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Characterization of the Berry Quality Traits and Metabolites of 'Beimei' Interspecific Hybrid Wine Grapes during Berry Development and Winemaking

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Abstract: The development of grape varieties with cold resistance can be an advantage for the wine industry. 'Beimei', an interspecific hybrid wine grape variety with cold resistance and pleasant rose aroma, is now extensively cultivated in China. In the present study, the berry quality traits and metabolites of 'Beimei' and other "Bei" varieties were characterized. The sugar (234 g/L–391 g/L) and acid (6.2–8.3 g/L) contents of 'Beimei' and four additional "Bei" varieties, i.e., 'Beihong', 'Beixi', 'Beixin', and 'Xinbeichun' berries were smilar to that of traditional *Vitis vinifera* varieties. 'Beimei' grapes has the highest volatile compounds composition, especially the content of rose aroma compounds (2-phenyl-ethyl alcohol and neryl alcohol), which was significantly higher than that of the other "Bei" wine grape varieties. After fermentation, 'Beimei' wines showed improved quality, with a high resveratrol content (18 mg/L) compared to traditional Eurasian wines. In addition, the high content of main rose aroma compounds (acetic acid 2-phenylethyl acetate, phenylethyl alcohol, neryl alcohol and beta-damascenone) contribute to a pleasant rose aroma in 'Beimei' wines. In summary, these results indicate that 'Beimei' grapes could be used as a winemaking grape variety considering global climate changes.

Keywords: 'Beimei'; interspecific hybridization; quality traits; winemaking

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1. Introduction

Grape is one of the most economically valuable fruit crops in the world. Current studies on grape composition mostly focus on traditional grape cultivars (*V. vinifera*) and cover aspects, such as soil type [1], light quality penetration [2,3], environment [4,5], terroir, vintage effect and viticultural practices, as well as trellis systems [6], leaf layer or canopy density [7,8], and irrigation [9–11]. Sugar, acid [12–18], phenolic [19–28], aroma [29], resveratrol [30,31], as well as other grape composition characteristics were characterized.

With global climate change, grape varieties with high- resistance to the harsh cultivation environment are needed, Traditional Eurasian grapes are difficult to adapt to climate change. Therefore, currently, most breeders are inclining their studies towards using interspecific hybridization breeding to create high-resistant grape varieties. For example, Hungarian breeders have used interspecific hybridization breeding to produce grape varieties that can tolerate winter frosts and low precipitation that occur every three years [32]. The interspecific hybrid grape varieties (*V. vinifera* × North American *vitis* species) created by Canadian breeders are extensively cultivated for wine production in Canada and are of economic importance in northern areas [33]. American breeders bred Norton grapes using a cross of *V. Lavrusca* from *V. Aestivalis*, which can adapt to disease infestation and low daytime temperatures [34]. Cold and drought are the biggest challenges faced in the wine regions of China. In China, traditional wine vines (*V. vinifera*) need to be buried with soil in the winter to survive cold winters which result in high production costs. China's wine industry needs to focus on grape varieties with high stress resistance. 'Beimei' and other "Bei" wine grape varieties, such as 'Beihong', 'Beixi', 'Beixin', and 'Xinbeichun', have high-stress resistance and are good quality, selected through interspecific hybridization of 'Muscat humberg' *(V. vinifera*) × Shanputao (*V. amurensis*)), bred by the Institute of Botany, Chinese Academy of Sciences, which can withstand temperatures of −23 °C ('Beimei') and −24 °C ('Beihong') [35] and can survive the winter safely without covering the soil in the main wine regions, such as Ningxia, Beijing, and Hebei in China. For example, the resveratrol content of 'Beihong' berry skin is 4–9 times higher than that of *V. vinifera* varieties [31]. At present, 'Beimei' and 'Beihong' have been cultivated in more than 20 provinces in China and are in the process of rapid popularization. However, there is no systematic research on 'Beimei' and other "Bei" wine grape varieties and wine at present.

In this study, changes of berry quality traits and metabolites during 'Beimei' berry development and winemaking stages were analyzed. These results will provide theoretical and technical support for the promotion and vinification of 'Beimei' and other "Bei" wine grape varieties.

2. Materials and Methods

2.1. Plants and Growth Conditions

'Beimei', 'Beihong', 'Beixi', 'Beixin', and 'Xinbeichun' wine grape varieties used in developmental experiment were sourced from the grape germplasm resources nursery at the Institute of Botany, Chinese Academy of Sciences in Beijing. All the vines were own-rooted and of the same age. The vines, trained to cordons, were spaced 1.5 m apart within the row and 2.5 m apart between rows, with north–south row orientation. The vineyard is characterized with stony loam, has 2600–2700 h of annual sunshine, 3400–3800 °C of active accumulated temperature, and 480 mm of average annual rainfall (most of rainfall from June to August). Similar parameters, such as soil management, fertilization, irrigation, pruning, and disease control, were maintained in the vineyard. About 35–45 berries from different positions within a cluster were mixed and considered as one replicate, and three replicates sampled from three independent clusters of each variety were prepared at each developmental stage. One representative cluster of each variety was selected for dynamic investigation and photographing, which started on 19 June (S1) 2019. Then the development of grape berries was recorded every two weeks, and the corresponding samples were referred to as S2–S9. Notably, the veraison stage during the investigation was recorded separately as VE. Finally, 10 developmental stages (S1–S9 and VE) were designed for each variety. In 2021, the "Bei" anthesis was delayed with 5 days compared with 2019, and sample S1 was missed because of the COVID-19 pandemic. Therefore, the 2021 sampling began on 8 July, corresponding to the S2 (3 July 2019). In addition, due to above average (630 mm) rain in Beijing in 2021, the samples on 13 October (S9) were rotten and dried up and hence could not be used. Therefore, the samples in S9 in 2021 were also missing. The sample selection and sampling method in 2021 were the same as that in 2019. The samples were stored at −40 °C prior to testing. Sampling was done at different dates and developmental stages (Table 1). Photos of 'Beimei' which were taken during sampling time in 2019 were shown in Figure 1. Photos of other "Bei" grape varieties that were taken during sampling time in 2019 are shown in Appendix A Figures A1–A4. The 'Beimei' vines used in winemaking experiment were planted in the vineyard of Ningxia Xixia King Winery Co., Ltd., Ningxia, China, at the Eastern foot of Helan Mountains, Ningxia Hui Autonomous Region, China. The vineyard has the average annual rainfall 180 mm, and 3360 °C active accumulated temperature from April to September, and 180 d frost-free period. The grapevines were spaced 0.8 m apart within the row and 3.5 m apart between rows with a North-South row orientation, and managed by traditional management. Moreover, the grapevines are nine years old and their yield is 6000 kg/ha.

Table 1. Sampling date at different developmental stages of 'Beimei' and other wine grape varieties in 2019 and 2021.

Variety	Year	S1	S2	S3	VE	S4	S5	S6	S7	S8	S9
Beimei	2019	6.19	7.03	7.17	7.22	7.31	8.14	8.28	9.11	9.25	10.09
	2021	-	7.08	7.22	7.26	8.05	8.20	9.02	9.16	9.30	-
Beihong	2019	6.19	7.03	7.17	7.22	7.31	8.14	8.28	9.11	9.25	10.09
	2021	-	7.08	7.22	7.26	8.05	8.20	9.02	9.16	9.30	-
Beixi	2019	6.19	7.03	7.17	7.28	7.31	8.14	8.28	9.11	9.25	10.09
	2021	-	7.08	7.22	8.02	8.05	8.20	9.02	9.16	9.30	-
Beixin	2019	6.19	7.03	7.17	7.19	7.31	8.14	8.28	9.11	9.25	10.09
	2021	-	7.08	7.22	7.25	8.05	8.20	9.02	9.16	9.30	-
Xinbeichun	2019	6.19	7.03	7.17	7.31	7.31	8.14	8.28	9.11	9.25	10.09
	2021	-	7.08	7.22	7.26	8.05	8.20	9.02	9.16	9.30	-

"-" indicate that there was no data at this sampling time.

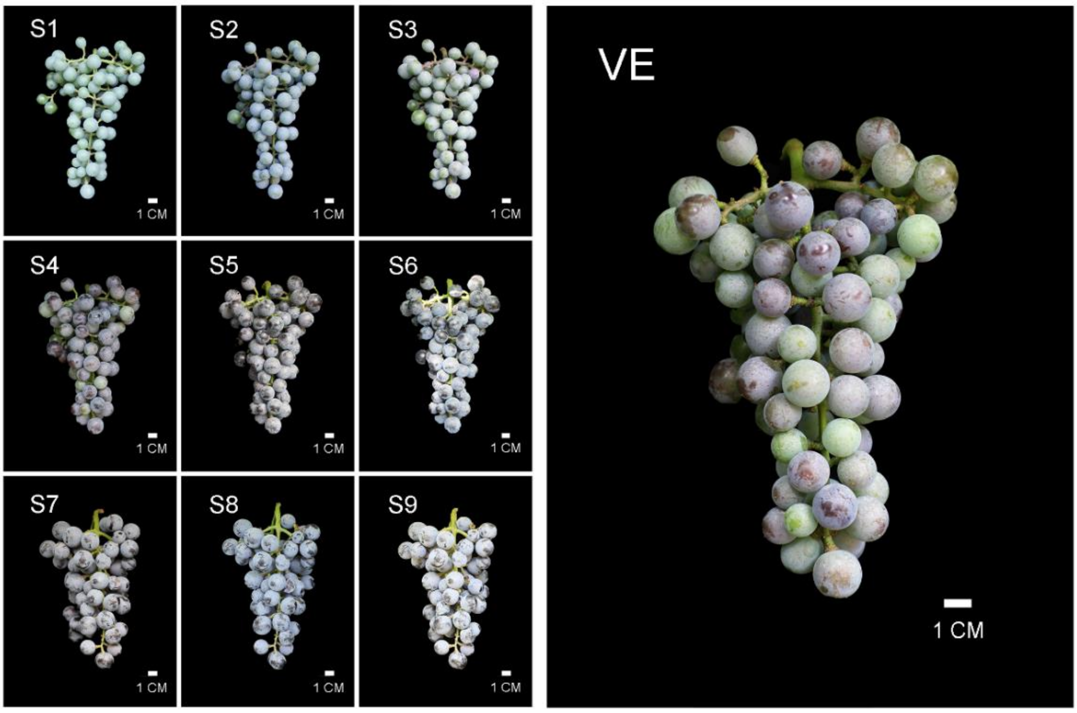

Figure 1. Photos of 'Beimei' were taken during sampling time in 2019. S1–S9 represent the sampling date in 2019, and bar = 1 cm.

2.2. Determination of Grape Berry Weight

To determine the weight of a single grape berry for each variety, 20 grape berries were randomly picked and pooled as a group for measurement of their weight. The resulting value was divided by 20 to obtain the average weight of a single berries. At least three groups were measured for each variety, and the experiment was repeated three times.

2.3. Determination of Water Content

To determine the water content of the grape berries, nine berries from each variety were randomly selected and weighted using an electronic balance (Sartorius) with an accuracy of 0.0001. After the measurement of fresh weight (FW), the berries were put in an oven and incubated at 65 °C until weight loss have ceased. Then, the weight was recorded as dry weight (DW). All the samples were measured three times, and the water content was calculated as [(Fw − Dw)/FW] × 100.

2.4. Determination of Soluble Solids

The soluble solids content was determined using a portable sugar meter, and ultrapure water was used as control.

2.5. Extraction and Determination of Sugar and Acid

The frozen grapes were thawed at room temperature and then crushed to collect the juice. About 2 mL of grape juice were centrifugated (8000 rpm, 10 min, 4 °C), and 1 mL of supernatant juice was diluted to 5 mL with Milli-Q water. Wine samples were thawed at room temperature, the solution was filtered through a 0.22-μm filter and put into the sample bottle for testing. Preparation of samples and determination of sugar and acid was performed according to Zhang et al. [36] and Liu et al. [37], respectively. Sugars were analyzed by a HPLC (Waters 2695, Milford, MA, USA) system which was equipped with a Waters RI-2414 detector. The separation column is an Aminex column HPX-87C, 300 × 7.8 mm (Bio-Rad, Hercules, CA, USA), equipped with a precolumn Carbo-C microguard cartridge (Bio-Rad). The eluant consists of Milli-Q water, filtered (0.2 μm) and degassed under vacuum. The column is kept at 85 °C, and the applied flow rate is 0.6 mL/min. Sugar reagents were purchased from Sigma Chemical Co., Saint Louis, MO, USA. Tartaric and malic acids were detected with a HPLC (Waters 2695, USA) system which was equipped with a Waters PDA-2998 detector, and the Intersil ODS-3 column (250 × 4.6 mm, 5 μm particle size) was used. The column was maintained at 40 °C, all samples were eluted with 0.02 mol/L KH_2PO_4 solution with pH 2.4. the flow rate was 0.8 mL/min, the acids were detected at 210 nm, and the acid reagents were purchased from Sigma Chemical Co. The β ratio of tartaric acid/malic acid is an important index for the evaluation of acid content [14,15]; β = tartaric acid content/malic acid content.

2.6. Winemaking and Sampling of Beimei Dry Red Wine

A total of 4000 kg of 'Beimei' grapes were transported to the winery immediately after being handpicked and pumped in a 5000 L fermenter by screw pump after being crushed. Then, 30 mg/L of enzymes (LALLZYME, EX-V, France) were added. The time was recorded after one centrifugal pump over (about 1/3 liquid) and was counted as 0 h. The first sample was collected in 50 mL centrifuge tube after half an hour, and recorded as stage A (0.5 h, density 1.107 g/mL). Then 60 mg/L of sulfur dioxide was added and pump over once. Samples at stage B were collected after 12 h (12 h, density 1.108 g/mL). Then 250 mg/L of yeast (Ceca, Angel Yeast Co., Ltd., Yichang, China) was added to start alcohol fermentation (AF), which is stage C (93 h, density 1.063 g/mL), and followed with stage D (206 h, density 0.997 g/mL) at the end of AF. Subsequently, bacteria (31MRB, Lallemand Co., Ltd., Toulouse, France) were added to start malolactic fermentation (MLF), which led to stage E (1099 h, density 0.993 g/mL) when the MLF was halfway. Stage F (1598 h, 0.993 g/mL) was reached after MLF was finished and wines were dry. Temperature and density were measured and recorded during winemaking. The fermentation temperature was maintained at 25–30 °C, Cap punching was performed four times a day during AF. Three technical replicates were performed for each sample, frozen in liquid nitrogen and stored at −80 °C. The fermentation curve and sampling intervals are shown in Figure 2.

2.7. Analysis of the General Parameters of Beimei Red Wine

Alcohol content, total acid, residual sugar, volatile acid, total sulfur, dry extract, and chroma were determined according to the methods of Wang et al. [38].

2.8. Determination of Resveratrol

The frozen samples were thawed at room temperature and the sample solution was filtered through a 0.22-μm filter and put into the sample bottle for testing. All samples were determined by a HPLC system (Waters 2695, Waters, USA) equipped with a Waters PDA-2998 detector, and C18 column of Atlantis T3 (Waters, USA) was used. Column temperature was 30 °C and injection volume was 10 µL. Trans-isomers were detected at 306 nm and cis-isomers at 288 nm. Determination of resveratrol was performed according to Liu et al. [30].

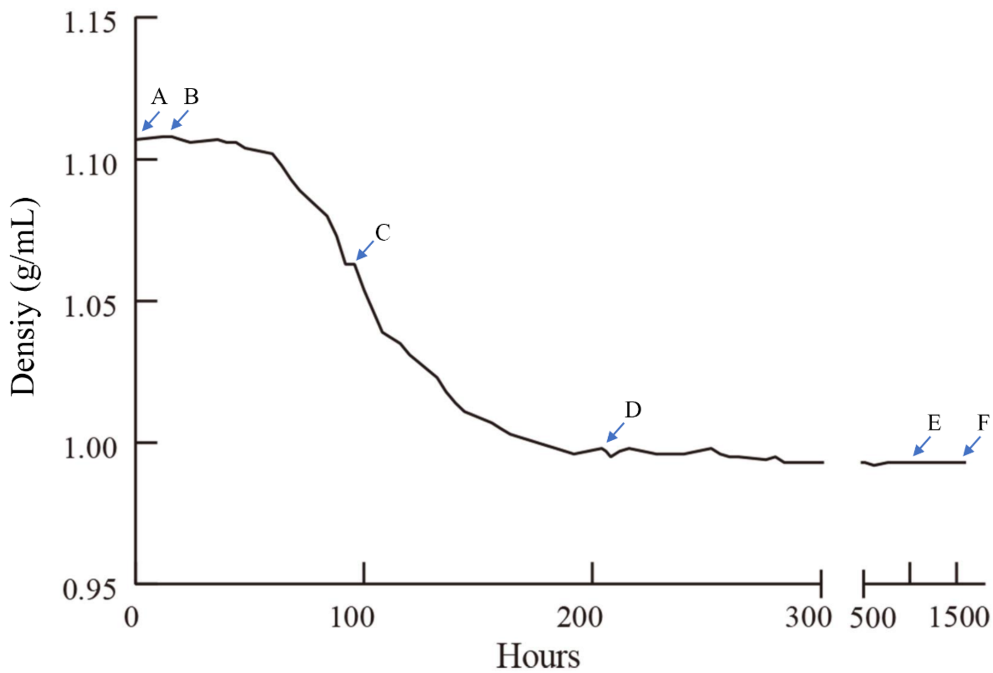

Figure 2. Fermentation curve and sampling intervals of Beimei red winemaking. Stage A (0.5 h, Density 1.107 g/mL, after grape crushed). Stage B (12 h, Density 1.108 g/mL, after SO_2 added). Stage C (93 h, Density 1.063 g/mL, middle AF). Stage D (206 h, Density 0.997 g/mL, between AF and MLF). Stage E (1099 h, Density 0.993 g/mL, middle MLF). Stage F (1598 h, Density 0.993 g/mL, after MLF).

2.9. Determination of Procyanidins

Procyanidins was extracted by a procyanidins kit from Nanjing Jiancheng Bioengineering Institute (kit number: A144-1-1) and then detected by a spectrophotometer (722, Yoke instrument, Shanghai, China).

2.10. Extraction and Determination of Flavor

To determine grape berry and wine flavor, the seeds were first removed from grape berries, then the seed-free berries were quickly frozen in liquid nitrogen and ground into powder with a mortar and pestle. A total of 5 g powder (5 mL wine sample) was transferred into a sample bottle and 10 µL of 3-octanol solution (41.10 mg/L) was added as the internal standard. Determination of flavor was performed according to the methods described by Zhang et al. [39]. Briefly, gas chromatography equipped with a DB-17-MS capillary column (30 m × 0.25 mm × 0.25 µm; J & W, Folsom, CA, USA) was used to perform GC-MS analysis where Agilent 7890A in conjunction with Agilent 5975 C quadrupole mass spectrometer (Agilent, Santa Clara, CA, USA) were applied. The samples were analyzed using the following oven program: 40 °C for 5 min, 40–70 °C at 2 °C/min, 70 °C for 2 min, 70–120 °C at 3 °C/min, 120–150 °C at 5 °C/min, 150–220 °C at 10 °C/min, and then 220 °C for 2 min. The injector, transfer line and ion source temperatures were maintained at 250 °C, 280 °C and 230 °C respectively. In this line, the electronic impact (EI) of 70 eV was applied, which scanned in the range of m/z 30–300 at a rate of 2.88 scans/s. Besides, helium was used as the carrier gas at a flow rate of 1 mL/min.

3. Results

3.1. Weight Changes of "Bei" Varieties' Berries

Determining of the weight of grape berries at different developmental stages showed that the weight generally increased during berry development and peaked at S5. Subsequently, the berry weight decreased at samples S6 to S7. In general, the changes in grape berries weight in both 2019 and 2021 were similar. However, in 2019, a decrease of 13% berry weight of 'Beimei' grapes was detected after reaching its peak of berry weight. The final weight of 'Beimei' grape berries at sample S9 was about 1.94 g (Figure 3A). 'Beihong' and 'Beixi' berry weight decreased by 22% and 23%, respectively, after they reached their peaks of berry weight, which resulted in a low berry weight (1.32 g and 1.19 g, respectively) at sample S9 (Figure 3B,C). By contrast, the other two varieties, 'Beixin' and 'Xinbeichun', showed a higher berry weight (2.47 g and 2.43 g, respectively) at sample S9. The percentage of berry weight changes for 'Beixin' and 'Xinbeichun' from samples S5 to S9 was 1% and 4%, respectively (Figure 3D,E).

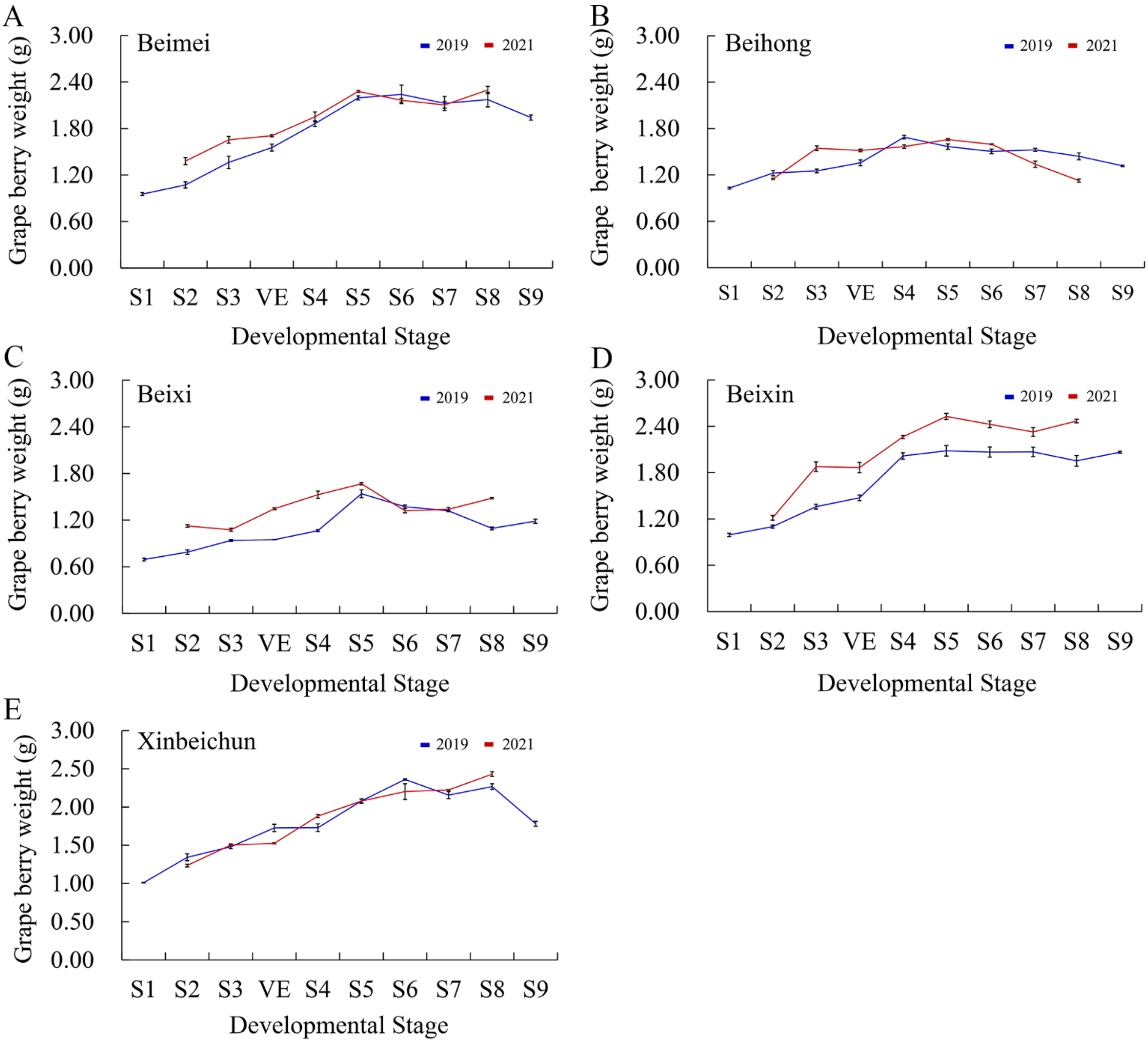

Figure 3. Berry weight changes at different stages of development of 'Beimei' (**A**), 'Beihong' (**B**), 'Beixi' (**C**), 'Beixin' (**D**) and 'Xinbeichun' (**E**). S1 sampling date was 19 June 2019. S2 sampling date in

2021 was 8 July. S1–S9 were sampled once every two weeks, VE = veraison. Data are mean values of three replicates, the error bars indicate the SE from repeats.

3.2. Water Content Changes of "Bei" Varieties

Determination of water content showed that the changing trend in water content of 'Beimei' and other "Bei" wine grape berries was similar over two years (Figure 4). However, the average water content in 2021 was higher than that in 2019 (Figure 4), probably due to the increased in the annual precipitation during 2021. The water content of grape berries decreased during the development. The water content of 'Beimei' was 71.38% at sample S9 (Figure 4A), and the biggest change was observed in 'Beihong', whose water content was as low as 56.68% at sample S9 (Figure 4B). The water content of the other varieties was 72.03% ('Beixi'), 68.05% ('Beixin'), and 79.43% ('Xinbeichun', sample S8) (Figure 4).

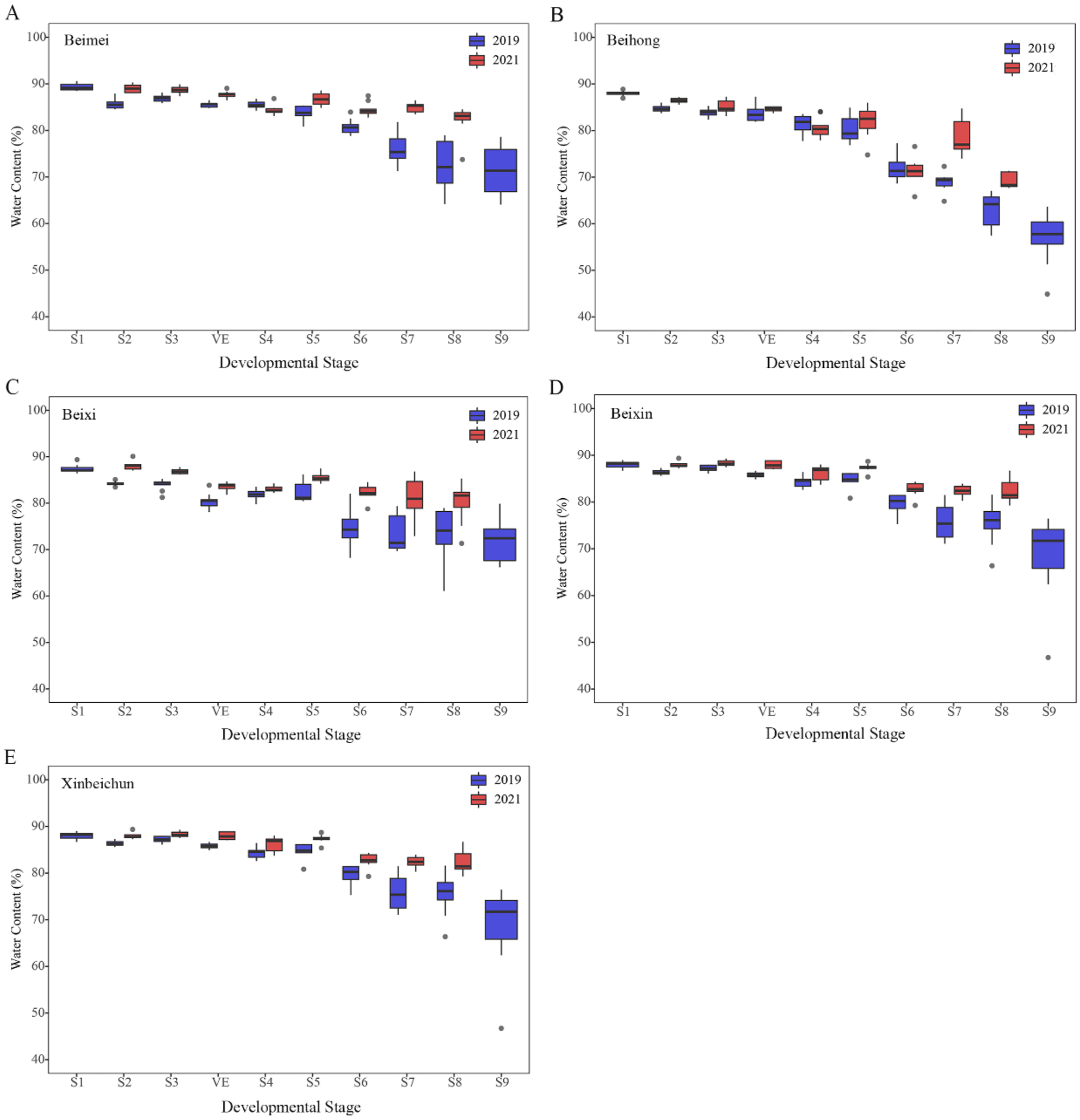

Figure 4. Water content changes at different development stages in 'Beimei' (**A**), 'Beihong' (**B**), 'Beixi' (**C**), 'Beixin' (**D**) and 'Xinbeichun' (**E**). S1 sampling date was 19 June 2019. S2 sampling date was 2021 is 8 July. S1–S9 were sampled once every two weeks, VE = veraison. Data are mean values of nine repeats.

3.3. Detection of Soluble Solids of "Bei" Varieties

Determination of soluble solids content in 'Beimei' and the other "Bei" wine grape berries revealed that it generally increased during the development, and the content was relatively stable between the two years (Figure 5). For example, in 2019, the soluble solids content of 'Beimei' increased significantly from samples S2 to S9, and the final content was 24.9 °Brix (Figure 5A). Furthermore, the soluble solids content of 'Beihong' increased significantly from samples S2 to S9, and the final content at sample S9 was as high as 32.2 °Brix (Figure 5B). Notably, soluble solid contents rapidly increased after stage VE and peaked at ripening sample S8 or S9 (Figure 5). Among the five varieties, 'Beihong' contained the highest content of soluble solids (32.2 °Brix), while 'Xinbeichun' had the lowest soluble solids content (22.7 °Brix) (Figure 5E).

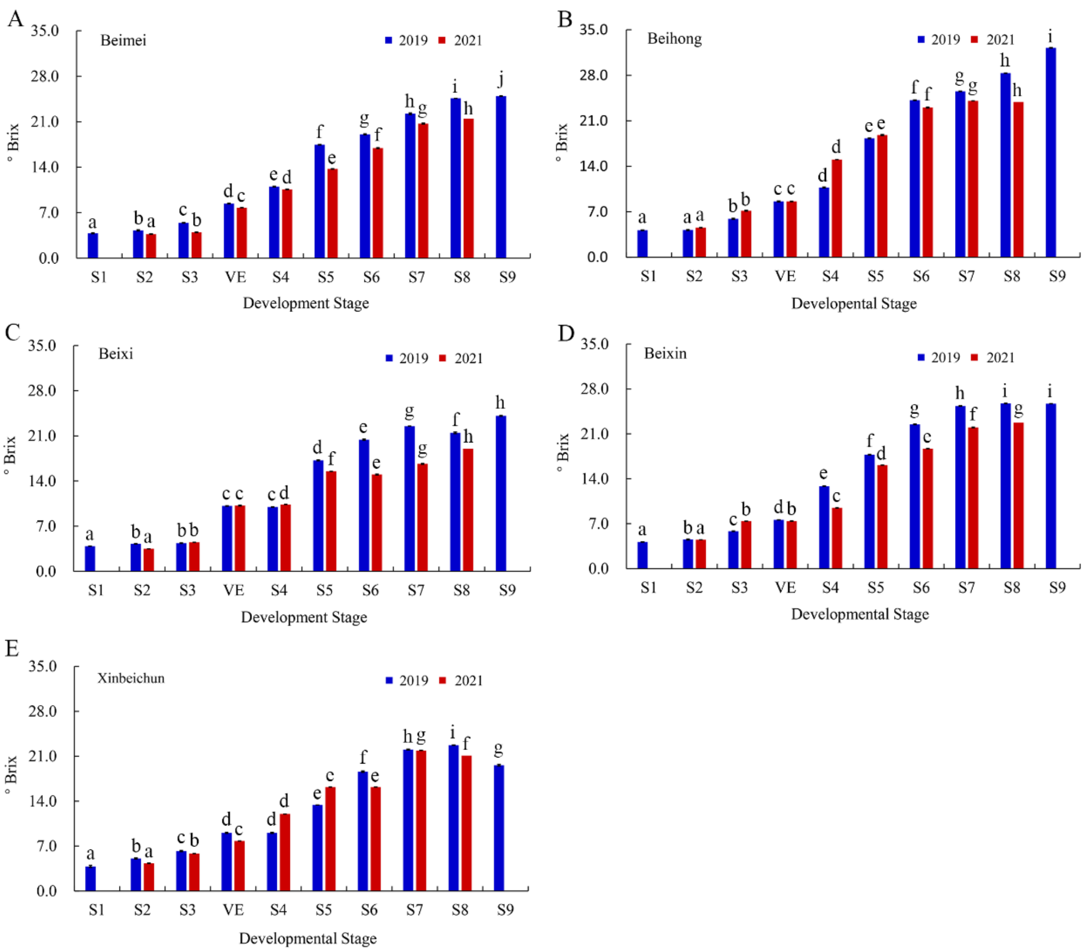

Figure 5. Soluble solid changes of 'Beimei' (**A**), 'Beihong' (**B**), 'Beixi' (**C**), 'Beixin' (**D**) and 'Xinbeichun' (**E**). S1 sampling date was 19 June 2019. S2 sampling date in 2021 was 8 July. S1–S9 were sampled once every two weeks, VE = veraison. Data are mean values of three replicates. The error bars indicate the SE from repeats. The letters above the bars indicate significant differences ($p < 0.01$, determined by one-way ANOVA).

3.4. Analysis of Sugars in "Bei" Varieties

Analysis of sugars in 'Beimei' and the other "Bei" wine grape berries showed that both glucose and fructose increased during berry development of the five varieties (Figure 6). The sugar content of 'Beimei' peaked at S8 and remained unchanged until S9 (Figure 6A). The sugar content of 'Beixi', 'Beixin', and 'Xinbeichun' varieties peaked at S7. 'Beixin' remained unchanged and 'Xinbeichun' decreased at sample S9 (Figure 6). However, in 'Beihong', the sugar content increased during all developmental stages (Figure 6B). The ratio of glucose and fructose content was about 1:1 at sample S9. Moreover, sucrose was also detected in 'Beihong' from samples S6 to S9, whereas the berries from the other varieties contained no sucrose (Figure 6). The sugar content of these varieties was analyzed in 2017. Consistent with previous results [14], only 'Beihong' sucrose was detected, and the sucrose content of 'Beihong' was about 2% of total sugars (Figure 6F). The results suggest that 'Beihong' was a variety with low sucrose content.

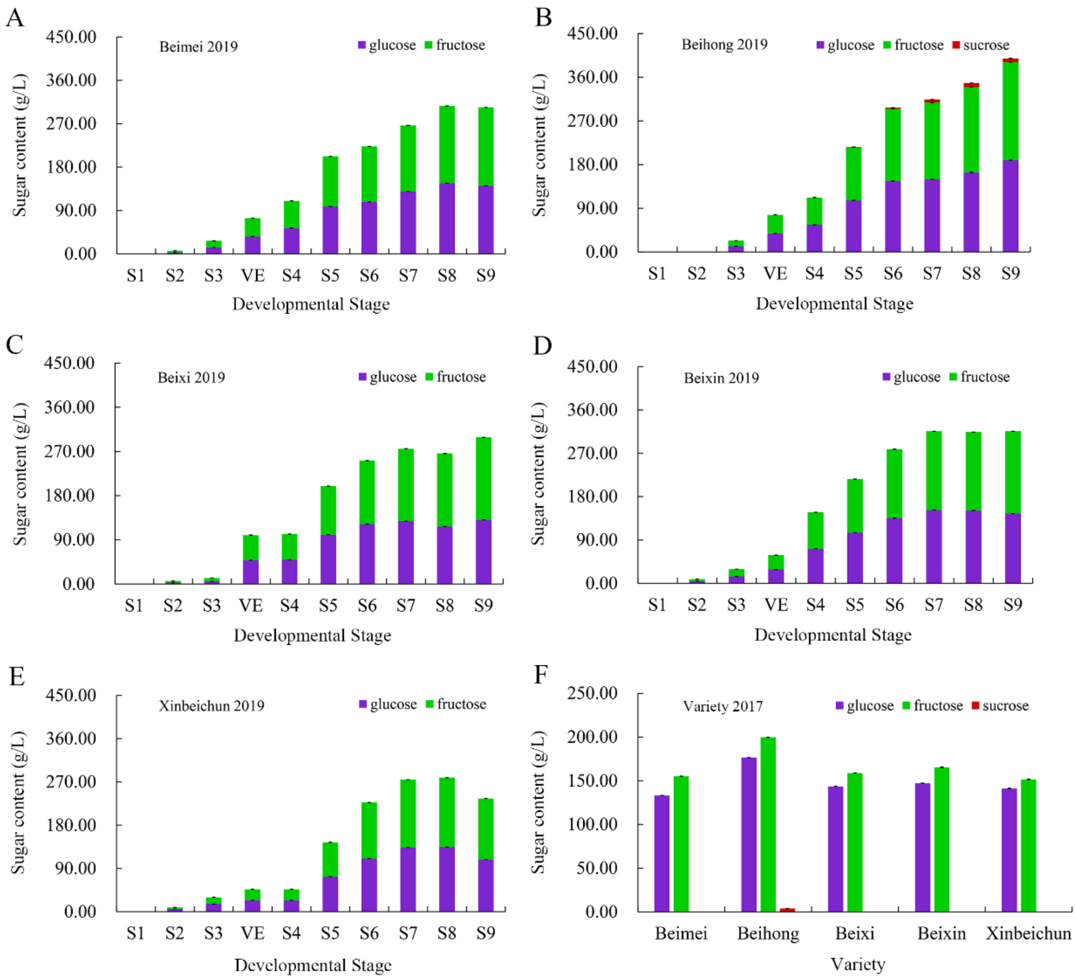

Figure 6. Changes of sugar content of 'Beimei' (**A**), 'Beihong' (**B**), 'Beixi' (**C**), 'Beixin' (**D**) and 'Xinbeichun' (**E**), 'Variety' (**F**). S1 sampling date was 19 June 2019. S2 sampling date in 2021 was 8 July. S1–S9 were sampled once every two weeks, VE = veraison. Data are mean values of three replicates, the error bars indicate the SE from triplicate repeats.

3.5. Analysis of Acids in "Bei" Varieties

The content of tartaric acid and malic acid in 'Beimei' and the other "Bei" wine grape berries during development was investigated, and results showed that both tartaric acid and malic acid accumulated at early developmental stages. In general, the accumulation of tartaric acid and malic acid reached the peaks at sample S2 and then decreased gradually (Figure 7). The total content of tartaric acid and malic acid in 'Beihong', 'Beimei', 'Xinbeichun', 'Beixin', and 'Beixi' at sample S9 was 8.3, 7.4, 7.3, 6.7, and 6.2 g/L, respectively (Figure 7). The corresponding β ratio values were 1.46 ('Beihong'), 2.17 ('Beimei'), 4.31 ('Xinbeichun'), 4.85 ('Beixi'), and 3.16 ('Beixin'), respectively (Figure 7F).

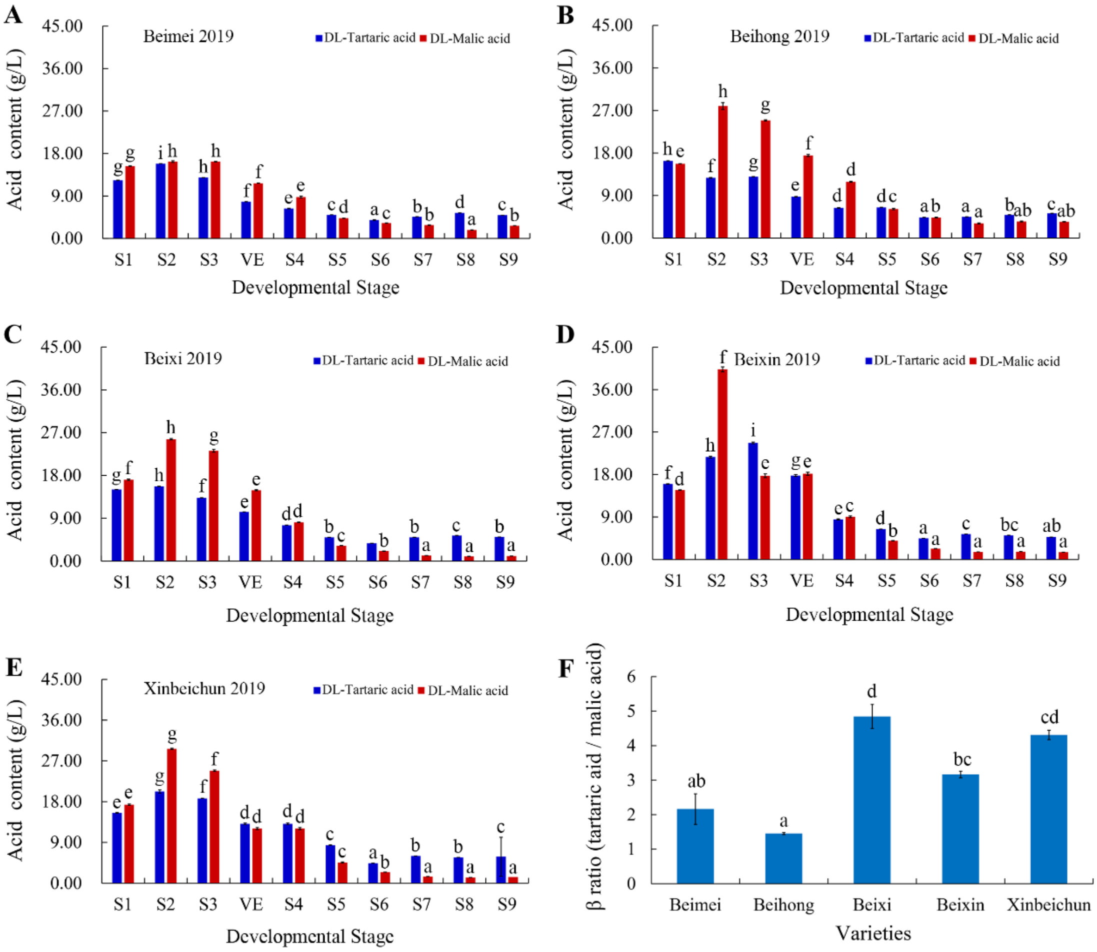

Figure 7. Acid content changes at different development stages of 'Beimei' (**A**), 'Beihong' (**B**), 'Beixi' (**C**), 'Beixin' (**D**) and 'Xinbeichun' (**E**), 'Variety' (**F**). S1 sampling date was 19 June 2019. S2 sampling date in 2021 was 8 July. S1–S9 were sampled once every two weeks, VE = veraison. Data are mean values of three repeats. The error bars indicate the SE from repeats. The letters above the bars indicate significant differences ($p < 0.01$, determined by one-way ANOVA).

3.6. Analysis of "Bei" Varieties for the Flavor Compounds

GC-MS was used to identify the flavor compounds in 'Beimei' and the other "Bei" wine grapes. A total of 33 compounds were detected in 'Beimei'. The number of compounds detected in 'Beixi', 'Beixin', 'Beihong', and 'Xinbeichun' were 28, 25, 21, and 18, respectively (Figure 8A). Identification of the detected flavor compounds in the five varieties showed that eight types of flavor compounds were shared by all varieties, including benzene acetaldehyde (Figure 8B). Given that these varieties are the hybrids of 'Muscat Humburg' (*V. vinifera*) and Shanputao (*V. amurensis*), the wines made from 'Beimei', 'Beixin', and 'Beixi' grapes are characterized by an intense rose aroma, and therefore the flowery compounds were analyzed. It was shown that benzene acetaldehyde was the main floral aroma compound in all varieties (Figure 8C). During berry development, the benzene acetaldehyde content decreased in all grape varieties. However, this compound was only detected in 'Beimei' at sample S8 (9.75 μg/kg) and 'Beihong' at sample S9 (9.21 μg/kg). In addition, phenylethyl alcohol (PA) was found in all varieties, except 'Xinbeichun' (Figure 8E). Interestingly, PA continued to increase at late developmental stages in these varieties except for 'Beixin', in which the PA content decreased at sample S9 (Figure 8E). Notably, the content of PA in 'Beimei' at sample S9 was 16.01 μg/kg, which is twice as high as that in 'Beihong' (Figure 8E). The high PA content may partially contribute to the intense rose aroma in 'Beimei' wine. In addition, the content of neryl alcohol (NA), another rose aroma compound, was also significantly higher in 'Beimei' than that in 'Beixi' and 'Beixin' grapes (Figure 8D). However, NA was not detected in 'Beihong' or 'Xinbeichun' grapes. Moreover, citronellol was also detected in 'Beimei' at sample S7 (5.92 μg/kg) and at sample S9 (12.96 μg/kg). Geraniol was detected in 'Beimei' at sample S9 (147.79 μg/kg). Citronellol and geraniol are compounds that can contribute to the sensory characteristics of rose aroma. Other flavor compounds of "Bei" varieties were shown in Appendix A Table A1.

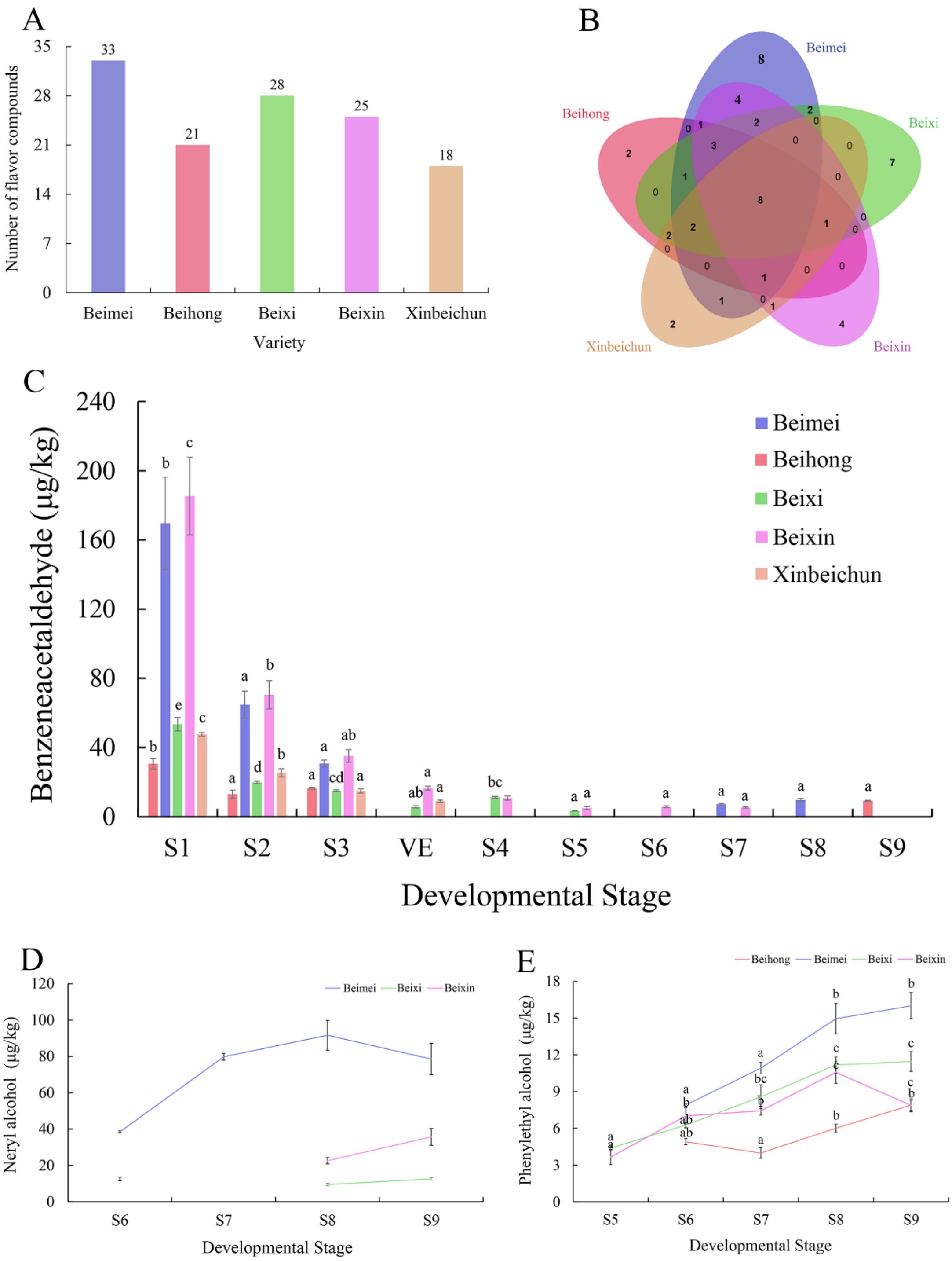

Figure 8. Analysis of 'Beimei' and other wine grape varieties' flavor compounds. (**A**) The number is the sum of compounds in all different stages of each variety of "Bei" wine grape varieties.

(**B**) Difference analysis of all flavor compounds of "Bei" wine grape varieties. Different color represents the corresponding variety, and the number represents the types of compounds. (**C**) S1 sampling date was 19 June 2019. S1–S9 were sampled once every two weeks, VE = veraison. Data are mean values of three repeats. The error bars indicate the SE from repeats. The letters above the bars indicate significant differences ($p < 0.01$, determined by one-way ANOVA). (**D**) S6–S9 represents the sampling date, Data are mean values of three replicates. The error bars indicate the SE from repeats. (**E**) S5–S9 represents the sampling date. Data are the mean values of three replicates. The error bars indicate the SE from repeats. The letters above the bars indicate significant differences ($p < 0.01$, determined by one-way ANOVA).

3.7. Evaluation of Beimei Red Wine

At the end of Beimei wine fermentation, the alcohol content was 14.2% vol, total acid content was 10.9 g/L, and residual sugar content was 5.9 g/L (Table 2). The general parameters of Beimei red wine were all in line with GB/T15037 National Standard. The sensory tasting panel concluded: deep ruby red, with bright pink edges, intense rose and violet aromas, cherry and cranberry sweetness, pleasant acidity, full-body, clear tannins, and a clean finish, an excellent base wine. This provides a reliable sample for subsequent research.

Table 2. General parameters of Beimei red wine.

Alcohol	Total Acid	Residual Sugar	Volatile Acid	Total Sulfur	Dry Extract
(%vol)	(g/L)	(g/L)	(g/L)	(mg/L)	(g/L)
14.2 ± 0.00	10.9 ± 0.10	5.9 ± 0.07	0.4 ± 0.01	56.3 ± 7.31	29.5 ± 0.10

Data are mean values ± SE of three technical repeats.

3.8. Changes of Metabolites during Beimei Red Winemaking

Fermentation is the conversion of sugars into alcohol. The sugar content of the samples from different fermentation stages were analyzed by HPLC (Figure 9). Glucose content was completely metabolized by yeast at stage D, whereas fructose was present-0.66 g/L at stage F. Mannitol was detected from stage C and increased to the highest level at stage E, then decreased at stage F (Figure 9A).

In this study, resveratrol was detected at stage C, resveratrol increased and reached its highest concentration at stage D. From stage D to F, the content of trans-PD (trans piceid) decreased, while the content of cis-PD (cis piceid) increased. The content of trans-RES (trans-resveratrol) remained unchanged, while the total content of resveratrol increased, and the final content was 18 mg/L (Figure 9B).

To distinguish color changes of Beimei red wine during fermentation, the chroma at different fermentation stages where tested (Figure 9C). It showed that there were no changes between stage A and B. However, the chroma increased significantly from stage C to E, and reached highest intensities at stage E and remained unchanged up to stage F.

Procyanidins at stage B increased in comparison to stage A, but there was no significant difference between stage A and B. Procyanidins at stage C increased significantly compared with stage A and B and reached the highest content at stage C. Procyanidins at stages D and E showed no significant change compared with that at stage C. Procyanidins at stage F was significantly lower than that at stage C (Figure 9D).

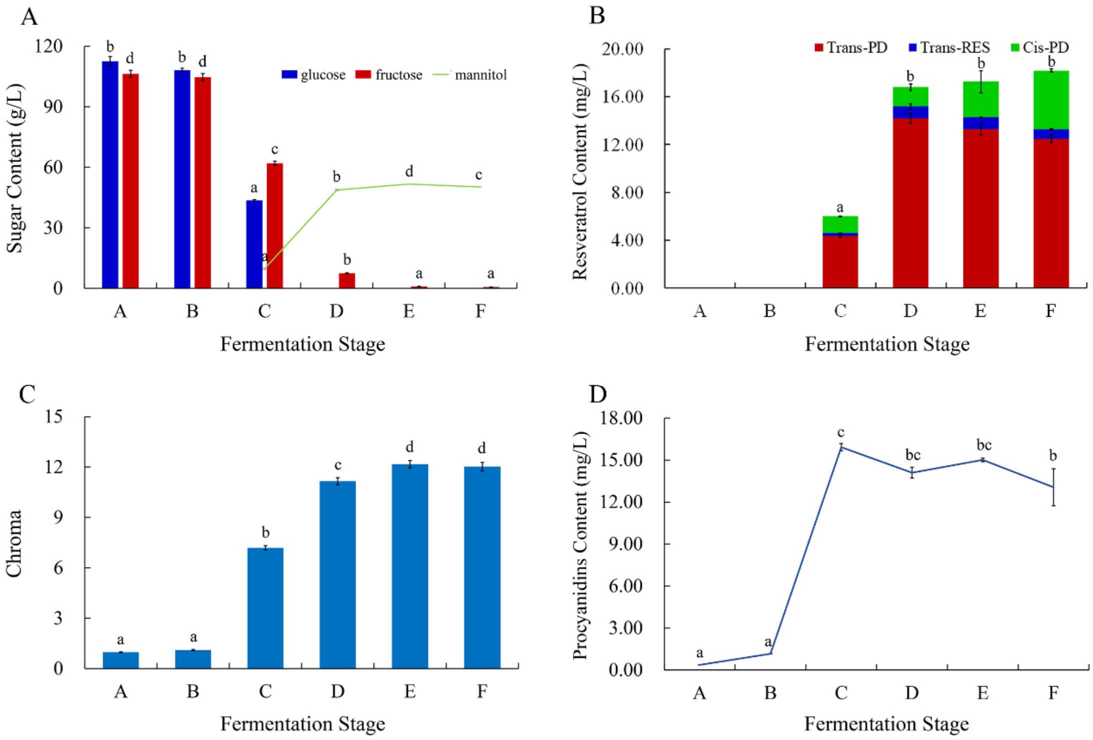

Figure 9. Changes of sugar (**A**), resveratrol (**B**), chroma (**C**) and procyanidins (**D**) content during Beimei red winemaking. Letters A to F on X-axis represent different fermentation stages during the winemaking of Beimei red wine. The data are mean values of three replicates. The error bars indicate SE from repeats. All letters above the bars indicate significant differences ($p < 0.01$, determined by one-way ANOVA).

3.9. Flavor Compounds of Beimei Red Wine

GC-MS was used to detect the flavor compounds of Beimei red wine samples at different fermentation stages. A total of 27 flavor compounds were identified (Table 3). Four were aromatic compounds of rose aroma, which were acetic acid, 2-phenylethyl acetate (APA), PA, NA, and Beta-Damascenone (BD). APA and PA were detected at stage C, and increased continuously to stage F, while NA and BD were only detected at stages E and F with a continuous increase. Furthermore, three types of compounds associated with other floral characteristics were detected: Tetradecanoic acid, ethyl ester (violets and iris), dodecanoic acid, ethyl ester (pleasant petal aroma), and styrene (fragrant). These three compounds were detected at stage C, and their content increased up to stage F. In addition, a total of 11 compounds, including one alcohol compound, one acid compound, and nine ester compounds, were detected. These were aromatic compounds of fruit. Nine other compounds were also detected, representing four other odorous compounds: Hexadecanoic acid, ethyl ester (creamy), octanoic acid (sweet), butanedioic acid, diethyl ester (unique), and methyl salicylate (holly leaf), as well as five odorless or unidentified compounds.

Table 3. Changes of flavor compounds during winemaking of Beimei red wine.

Compounds	Flavour	A (μg/L)	B (μg/L)	C (μg/L)	D (μg/L)	E (μg/L)	F (μg/L)
Beta-Damascenone	rose	ND	ND	ND	ND	22.81 ± 7.33^{a}	36.35 ± 19.85^{a}
Neryl alcohol	rose	ND	ND	ND	ND	97.74 ± 31.88^{a}	177.08 ± 100.61^{a}
Acetic acid, 2-phenylethyl ester	rose	ND	ND	82.01 ± 1.66^{a}	86.75 ± 4.39^{a}	164.93 ± 52.99^{a}	248.15 ± 133.76^{a}
Phenylethyl Alcohol	rose	ND	ND	277.09 ± 4.05^{a}	555.89 ± 26.48^{a}	1570.81 ± 465.91^{a}	2648.54 ± 1489.78^{a}
Tetradecanoic acid, ethyl ester	violet, iris	ND	ND	66.00 ± 0.60^{a}	ND	138.79 ± 47.39^{a}	191.95 ± 106.14^{a}
Dodecanoic acid, ethyl ester	petal aroma	ND	ND	970.26 ± 30.25^{a}	964.64 ± 42.76^{a}	1857.22 ± 616.60^{a}	2394.99 ± 1322.48^{a}
Styrene	fragrant	ND	ND	15.61 ± 1.12^{a}	39.95 ± 7.49^{a}	110.38 ± 41.23^{a}	222.67 ± 28.74^{a}
Hexanoic acid	fruit	ND	ND	28.73 ± 3.03	34.41 ± 2.26	ND	ND
1-Hexanol	fruit	43.05 ± 15.64^{a}	73.98 ± 0^{a}	60.40 ± 4.93^{a}	100.28 ± 29.63^{a}	278.09 ± 80.28^{ab}	347.40 ± 47.45^{b}
Acetic acid, octyl ester	fruit	ND	ND	32.33 ± 0.70^{b}	15.33 ± 2.20^{a}	20.35 ± 7.36^{ab}	ND

Table 3. *Cont.*

Compounds	Flavour	A (μg/L)	B (μg/L)	C (μg/L)	D (μg/L)	E (μg/L)	F (μg/L)
Decanoic acid, ethyl ester	fruit, brandy	ND	ND	5356.76 ± 104.94 ^{a}	2587.12 ± 220.32 ^{a}	5257.99 ± 1853.51 ^{a}	7514.63 ± 4214.19 ^{a}
Octanoic acid, 3-methylbutyl ester	fruit, brandy	ND	ND	154.65 ± 4.68 ^{b}	67.99 ± 6.93 ^{a}	83.83 ± 29.39 ^{a}	33.15 ± 17.80 ^{a}
Butanoic acid, ethyl ester	pineapple, banana	ND	ND	17.13 ± 0.77 ^{a}	39.79 ± 2.61 ^{a}	101.27 ± 31.20 ^{a}	172.86 ± 93.88 ^{a}
Isoamyl acetate	banana	ND	ND	1182.75 ± 75.94 ^{a}	1772.34 ± 151.97 ^{a}	3010.50 ± 957.49 ^{a}	4852.33 ± 2704.97 ^{a}
Octanoic acid, methyl ester	sweet orange	ND	ND	37.16 ± 4.12 ^{a}	36.11 ± 3.22 ^{a}	129.36 ± 45.92 ^{a}	235.90 ± 133.36 ^{a}
Octanoic acid, ethyl ester	pineapple, apple	ND	ND	5596.42 ± 139.47 ^{a}	5122.37 ± 553.93 ^{a}	12,921.28 ± 4565.12 ^{a}	22,822.73 ± 12,941.19 ^{a}
Hexanoic acid, ethyl ester	pineapple	ND	ND	1412.64 ± 92.31 ^{a}	1974.88 ± 174.86 ^{a}	3857.39 ± 1283.73 ^{a}	6668.49 ± 3767.05 ^{a}
Acetic acid, hexyl ester	pear, apple	ND	ND	584.48 ± 36.43 ^{a}	331.95 ± 27.93 ^{a}	498.78 ± 164.81 ^{a}	790.87 ± 446.95 ^{a}
Hexadecanoic acid, ethyl ester	cream	ND	ND	14.65 ± 0.84 ^{a}	ND	300.22 ± 94.96 ^{ab}	470.10 ± 238.06 ^{b}
Octanoic Acid	Sweat smell	ND	ND	ND	137.99 ± 9.18 ^{a}	372.41 ± 119.37 ^{a}	587.64 ± 308.71 ^{a}
Butanedioic acid, diethyl ester	spcial smell	ND	ND	ND	ND	58.44 ± 18.20 ^{a}	132.14 ± 70.95 ^{a}
Methyl salicylate	holly leaf	ND	3.04 ± 0.02 ^{a}	30.78 ± 0.30 ^{ab}	44.51 ± 2.42 ^{ab}	121.28 ± 38.98 ^{ab}	193.53 ± 105.82 ^{b}
Decanoic acid, methyl ester	OU	ND	ND	59.42 ± 1.05 ^{a}	15.60 ± 1.52 ^{a}	36.57 ± 12.93 ^{a}	59.34 ± 33.17 ^{a}
Pentadecanoic acid, 3-methylbutyl ester	OU	ND	ND	40.98 ± 1.90 ^{a}	72.61 ± 7.45 ^{a}	95.30 ± 32.00 ^{a}	72.82 ± 40.25 ^{a}
neryl propanoate	OU	ND	ND	18.89 ± 0.49 ^{a}	20.89 ± 0.94 ^{a}	22.12 ± 7.48 ^{a}	ND
Dodecane	OU	ND	ND	10.03 ± 1.06 ^{a}	17.63 ± 2.77 ^{a}	57.78 ± 16.70 ^{a}	87.68 ± 48.99 ^{a}
2,3-Butanediol	OU	ND	ND	ND	41.61 ± 10.46 ^{a}	ND	100.23 ± 54.19 ^{a}

A, B, C, D, E and F represent the samples at different stages of Beimei red winemaking. Data are mean values ± SE of three replicates. Different letters on the same line indicate significant differences (p < 0.01, determined by one-way ANOVA). OU means odorless, ND represent not detected.

4. Discussion

This study investigated the quality traits of 'Beimei' grapes and other "Bei" grape varieties in 2019 and 2021. The development of grape berries was affected by unusually high precipitation in Beijing in 2021. The high precipitation led to the lack of data at the sample S9 in 2021 due to fruit rot. This study, therefore, mainly focused on the data collected in 2019, while the data collected in 2021 were used as a reference.

The results showed that the ratio of glucose and fructose in the grapes of 'Beimei' and other varieties were 1:1, which is consistent with a previous result [40]. In the process of Beimei red winemaking, glucose is preferentially converted into alcohol by yeast, which is consistent with the results of a previous study [41]. At the end of fermentation, only 0.66 g/L fructose remained, indicating that this wine is a qualified dry wine. Mannitol is half as sweet as sucrose in the same concentration [42]. This provides a new explanation for the more obvious sweetness and rounder body of Beimei red wine (residual sugar < 4 g/L).

Sugar and acid contents of 'Beimei' grapes were similar to *V. vinifera* [43], and types of aroma and NA and PA contents of 'Beimei' grapes were higher than that in other "Bei" varieties. Through analysis of the results from flavor detection by GC-MS and sensory evaluation, it is concluded that the typical aroma of Beimei wine was rose, and the main aroma substances were APA, PA, NA, and BD. APA and PA appeared in the middle of AF, and their contents increased as fermentation progressed, while NA and BD appeared in the middle of MLF (stage E). NA is sensually expressed as a rose-floral and red-berry aroma [44–46]. BD is the main component of rose essential oil, which has a strong rose fragrance and a sensory threshold of 1–2 μg/L in wine [47]. However, the BD content in Beimei red wine is as high as 36.35 μg/L. Although PA and NA were also detected in Beimei grapes, the content was only 16 μg/L and 98 μg/L, respectively. In Beimei wine, the concentration of these two compounds was up to 2648 μg/L and 177 μg/L, respectively. This might be the reason why Beimei wine has a strong bouquet of roses, while Beimei grapes taste unscented. The resveratrol content of 'Beimei' red wine was higher than that in *V. vinifera* wines [48,49].

5. Conclusions

By analyzing the changes in the quality parameters during developmental stages and winemaking, it was found that 'Beimei' grapes have enough sugar content and a suitable acid content. The characteristic rose aroma compounds, such as PA and NA, were significantly higher than that of the other "Bei" wine grape varieties. Beimei red wine has enough secondary metabolites, especially resveratrol, and rich rose aromas, and the main aroma substances of rose fragrance are APA, PA, NA, and BD. The results suggest that 'Beimei' could be used for high quality wines, providing a new choice for the development of the wine industry of China.

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Appendix A

Figures A1–A4 Photos of "Bei" wine grape varieties were taken during the sampling time in 2019. Table A1 shows the types and content of flavor compounds at different developmental stages of "Bei" wine grape varieties.

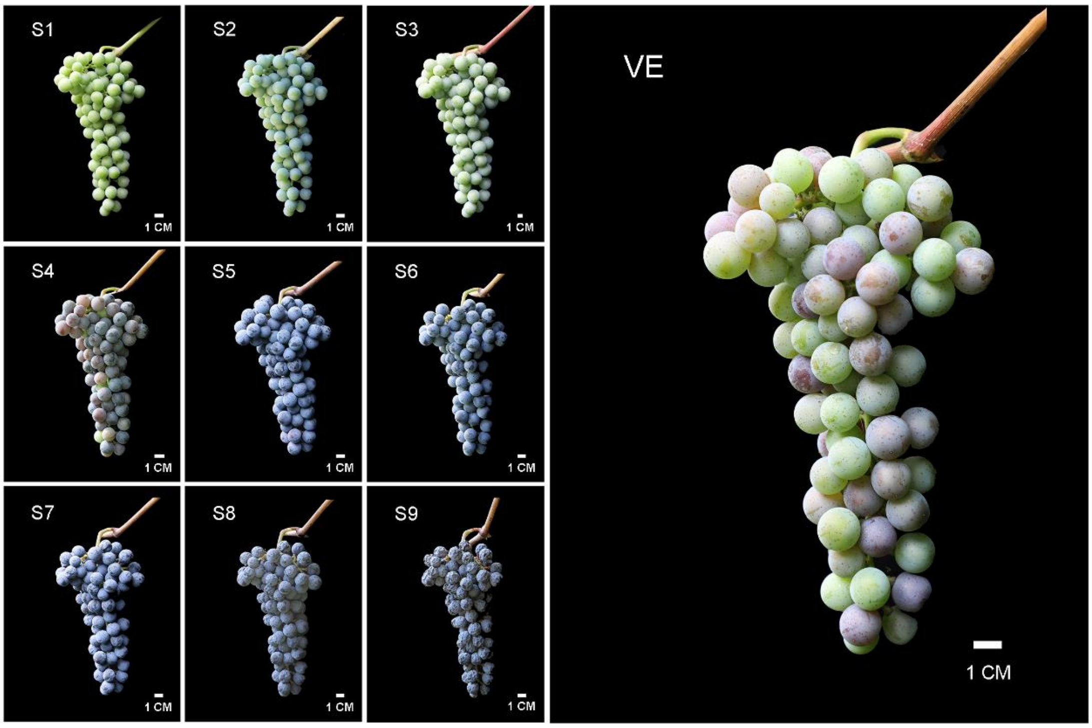

Figure A1. Photos of 'Beihong' were taken during sampling time in 2019. S1–S9 represent the sampling date in 2019, and bar = 1 cm.

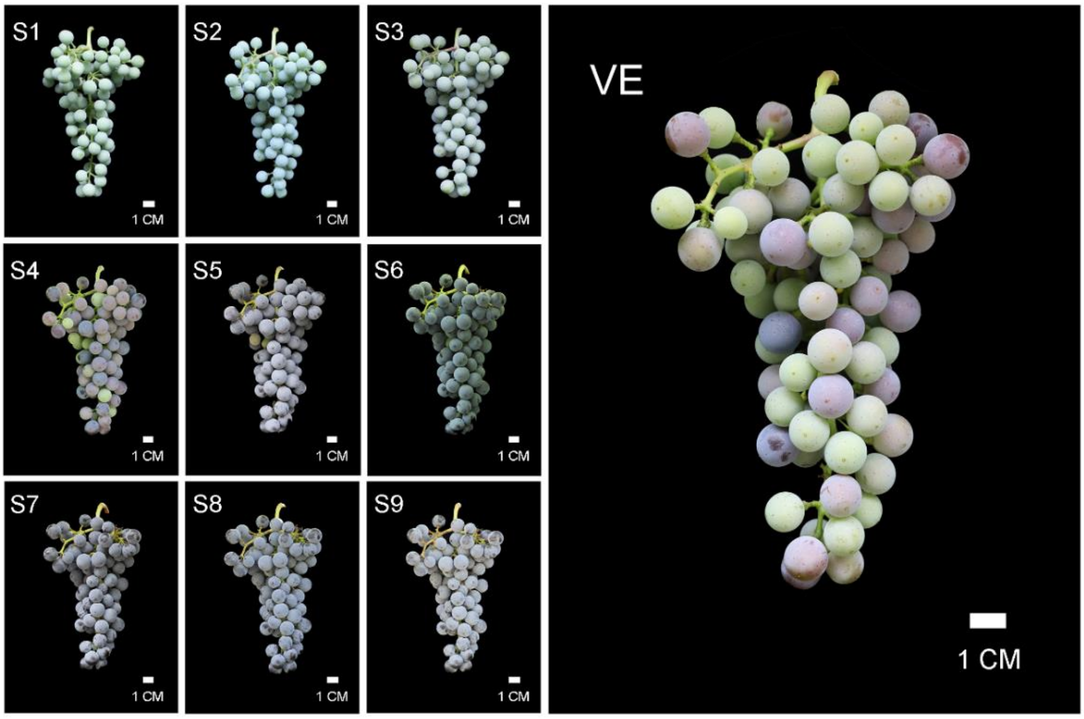

Figure A2. Photos of 'Beixi' were taken during sampling time in 2019. S1–S9 represent the sampling date in 2019, and bar = 1 cm.

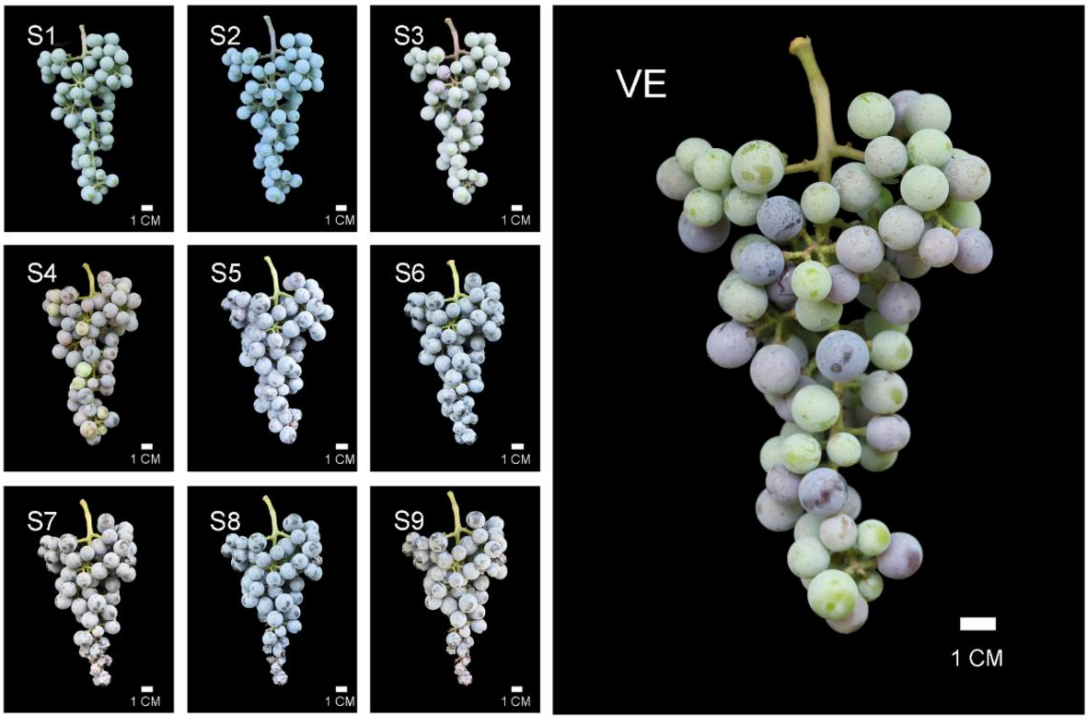

Figure A3. Photos of 'Beixin' were taken during sampling time in 2019. S1–S9 represent the sampling date in 2019, and bar = 1 cm.

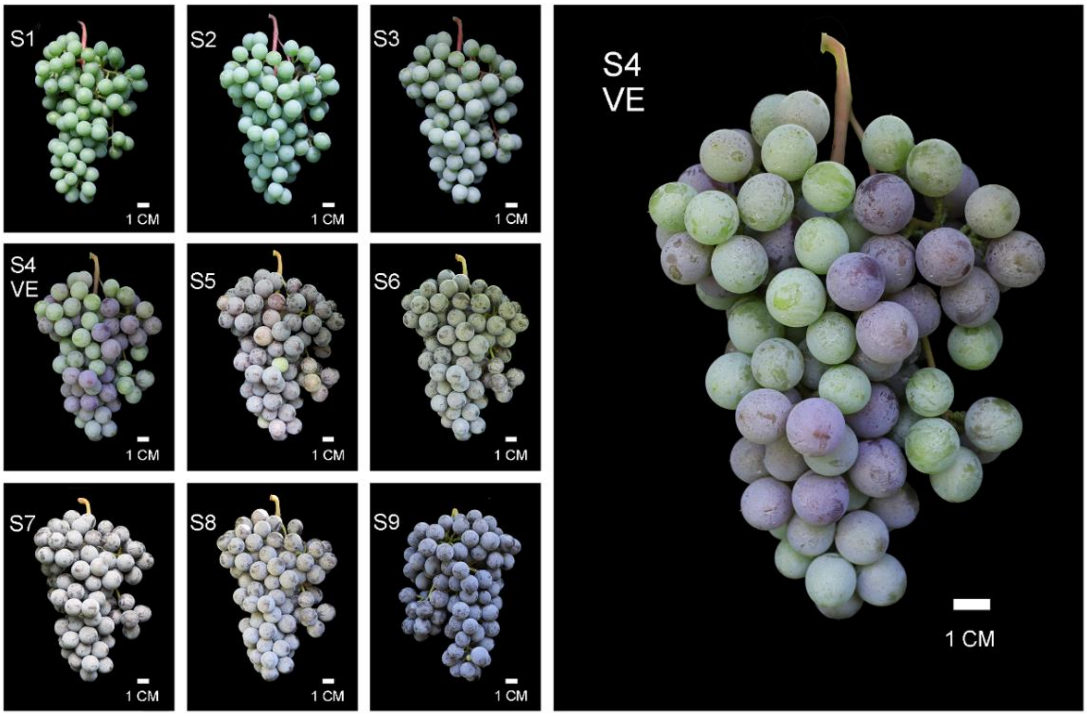

Figure A4. Photos of 'Xinbeichun' were taken during sampling time in 2019. S1–S9 represent the sampling date in 2019, and bar = 1 cm.

Table A1. Changes of flavor compounds at different sampling date in developmental stages of "Bei" wine grape varieties.

Variety	Compounds	CAS	Flavour	S1 (μg/kg)	S2 (μg/kg)	S3 (μg/kg)	VE (μg/kg)	S4 (μg/kg)	S5 (μg/kg)	S6 (μg/kg)	S7 (μg/kg)	S8 (μg/kg)	S9 (μg/kg)
	3-Hexen-1-ol, acetate, (Z)-	003681-71-8	Grass	19.21 ± 2.75 a	33.14 ± 4.59 a	17.12 ± 0.84 a	ND	ND	ND	ND	ND	ND	ND
	2-Hexenal	000505-57-7	Grass	124.96 ± 13.61 a	139.82 ± 15.98 a	124.72 ± 8.38 a	543.70 ± 196.96 b	616.65 ± 9.79 b	1326.71 ± 11.00 d	803.89 ± 9.35 bc	1251.12 ± 31.86 d	1055.48 ± 52.50 cd	599.81 ± 60.18 b
	3-Hexen-1-ol	000544-12-7	Grass	82.06 ± 8.88 a	109.50 ± 14.98 a	115.12 ± 5.62 a	ND	ND	ND	ND	ND	ND	ND
	Methyl salicylate	000119-36-8	Holly Leaf	ND	ND	ND	ND	ND	ND	3.18 ± 0.07 a	5.44 ± 0.29 a	4.72 ± 0.46 a	14.57 ± 1.55 b
	2-Hexen-1-ol, (Z)-	000928-95-0	Apple	ND	33.03 ± 3.85 a	61.46 ± 3.32 a	67.75 ± 11.57 ab	48.67 ± 0.70 a	35.23 ± 3.78 a	150.51 ± 12.43 c	78.49 ± 4.39 ab	124.71 ± 34.02 bc	148.97 ± 13.93 c
	2-Octenal, (E)-	002548-87-0	Cucumber	ND	4.48 ± 0.86	ND	ND	ND	ND	ND	ND	ND	ND
	Geranial	005392-40-5	Lemon	ND	ND	ND	ND	ND	ND	3.27 ± 0.08 a	4.38 ± 0.21 ab	5.86 ± 0.79 b	9.40 ± 0.37 c
	Cis-3-Hexenyl hexanoate	031501-11-8	Pear	1.65 ± 0.17	2.82 ± 0.37	ND	ND	ND	ND	ND	ND	ND	ND
	Myrcene	000123-35-3	Orange	ND	ND	ND	ND	ND	ND	ND	11.41 ± 0.39 a	11.96 ± 0.98 a	19.50 ± 2.08 b
	Nonanal	000124-19-6	Sweet Orange	5.23 ± 0.67 ab	5.72 ± 0.63 ab	5.14 ± 0.19 ab	4.75 ± 0.42 a	6.98 ± 0.16 bc	5.34 ± 0.19 ab	8.10 ± 0.23 c	7.04 ± 0.10 bc	8.51 ± 0.62 cd	10.30 ± 0.82 d
Beimei	Hexanoic acid, ethyl ester	000123-66-0	Fruit	7.93 ± 1.87	ND	ND	ND	ND	ND	7.90 ± 0.10	ND	ND	ND
	1-Hexanol, 2-ethyl-	000104-76-7	Special Smell	7.06 ± 0.79	ND	ND	ND	ND	ND	ND	ND	ND	ND
	Benzene acetaldehyde	000122-78-1	Hosta Flower	169.62 ± 26.80 b	64.80 ± 7.83 a	30.78 ± 2.02 a	ND	ND	ND	ND	7.35 ± 0.40 a	9.75 ± 0.83 a	ND
	Caryophyllene	000087-44-5	Clove	4.24 ± 0.75 ab	3.30 ± 0.12 a	6.12 ± 0.86 ab	10.36 ± 2.63 b	6.41 ± 0.49 ab	ND	ND	ND	ND	ND
	Phenylethyl Alcohol	000060-12-8	Rose	ND	ND	ND	ND	ND	ND	7.91 ± 0.14 a	10.92 ± 0.46 a	14.96 ± 1.23 b	16.01 ± 1.07 b
	Citronellol	000106-22-9	Rose	ND	ND	ND	ND	ND	ND	ND	5.92 ± 0.10	ND	12.96 ± 0.78
	Rose oxide	016409-43-1	Rose	4.69 ± 0.87	ND	ND	ND	ND	ND	ND	ND	ND	ND
	Geraniol	000106-24-1	Rose	ND	ND	ND	ND	ND	ND	ND	ND	ND	147.79 ± 13.20
	Neryl alcohol	000106-25-2	Rose	ND	ND	ND	ND	ND	ND	38.48 ± 0.50 a	79.79 ± 1.87 b	91.63 ± 8.22 b	78.56 ± 8.66 b
	2-Hexen-1-ol, acetate, (E)-	002497-18-9	Fragrant	ND	8.67 ± 1.00	7.34 ± 0.59	ND	ND	ND	ND	ND	ND	ND
	Benzaldehyde	000100-52-7	Fragrant	12.17 ± 2.26 a	7.93 ± 4.89 a	3.81 ± 2.06 a	ND	ND	ND	ND	ND	ND	ND
	Styrene	000100-42-5	Fragrant	ND	ND	ND	ND	ND	ND	ND	ND	23.81 ± 3.49	ND
	Benzyl Alcohol	000100-51-6	Fragrant	ND	ND	ND	ND	ND	4.83 ± 0.24 a	5.26 ± 0.04 a	11.19 ± 0.84 b	16.46 ± 1.34 c	22.88 ± 1.48 d
	2,4-Hexadienal, (E,E)-	000142-83-6	OU	ND	ND	ND	ND	ND	5.08 ± 0.22	ND	ND	ND	10.94 ± 1.05
	3,7-dimethyl-, (Z)-1,3,6-Octatriene,	003338-55-4	OU	ND	ND	ND	ND	ND	ND	ND	8.14 ± 0.18	13.43 ± 1.39	ND
	Geranic acid	000459-80-3	OU	ND	ND	ND	ND	ND	ND	7.04 ± 0.29 a	9.62 ± 0.30 ab	11.95 ± 1.15 b	ND
	(1S)-(+)-3-Carene	000498-15-7	OU	ND	ND	ND	ND	ND	ND	ND	32.48 ± 0.75	ND	ND
	Cis-citral	000106-26-3	OU	ND	ND	ND	ND	ND	ND	ND	ND	ND	5.13 ± 0.18
	(E)-2-Hexenyl butyrate	053398-83-7	OU	ND	9.83 ± 1.09	5.74 ± 0.11	ND	ND	ND	ND	ND	ND	ND
	cis-Linaloloxide	1000121-97-4	OU	ND	5.75 ± 0.74	ND	ND	ND	ND	ND	ND	ND	ND
	Cis-3-Hexenyl butyrate	016491-36-4	OU	5.62 ± 0.77 a	13.93 ± 1.40 b	4.74 ± 0.37 a	ND	ND	ND	ND	ND	ND	ND
	1-Hexanol	000111-27-3	OU	ND	ND	ND	127.30 ± 2.26 b	153.21 ± 2.47 bc	71.80 ± 2.94 a	158.30 ± 11.20 bc	112.37 ± 4.41 ab	141.38 ± 21.72 b	191.91 ± 11.54 c
	Hexanal	000066-25-1	OU	95.52 ± 11.92 a	115.75 ± 15.51 a	112.57 ± 5.32 a	258.98 ± 65.16 b	411.64 ± 5.98 c	743.46 ± 20.36 e	557.73 ± 8.27 d	777.44 ± 20.44 e	698.36 ± 30.15 e	508.36 ± 45.94 cd
Beihong	Eucalyptol	000470-82-6	Cool	9.82 ± 2.28	ND	ND	ND	ND	ND	ND	ND	ND	ND
	3-Hexen-1-ol	000928-96-1	Grass	111.35 ± 17.31 ab	192.66 ± 33.45 c	175.37 ± 12.56 bc	131.18 ± 4.49 abc	153.21 ± 2.47 bc	71.80 ± 2.94 a	ND	ND	ND	ND
	2-Hexenal	000505-57-7	Grass	177.26 ± 18.19 ab	94.45 ± 17.18 a	164.16 ± 3.88 ab	439.26 ± 164.55 bc	608.06 ± 1.21 c	1326.71 ± 11.00 d	1122.80 ± 159.39 d	594.25 ± 11.62 c	598.54 ± 31.83 c	341.46 ± 17.87 abc
	Cis-3-Hexenyl hexanoate	031501-11-8	Pear	17.49 ± 12.50	11.05 ± 1.84	ND	ND	ND	ND	ND	ND	ND	ND
	Butanoic acid, hexyl ester	002639-63-6	Sweet Fruit	ND	10.02 ± 1.73	4.76 ± 0.29	ND	ND	ND	ND	ND	ND	ND
	Hexanoic acid, ethyl ester	000123-66-0	Fruit	ND	ND	ND	ND	ND	ND	ND	ND	ND	18.30 ± 2.35
	Nonanal	000124-19-6	Sweet Orange	4.80 ± 0.47 ab	5.07 ± 0.75 b	6.04 ± 0.33 bc	4.69 ± 0.36 ab	6.98 ± 0.16 c	5.34 ± 0.19 b	5.284 ± 0.24 b	3.36 ± 0.08 a	4.40 ± 0.24 ab	ND
	2-Hexen-1-ol, (E)-	000928-95-0	Apple	ND	ND	31.42 ± 1.89 a	57.91 ± 1.76 abc	48.67 ± 0.70 ab	35.23 ± 3.78 a	73.55 ± 25.94 abc	101.41 ± 11.68 c	83.74 ± 4.63 bc	43.18 ± 4.26 ab
	Benzene acetaldehyde	000122-78-1	Hosta flower	30.64 ± 3.00 b	13.03 ± 2.27 a	16.48 ± 0.42 a	ND	ND	ND	ND	ND	ND	9.21 ± 0.36
	Caryophyllene	000087-44-5	Clove	17.39 ± 7.27 a	49.96 ± 10.69 b	35.77 ± 2.82 ab	18.70 ± 5.75 a	6.41 ± 0.49 a	ND	ND	ND	ND	ND
	Geraniol	000106-24-1	Rose	ND	ND	ND	ND	ND	ND	ND	ND	ND	2.54 ± 0.04
	Benzaldehyde	000100-52-7	Fragrant	ND	ND	ND	ND	ND	ND	ND	ND	ND	4.92 ± 0.23
	Phenylethyl Alcohol	000060-12-8	Rose	ND	ND	ND	ND	ND	ND	4.91 ± 0.25 ab	3.99 ± 0.42 a	6.02 ± 0.32 b	7.90 ± 0.43 b
	Benzyl Alcohol	000100-51-6	Fragrant	ND	ND	ND	ND	ND	4.83 ± 0.24 a	13.04 ± 4.47 ab	19.12 ± 2.47 b	18.65 ± 0.88 b	23.68 ± 1.44 b

Table A1. *Cont.*

Variety	Compounds	CAS	Flavour	S1 (µg/kg)	S2 (µg/kg)	S3 (µg/kg)	VE (µg/kg)	S4 (µg/kg)	S5 (µg/kg)	S6 (µg/kg)	S7 (µg/kg)	S8 (µg/kg)	S9 (µg/kg)
	Cis-3-Hexenyl butyrate	016491-36-4	OU	12.66 ± 5.08 a	42.65 ± 7.18 b	20.54 ± 1.28 ab	ND	ND	ND	ND	ND	ND	ND
	(E)-2-Hexenyl butyrate	053398-83-7	OU	ND	5.07 ± 0.82	4.57 ± 0.22	ND	ND	ND	ND	ND	ND	ND
	2,4-Hexadienal, (E,E)-	000142-83-6	OU	ND	ND	ND	ND	ND	5.08 ± 0.22	ND	ND	ND	ND
	Hexanoic acid	000142-62-1	OU	ND	ND	ND	ND	ND	ND	ND	ND	4.54 ± 0.68	ND
	1-Octanol	000111-87-5	OU	ND	ND	ND	ND	ND	ND	ND	ND	ND	3.08 ± 0.14
	1-Hexanol	000111-27-3	OU	ND	ND	ND	ND	ND	ND	121.18 ± 27.60 a	193.26 ± 29.19 a	165.36 ± 9.90 a	203.44 ± 25.72 a
	Hexanal	000066-25-1	OU	158.98 ± 20.33 a	118.24 ± 23.87 a	166.29 ± 12.43 a	266.18 ± 58.03 ab	411.64 ± 5.98 b	743.46 ± 20.36 c	716.71 ± 7.36 c	664.96 ± 34.61 c	741.64 ± 32.69 c	751.46 ± 83.79 c
	3-Hexen-1-ol, (Z)-	000928-96-1	Grass	75.58 ± 3.76	126.97 ± 7.91	ND	ND	ND	ND	ND	ND	ND	ND
	2-Hexenal, (E)-	000505-57-7	Grass	136.88 ± 8.14 a	194.57 ± 7.98 a	83.70 ± 0.90 a	339.99 ± 23.46 b	481.48 ± 21.66 c	1265.05 ± 21.57 e	806.21 ± 43.63 d	822.22 ± 66.70 d	190.93 ± 20.29 a	428.64 ± 29.85 bc
	Eucalyptol	000470-82-6	Cool	34.70 ± 2.15	13.52 ± 1.25	ND	ND	ND	ND	ND	ND	ND	ND
	Methyl salicylate	000119-36-8	Holly Leaf	ND	ND	ND	ND	ND	4.14 ± 0.21	ND	ND	ND	ND
	2-Hexen-1-ol, (E)-	000928-95-0	Apple	ND	ND	ND	115.37 ± 6.39 ab	80.88 ± 3.30 ab	39.22 ± 0.94 a	50.76 ± 5.87 a	62.70 ± 18.51 a	677.90 ± 50.50 c	170.28 ± 10.21 b
Beixi	g-Terpinene	000099-85-4	Orange	8.23 ± 0.74	ND	ND	ND	ND	ND	ND	ND	ND	ND
	Nonanal	000124-19-6	Sweet Orange	6.76 ± 0.12 cd	7.09 ± 0.52 d	5.61 ± 0.34 bcd	6.27 ± 0.29 bcd	3.87 ± 0.09 a	5.02 ± 0.25 ab	5.95 ± 0.52 bcd	5.66 ± 0.42 bcd	5.22 ± 0.44 abc	5.65 ± 0.20 bcd
	Ethyl hexanoate	000123-66-0	Fruit	ND	ND	ND	ND	10.58 ± 0.50	ND	ND	ND	ND	ND
	Hexyl butyrate	002639-63-6	Sweet Fruit	ND	5.88 ± 0.37	ND	3.47 ± 0.31	ND	ND	ND	ND	ND	ND
	Benzaldehyde	000100-52-7	Bitter Almond	6.37 ± 2.72	2.74 ± 0.12	ND	ND	ND	ND	ND	ND	ND	ND
	Phenylacetaldehyde	000122-78-1	Hosta Flower	53.40 ± 3.88 e	19.86 ± 0.74 d	15.07 ± 0.44 cd	5.71 ± 0.59 ab	11.28 ± 0.48 bc	3.36 ± 0.18 a	ND	ND	ND	ND
	Phenylethyl Alcohol	000060-12-8	Rose	ND	ND	ND	ND	ND	4.39 ± 0.19 a	6.27 ± 0.23 ab	8.58 ± 0.98 bc	11.18 ± 0.67 c	11.45 ± 0.80 c
	Citronellol	000106-22-9	Rose	2.60 ± 0.11 a	3.24 ± 0.08 ab	4.17 ± 0.14 ab	3.94 ± 0.25 ab	4.68 ± 0.17 b	9.39 ± 0.50 d	10.26 ± 0.97 de	11.64 ± 0.43 e	6.93 ± 0.51 c	11.84 ± 0.37 e
	Neryl alcohol	000106-25-2	Rose	ND	ND	ND	ND	ND	ND	ND	ND	9.52 ± 0.69	12.58 ± 0.72
	Geraniol	000106-24-1	Rose	2.39 ± 0.19 a	ND	2.44 ± 0.16 a	ND	2.38 ± 0.10 a	ND	21.66 ± 1.64 bc	26.98 ± 3.34 cd	19.41 ± 1.44 b	33.37 ± 1.80 d
	o-Xylene	000095-47-6	Fragrant	ND	ND	40.57 ± 3.10	ND	ND	ND	ND	ND	ND	ND
	Benzyl Alcohol	000100-51-6	Fragrant	ND	ND	ND	ND	ND	2.69 ± 0.18 a	5.44 ± 0.05 a	8.56 ± 2.22 a	7.47 ± 0.53 a	16.43 ± 1.53 b
	Methyl geranate	002349-14-6	OU	ND	ND	ND	ND	ND	ND	2.21 ± 0.22 a	3.40 ± 0.15 a	6.48 ± 0.44 b	6.82 ± 0.27 b
	3-Hexenyl hexanoate	084434-19-5	OU	ND	2.45 ± 0.07	ND	ND	ND	ND	ND	ND	ND	ND
	Geranic acid	000459-80-3	OU	ND	ND	ND	ND	ND	ND	4.44 ± 0.51	5.37 ± 0.15	ND	ND
	Octylbutyrate	000110-39-4	OU	ND	ND	2.90 ± 0.19	ND	ND	ND	ND	ND	ND	ND
	Cis-3-Hexenyl butyrate	016491-36-4	OU	9.59 ± 0.20 a	18.97 ± 1.28 b	11.10 ± 0.14 a	ND	ND	ND	ND	ND	ND	ND
	Trans-2-Hexenyl butyrate	053398-83-7	OU	ND	10.87 ± 0.62 b	5.96 ± 0.17 a	10.52 ± 0.25 b	ND	ND	ND	ND	ND	ND
	2,4-Hexadienal, (E,E)-	000142-83-6	OU	ND	ND	ND	ND	ND	6.19 ± 0.18	5.07 ± 0.40	ND	ND	ND
	Hexyl acetate	000142-92-7	OU	ND	ND	ND	ND	4.91 ± 0.13	ND	ND	ND	ND	ND
	m-isopropyltoluene	000535-77-3	OU	33.51 ± 1.55 d	26.51 ± 1.17 c	13.32 ± 0.71 b	11.11 ± 0.88 ab	8.24 ± 0.44 a	ND	ND	ND	ND	ND
	1-Hexanol	000111-27-3	OU	ND	ND	ND	136.19 ± 7.84 ab	98.32 ± 3.33 a	57.76 ± 1.41 a	72.29 ± 2.88 a	104.84 ± 6.76 a	693.83 ± 68.67 c	238.38 ± 13.27 b
	Hexanal	000066-25-1	OU	121.82 ± 8.00 a	128.73 ± 4.97 a	88.64 ± 4.78 a	263.35 ± 25.62 b	292.85 ± 10.59 b	645.10 ± 23.66 de	538.18 ± 43.80 c	718.77 ± 39.32 e	139.99 ± 14.14 a	559.42 ± 35.05 cd
	2-Hexenal	000505-57-7	Grass	102.82 ± 15.66 a	118.90 ± 5.88 a	190.52 ± 22.79 a	178.68 ± 15.60 a	713.47 ± 93.30 b	1024.10 ± 245.40 bc	1025.57 ± 112.34 bc	1305.57 ± 92.54	949.66 ± 92.21 bc	853.69 ± 27.18 b
	3-Hexen-1-ol	000544-12-7	Grass	72.94 ± 11.47 ab	118.31 ± 11.44 cd	112.75 ± 10.70 cd	88.86 ± 7.59 bc	ND	45.29 ± 8.35 a	61.87 ± 6.53 ab	ND	132.57 ± 4.84 d	53.45 ± 2.41 ab
	Leaf acetate	003681-71-8	Grass	ND	50.83 ± 0.89	ND	ND	ND	ND	ND	ND	ND	ND
	2-Hexen-1-ol, (E)-	000928-95-0	Apple	ND	ND	61.08 ± 6.87 a	125.42 ± 10.91 b	116.27 ± 14.19 b	41.93 ± 8.27 a	51.25 ± 4.78 a	31.73 ± 1.81 a	119.66 ± 12.65 b	ND
Beixin	Nonanal	000124-19-6	Sweet Orange	ND	ND	ND	ND	ND	3.31 ± 0.49 a	5.52 ± 0.78 ab	4.96 ± 0.18 ab	6.39 ± 0.49 b	ND
	Geranial	005392-40-5	Lemon	ND	ND	ND	ND	ND	ND	ND	ND	2.28 ± 0.13	ND
	Cis-3-Hexenyl hexanoate	031501-11-8	Pear	ND	4.66 ± 0.23	ND	ND	ND	ND	ND	ND	ND	ND
	Heptanal	000111-71-7	Fruit	ND	4.72 ± 0.15	ND	ND	ND	ND	ND	ND	ND	ND
	Hexanoic acid, ethyl ester	000123-66-0	Fruit	7.92 ± 1.94	ND	ND	ND	ND	ND	5.19 ± 0.87	ND	ND	ND
	Butanoic acid, hexyl ester	002639-63-6	Sweet Fruit	ND	2.85 ± 0.12	ND	ND	ND	ND	ND	ND	ND	ND
	Caryophyllene	000087-44-5	Clove	5.32 ± 0.55 ab	15.10 ± 2.02 d	13.57 ± 0.23 cd	9.30 ± 0.77 bc	4.10 ± 0.16 a	ND	ND	ND	ND	ND
	Benzene acetaldehyde	000122-78-1	Hosta Flower	185.32 ± 22.43 c	70.52 ± 8.18 b	35.13 ± 3.68 ab	16.47 ± 1.12 a	10.77 ± 1.17 a	5.03 ± 0.90 a	5.78 ± 0.44 a	5.30 ± 0.34 a	ND	ND
	Phenylethyl Alcohol	000060-12-8	Rose	ND	ND	ND	ND	ND	3.66 ± 0.61 a	7.01 ± 0.67 b	7.44 ± 0.36 b	10.58 ± 0.89 c	7.88 ± 0.53 bc
	Citronellol	000106-22-9	Rose	ND	ND	ND	ND	ND	ND	ND	ND	3.60 ± 0.46	ND
	Neryl alcohol	000106-25-2	Rose	ND	ND	ND	ND	ND	12.51 ± 1.04 a	ND	22.63 ± 1.70 ab	35.70 ± 4.61 b	ND
	Benzyl Alcohol	000100-51-6	Rose	ND	ND	ND	ND	ND	ND	4.35 ± 0.47 a	5.75 ± 0.76 a	11.53 ± 0.64 b	5.32 ± 0.68 a
	Styrene	000100-42-5	Fragrant	ND	ND	ND	ND	ND	ND	ND	ND	11.81 ± 1.52	ND
	Benzaldehyde	000100-52-7	Fragrant	1.87 ± 0.55	ND	ND	ND	ND	ND	ND	ND	ND	ND

Table A1. *Cont.*

Variety	Compounds	CAS	Flavour	S1 (µg/kg)	S2 (µg/kg)	S3 (µg/kg)	VE (µg/kg)	S4 (µg/kg)	S5 (µg/kg)	S6 (µg/kg)	S7 (µg/kg)	S8 (µg/kg)	S9 (µg/kg)
	Butanoic acid, 2-hexenyl ester, (E)-	053398-83-7	OU	ND	11.77 ± 0.28	ND	ND	ND	ND	ND	ND	ND	ND
	Cyclopentene, 1-methyl-	000693-89-0	OU	27.09 ± 3.21	ND	ND	13.07 ± 0.24	ND	ND	ND	ND	ND	ND
	Cyclohexene oxide	000286-20-4	OU	ND	ND	ND	ND	ND	ND	ND	20.77 ± 0.03	ND	ND
	Cis-3-Hexenyl 2-methylbutanoate	053398-85-9	OU	ND	4.34 ± 0.21	ND	ND	ND	ND	ND	ND	ND	ND
	Butanoic acid, 3-hexenyl ester, (Z)-	016491-36-4	OU	ND	19.69 ± 1.43 b	4.93 ± 0.08 a	2.86 ± 0.13 a	ND	ND	ND	ND	ND	ND
	3-Carene	013466-78-9	OU	ND	ND	ND	ND	ND	5.18 ± 0.17 a	12.98 ± 1.03 b	13.59 ± 0.82 b	23.77 ± 1.65 c	9.82 ± 0.28 b
	Hexanal	000066-25-1	OU	130.67 ± 18.82 a	159.03 ± 8.79 a	146.58 ± 18.57 a	144.25 ± 16.18 a	346.93 ± 46.91 ab	455.18 ± 118.00 bc	640.12 ± 72.28 cd	829.96 ± 67.64 d	615.34 ± 64.76 cd	605.88 ± 38.50 cd
	2-Hexenal	000505-57-7	Grass	85.37 ± 1.14 a	46.71 ± 4.26 a	93.25 ± 10.77 a	69.11 ± 1.71 a	69.11 ± 1.71 a	1195.75 ± 45.83 c	634.12 ± 53.83 b	744.92 ± 73.67 b	795.91 ± 158.28 b	ND
	3-Hexen-1-ol, (Z)-	000928-96-1	Grass	121.29 ± 1.51 a	128.76 ± 8.39 a	131.02 ± 14.49 a	ND	ND	ND	ND	ND	ND	ND
	Eucalyptol	000470-82-6	Cool	59.14 ± 2.52 c	26.82 ± 2.32 b	9.31 ± 1.00 a	2.37 ± 0.12 a	2.37 ± 0.12 a	ND	ND	ND	ND	ND
	Hexanoic acid, 3-hexenyl ester, (Z)-	031501-11-8	Pear	6.57 ± 0.04	6.40 ± 0.55	ND	ND	ND	ND	ND	ND	ND	ND
	2-Hexen-1-ol, (E)-	000928-95-0	Apple	ND	42.14 ± 2.97 b	48.63 ± 5.24 b	96.14 ± 2.71 d	96.14 ± 2.71 d	63.15 ± 0.62 c	16.94 ± 0.63 a	23.39 ± 2.91 a	27.63 ± 3.77 a	ND
Xinbei-chun	Nonanal	000124-19-6	Sweet Orange	6.75 ± 0.21 a	8.80 ± 0.58 ab	8.18 ± 0.51 ab	10.01 ± 0.28 b	10.01 ± 0.28 b	14.07 ± 0.35 c	10.47 ± 0.92 b	6.24 ± 0.62 a	7.89 ± 1.18 ab	ND
	Hexanoic acid, ethyl ester	000123-66-0	Fruit	ND	ND	ND	3.65 ± 0.38	3.65 ± 0.38	ND	ND	ND	ND	ND
	Butanoic acid, hexyl ester	002639-63-6	Sweet Fruit	ND	7.35 ± 0.71	ND	ND	ND	ND	ND	ND	ND	ND
	Benzene acetaldehyde	000122-78-1	Hosta Flower	47.60 ± 1.06 c	25.41 ± 2.39 b	14.88 ± 1.20 a	9.02 ± 0.56 a	9.02 ± 0.56 a	ND	ND	ND	ND	ND
	Benzene, 1,3-dimethyl-	000108-38-3	Fragrant	47.14 ± 3.02	ND	ND	ND	ND	ND	ND	ND	ND	ND
	Cis-3-Hexenyl 2-methylbutanoate	053398-85-9	OU	ND	7.40 ± 0.84	ND	ND	ND	ND	ND	ND	ND	ND
	2-Hexen-1-ol, acetate	002497-18-9	OU	ND	8.53 ± 0.70 b	ND	4.58 ± 0.12 a	4.58 ± 0.12 a	ND	ND	ND	ND	ND
	Butanoic acid, 3-hexenyl ester, (Z)-	016491-36-4	OU	15.36 ± 0.66 b	30.96 ± 3.04 c	7.89 ± 0.84 a	2.68 ± 0.09 a	2.68 ± 0.09 a	ND	ND	ND	ND	ND
	Butanoic acid, 2-hexenyl ester, (E)-	053398-83-7	OU	ND	14.99 ± 1.43 b	7.21 ± 0.93 a	5.74 ± 0.01 a	5.74 ± 0.01 a	ND	ND	ND	ND	ND
	1-Hexanol	000111-27-3	OU	ND	ND	ND	ND	ND	69.21 ± 2.71 b	47.89 ± 2.56 a	62.28 ± 6.48 ab	51.09 ± 1.14 ab	ND
	2,4-Hexadienal, (E,E)-	000142-83-6	OU	ND	ND	ND	ND	ND	ND	ND	ND	3.55 ± 0.63	ND
	Octanal	000124-13-0	OU	ND	ND	ND	ND	ND	4.27 ± 0.19	10.40 ± 0.96	ND	ND	ND
	Hexanal	000066-25-1	OU	112.88 ± 5.74 a	82.85 ± 8.11 a	162.87 ± 20.37 a	169.06 ± 7.98 a	169.06 ± 7.98 a	461.04 ± 21.82 b	429.89 ± 36.03 b	453.90 ± 39.32 b	552.82 ± 88.47 b	ND

S1–S9 represent sampled date, VE is Veraison. OU means odorless. Data are mean values ± SE of three replicates. Different letters on the same line indicate significant differences ($p < 0.01$, determined by one-way ANOVA). ND represent not detected.

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