated compared with the above two groups. The PCA score plot based on the color parameters of the regions could well distinguish different regions (Fig. 6b). As shown in the loading plot,  $a^*$  and  $L^*$  were the largest contribution to PC1, so did  $b^*$  to PC2 (Fig. 6c). According to PC1, regions could be divided into two groups. The first group was consisted of Ningxia, Xinjiang, Hebei, and Nei Mongol mainly distributed in the negative PC1 direction. Another group was consisted of Shandong, Gansu and Beijing mainly distributed in the positive part of PC1. According to PC2, Shandong was distinguished from the other regions.

Fig. 6d showed the Pearson correlation of color parameters ( $h^\circ$ ,  $a^*$ ,  $b^*$ ,  $C^*$ , and  $L^*$ ) with the contents of organic acids, monomeric anthocyanins, monomeric phenolic acids, total phenol, and total anthocyanin. Total phenol ( $R = 0.57$ ) and total anthocyanin ( $R = 0.53$ ) were positively correlated with the  $a^*$  and  $C^*$  values. Additionally, both of them have a highly significant negative correlation with the  $L^*$  value. In this study, the total anthocyanin had the greatest negative correlation coefficient among all negative correlations, followed by delphinidin-3-O-glucoside. The total phenol showed a positive correlation with  $b^*$  value and the total anthocyanin showed a negatively correlation with  $h^\circ$  values.

Only oxalic acid showed a significant negative correlation with  $b^*$  value and other organic acids studied were not found significant correlation with the color parameters. However, oxalic acid and tartaric acid were clustered into one group, whereas other organic acids were clustered into another according to the correlation heatmap clustering (Fig. 6d). Cyanidin-3-O-glucoside and *trans*-malvidin-3-O-(6-O-*p*-coumaryl)-glucoside had no significant correlation with any color parameters. Therefore, the two monomeric anthocyanins were grouped into one group, while the other seven anthocyanins were grouped into another. Except for the above two anthocyanins, the remaining seven monomeric anthocyanins showed a negative correlation with  $h^\circ$ ,  $b^*$ , and  $L^*$  values, although the correlation of peonidin-3-O-glucoside and  $L^*$  was not significant. It suggested a relation between the contents of these seven monomeric anthocyanins and purplish red color tone in ‘Marselan’ wines. Most of the non-anthocyanin monomeric phenolic acids showed negative correlations with the color parameters, especially with the  $L^*$  values. However, a few monomeric phenolic acids showed a positive correlation with some color parameters, such as ethyl 4-hydroxybenzoate with  $h^\circ$  and  $b^*$ , 3-hydroxybenzoic acid with  $C^*$ , apigenin with  $a^*$  and  $C^*$ , and *trans*-monoferuloyl tartrate with  $a^*$ .

### 3.5.2. E-nose

E-nose was used to analyze the overall aroma profile of ‘Marselan’ wine. Sensor S2 had the highest response value to the wines (Fig. S3). Sample N10 (2019) from Ningxia had the highest S2 response value and Hebei was the highest region. Meanwhile, sensor S2 showed the largest variance in samples. The response value of the highest sample was 3.45 times that of the lowest sample. Hebei was the highest region, and the response value was 2.35 times that of the lowest Beijing. Sensor S9 had the smallest variance in the response value since the highest value was only 1.01 times its lowest value. Samples had distinguishable different response profiles. Sample N10 had the lowest S3 and S5 response values but it had the highest S2 and S6 response values. Regions had different response features. Nei Mongol had the lowest S3 and S5 response values but was the highest in S7 and S9 sensors. Shandong had the highest S4 and S6 response values among the seven regions. Beijing was characterized by generally low response values.

All regions could be clustered into three groups based on the

response values (Fig. S3). The first group was consisted of Shandong, Ningxia, and Hebei. Nei Mongol and Xinjiang formed the second group. The third group was consisted of Gansu and Beijing. As shown in Fig. S3, the e-nose response profiles of wine samples were mostly similar. The response values of different sensors varied distinguishably. The sensor S6, S7, and S8 responses were similar. Regions had distinguishable different response profiles. The profile of Beijing was most distinguished from other regions mainly characterized by the lowest response values of sensors S7, S6, S8 and S2. Nei Mongol was distinguished by the highest response value of sensor S7. Hebei was characterized by sensor S2.

Fig. S3 showed the score plot of linear discriminant analysis (LDA) based on e-nose response values. The samples of the same area were grouped and regions were distinguished. Shandong, Xinjiang, and Hebei were close in the first quadrant that was the positive direction of LD1 and LD2. Gansu and Beijing were close in the second quadrant. Nei Mongol was distributed in the fourth quadrant separately. Gansu and Beijing could be distinguished from other regions in the negative direction of LD1. Nei Mongol could be distinguished in the negative direction of LD2.

## 4. Conclusions

Commercial ‘Marselan’ wines from seven Chinese regions were successfully characterized. Nei Mongol had the highest contents of total reducing sugar, fructose, ammonia nitrogen, total phenol, quinic acid and 18 kinds of monomeric phenolic acid among the seven regions.  $\beta$ -Damascenone had the highest OAV indicated as an important influence on aroma characteristics of ‘Marselan’ wine. ‘Floral’, ‘Sweet’ and ‘Fruity’ were the strongest aromatic series. As for color characteristics, the brightness ( $L^*$ ) of the samples in Ningxia was lowest in all regions, whereas the  $a^*$  and  $C^*$  values were the highest. It was indicated that the Ningxia wines were characterized by dark-red color tone. E-nose results showed that the aroma profiles of wines studied were mostly similar but there were differences between regions. The PCA models based on physicochemical and color parameters, the LDA model based on response values of e-nose, and the OPLS-DA models based on the monomeric organic acids and monomeric anthocyanins could clearly distinguish the regions. The OPLS-DA model based on the volatile compounds had the worst differentiation effect among all the models. Currently, ‘Marselan’ is still developing fast in China. As the amount of samples collected and detection conditions are limited, the chemical and sensory characteristics of ‘Marselan’ remain to be explored. However, this comprehensive data of ‘Marselan’ wines from different regions implies the possibility of geographical traceability of Chinese ‘Marselan’ wines and the development of regional featured quality.

## CRediT authorship contribution statement

**Yue Wang:** Writing – original draft, Software, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Caihong Li:** Writing – original draft, Methodology, Investigation, Conceptualization. **Qian Ge:** Writing – review & editing, Investigation, Conceptualization. **Xingsan Huo:** Investigation, Methodology, Resources. **Tingting Ma:** Writing – review & editing, Investigation, Conceptualization. **Yulin Fang:** Writing – review & editing, Resources, Conceptualization. **Xiangyu Sun:** Writing – review & editing, Visualization, Resources, Funding acquisition, Conceptualization.

## Declaration of competing interest

The authors declare that there are no conflicts of interest.

## 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.2024.101606>.

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