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Evolution of green leaf volatile profile and aroma potential during the berry development in five *Vitis vinifera* L. Cultivars

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Nerol, PubChem CID: 643820
Nerol oxide, PubChem CID: 61275
Citronellol, PubChem CID: 8842
Limonene, PubChem CID: 22311
γ-geraniol, PubChem CID: 518689
Geraniol, PubChem CID: 637566
β-*cis*-ocimene, PubChem CID: 5320250
Terpinolene, PubChem CID: 11463

ABSTRACT

Green leaf volatiles (GLVs), play important roles in the green and fresh aroma characteristics of grape berries. The evolution of GLV profiles regarding the varietal difference during grapevine phenological ripening is not well understood. This study generated the GLV profiles of five *Vitis vinifera* L. cultivars (‘Cabernet Sauvignon,’ ‘Cabernet Franc,’ ‘Cabernet Gernischt,’ ‘Chardonnay,’ and ‘Sauvignon Blanc’) at five ripening stages. GLVs were distinctive at different E-L stages for each grape variety. (E)-2-hexen-1-ol, 1-hexanol, and hexanal were the dominant components in all mature berries. In terms of total GLV content, all varieties reached the maximum at maturity in the 2019 vintage, and the total GLV content was higher in mature Sauvignon Blanc and Cabernet Sauvignon grapes. In the 2020 vintage, the total GLV content in Chardonnay and Sauvignon Blanc berries rapidly accumulated at veraison and peaked before harvest. The present results could help winemakers create a good balance of wine aroma.

1. Introduction

Aroma, which is an important quality factor for grapes and wine, is composed of terpenes, C13-norisoprene, C_6/C_9, methoxypyrazine, and thiol compounds (González-Barreiro et al., 2015; Yue et al., 2022). Among these, C_6/C_9 volatiles have a high content in grapes and wine, which can reach a level of mg/L and are synthesized from precursor fatty acids (linoleic acid and linolenic acid) through the lipoxygenase pathway (Matsui, 2006). Furthermore, C_6/C_9 compounds include C_6 and C_9 aldehydes, alcohols, and esters, which are important aroma components in grapes and wine. C_6/C_9 compounds are also known as green leaf volatiles (GLVs), and GLVs can be released as signal molecules when plants face environmental stress and play a role in signal transduction (Hubert et al., 2018; Ju et al., 2021). In grapes and wine, the olfactory threshold of GLVs is lower, with green, fresh, fruity, floral, and cucumber aroma characteristics, and they are important sources of aroma in wine varieties, such as ‘Cabernet Sauvignon’ and ‘Cabernet Franc’ (Matsui, 2006; Wang et al., 2016).

Previous studies have found that the GLV content in grapes and wine is affected by viticultural practices, such as leaf removal (Moreno et al., 2017; Yue et al., 2020a), plant growth regulators (Ju et al., 2016; Yue et al., 2021; Wang et al., 2022), irrigation (Ju et al., 2018), and training systems (Xu et al., 2015). In addition, many studies have also explored the dynamic changes in volatile content during grape berry development (Luo et al., 2019; Wu et al., 2019; Yue et al., 2020b; Wang et al., 2021). Previous studies have mostly focused on a single grape variety or viticultural practice (Chen et al., 2019; Bekzod et al., 2022), and few studies have evaluated the development process of different varieties, especially the dynamic changes in GLVs. Luo et al. (2019) compared the changes in free terpenes during the maturation of ‘Shiraz,’ Cabernet Sauvignon, ‘Riesling,’ ‘Chardonnay,’ and ‘Pinot Gris.’ Another study comparing six Chinese wild spine grapes quantified 13 GLVs during the harvest period

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(Ju et al., 2021). Xu et al. (2015) reported the influence of training systems on the profiles of GLVs in Cabernet Sauvignon grapes and wine. The GLVs of different grape varieties during maturation have been largely ignored, as these compounds give wine aromas of grasses and green peppers, among others, that are generally considered unpleasant. However, this is also the source of the characteristic aroma of many red grape varieties, such as Cabernet Sauvignon, 'Cabernet Gernischt,' and Cabernet Franc, since some GLVs in wines are grape-dependent (Keyzers & Boss, 2009). Thus, the dynamic changes of GLV components during fruit development of different grape varieties need further research.

Therefore, to our knowledge, this is the first study to reveal differences in wine grape varieties that are typical of GLVs. Although GLV components exist in both free and bound forms, the free GLV content is much higher than that of the bound state (Wen et al., 2014). Here, we compared the evolution patterns of the free GLVs of five *Vitis vinifera* L. cv. varieties, namely Cabernet Sauvignon, Cabernet Gernischt, Cabernet Franc, Chardonnay, and 'Sauvignon Blanc,' during maturation to reveal the variations in grape GLV profiles during varietal and developmental stages. These results could explain the dynamic changes in GLV components during fruit development in different grape varieties and also lay a foundation for revealing the characteristic aroma formation of grapes and wine, such as Cabernet Sauvignon.

2. Materials and methods

2.1. Field conditions and materials

Five *Vitis vinifera* L. grape materials, namely Cabernet Sauvignon, Cabernet Franc, Cabernet Gernischet, Chardonnay, and Sauvignon Blanc, were sampled from a commercial vineyard in Wuwei, Gansu province, China (37.93′N, 102.63′E) in 2019 and 2020. Vineyard management practices, such as irrigation, pest control, and fertilization, followed local standards. Grapevines were planted with 0.5 × 3 m spacing. All samples were randomly collected at the following stages: EL-34 (berries begin to soften; Brix starts increasing); E-L 35 (berries begin to color and enlarge); EL-36 (berries with intermediate Brix values); E-L 37 (berries not quite ripe); and E-L 38 (berries harvest-ripe). The E-L system was developed according to Coombe et al. (1995). About 400 berries were sampled for each replicate, with 3 replicates for each variety. All samples were promptly transported to the laboratory on ice and stored at −80 °C for further analysis.

2.2. Chemicals

Hexanal, (E)-2-hexanal, 1-hexanol, (E)-2-hexenol, (E)-3-hexenol, (Z)-2-hexenol, (Z)-3-hexenol, hexyl acetate, and ethyl hexanoate standards used for identification and quantification were obtained from Sigma (≥99%, St Louis, MO, USA).

2.3. Determination of physicochemical parameters of grape berries

The grapes physicochemical parameters (berry fresh weight, total soluble solid and titratable acids) were determined as described by Yue et al. (2021). The pH value was determined following the International Organization of Vine and Wine method (OIV, 2014).

2.4. Determination of meteorological

The China Meteorological Data Service Center (CMDC) (https://data.cma.cn/en) was used to download the meteorological data, including rainfall, average, maximum, and minimum temperatures, and sunshine hours, in 2019 and 2020 (Table S1).

2.5. GC–MS analysis of the GLV compounds of grape berries

GLV compounds were extracted from grapes according to our previous method, with some modifications (Ju et al., 2018). Briefly, 50 g of frozen grape berries were placed in a mortar, deseeded, blended with 1 g polyvinylpolypyrrolidone (PVPP) and 0.5 g D-(+) gluconic acid δ-lactone, and ground under liquid nitrogen. The mixture was then macerated at 4 °C for 2 h, followed by centrifuging at 8000 rpm for 10 min. Next, 5 mL of supernatant was placed in a 15 mL sample vial, and 1.00 g of NaCl, 10 μL of internal standard (4-methyl-2-pentanol, Sigma Aldrich, MO, USA), and a magnetic rotor were added. The sample vial was placed on a magnetic stirring heating table. After heating and stirring at 40 °C for 30 min, the extraction head was inserted into the headspace part of the sample vial, and the extraction head was inserted into the gas chromatography injection port after adsorption for 30 min.

An Agilent 7890B GC equipped with an Agilent MS, and an HP-INNOWAX (60 m × 0.25 mm × 0.25 μm) column were used to analyze the GLV compounds. The helium flow rate was 1 mL/min (carrier gas). The MS inlet temperature was 250 °C. The heating program was as follows: 50 °C for 1 min, which increased to 220 °C at a rate of 3 °C/min and retained at 220 °C for 5 min. Mass spectrometry conditions were as follows: ionization mode of the mass spectrometer was EI, ion source temperature was 250 °C, electron energy was 70 ev, and the mass scan range was 30–350 u. Each sample was tested in triplicate.

The GLV compounds were identified by the National Institute of Standards and Technology Library (NIST 11) and the retention times of the authentic standards. Quantitative analysis was carried out using the standard curve of the standards.

2.6. Odor activity values and aroma series

The contribution of GLV compounds to the grapes' characteristic aromas was quantitatively evaluated by OAV. OAV was calculated by the ratio of the concentration of a molecule to its odor threshold. These aroma thresholds and descriptive vocabulary for aroma compounds have been referenced in published studies (Rosen et al., 1963; ; Masanetz & Grosch, 1998; Tamura et al., 2001; Czerny et al., 2008; Wen et al., 2014).

2.7. Statistical analysis

All experimental data were statistically analyzed using SPSS 20.0 (IBM, Armonk, NY, USA). Analysis of variance (ANOVA) and Duncan's test were performed for statistical treatment ($P < 0.05$). Data were presented as the mean ± standard deviation in tables and figures. Principal component analyses and line graphs were performed using OriginPro 2022 (OriginLab, Massachusetts, USA). Heat maps of the GLV concentrations were drawn using R (v3.6.1) software.

3. Results and discussion

3.1. Basic physical and chemical indexes of berries

We detected physicochemical parameters in the berries of five *Vitis vinifera* L. cultivars at harvest (Table S2). The fresh berry weights of Cabernet Gernischt and Sauvignon Blanc grapes were higher than the other three grape varieties. The TSS content in grapes was higher in Cabernet Franc in two years. The maturity index of grapes ranged from 3.18 to 5.19 in 2019 and from 3.00 to 8.90 in 2020. Except for Cabernet Gernischt and Sauvignon Blanc, the titratable acidity content of the other three grape varieties in 2020 was significantly lower than in 2019.

3.2. GLV evolution pattern recognition

Five *Vitis vinifera* L. berry samples were collected using the E-L system to enable comparisons between berries within the same vintages (Coombe, 1995). GLVs are primarily synthesized by the lipoxygenase-hydroperoxide lyase (LOX-HPL) pathway and are regulated by viticultural and environmental factors (Wang et al., 2019). The genetic profile

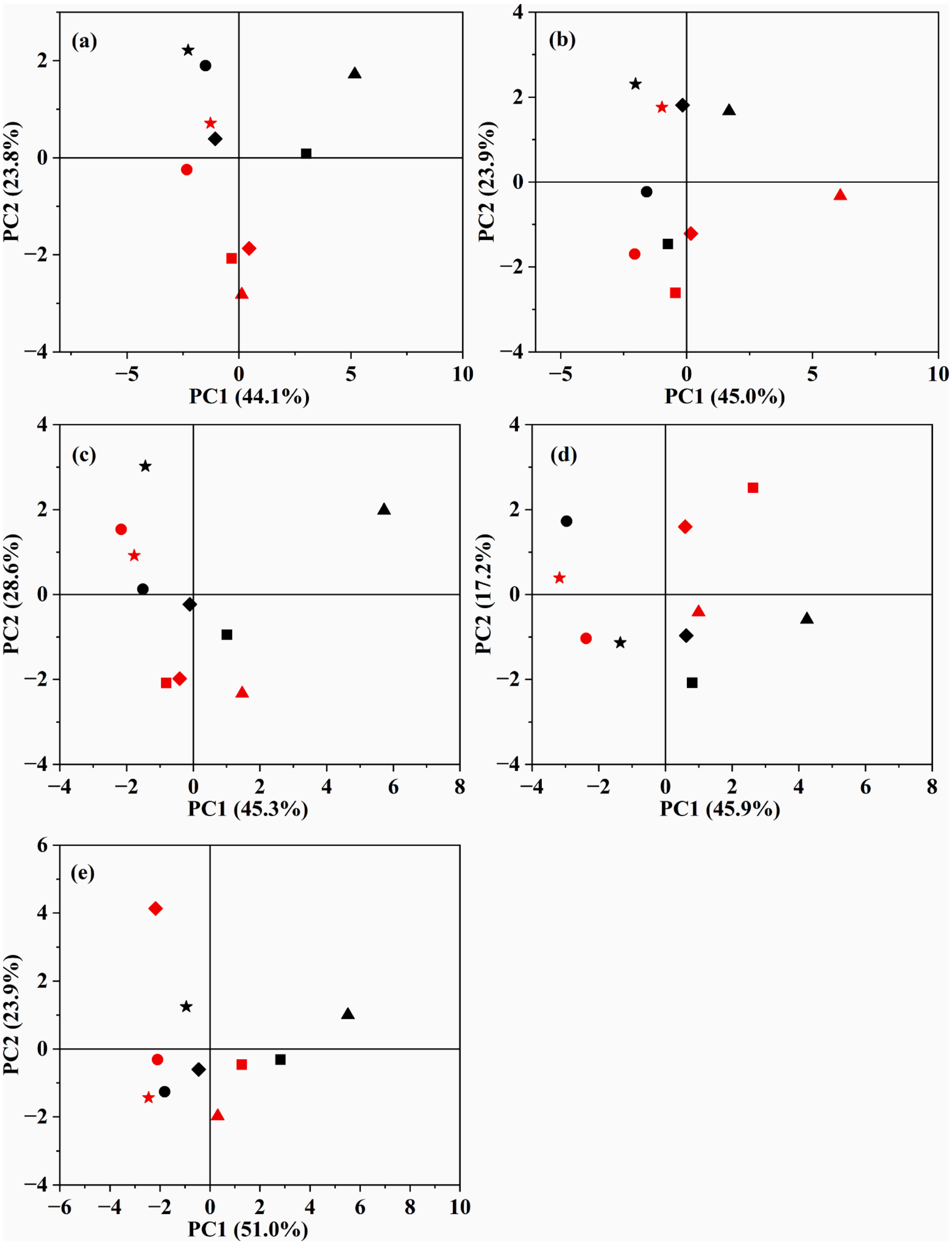

Fig. 1. Principal component analysis biplot illustrating the pattern of green leaf volatiles (GLVs) production at different stages of the berry maturation in vintage 2019 (black labels) and vintage 2020 (red labels). (a) Cabernet Sauvignon, (b) Cabernet Franc, (c) Cabernet Gernischt, (d) Chardonnay and (e) Sauvignon Blanc. Legend: ●-E-L 34, ★-E-L 35, ◆-E-L 36, ■-E-L 37, ▲-E-L 38. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

of the grape varieties was a more important factor affecting the synthesis of GLVs (Ju et al., 2018). A previous study illustrated that the C_6 volatiles were discriminatory among different table grape varieties (Qian et al., 2017). Few studies have indicated the evolution of GLV profiles among a broad selection of white and red wine varieties. Therefore, the GLV profiles of five wine grape varieties over two years were subjected to principal component analysis (Fig. 1).

The first two principal components explained 63.4% and 66% of the variation in the GLV profiles for vintages 2019 and 2020, respectively (Fig. S1). Principal component analysis illustrated that the GLV components of the five cultivars were not clearly distinctive. Principal component analysis was also performed to investigate the variations in GLV components during berry ripening in individual cultivars (Fig. 1a–e). For all varieties, the first two functions (>60%) explained most of the variance. Principal component analysis showed that the GLV components in the five cultivars were clearly differentiated at different developmental stages. Additionally, the GLV profiles of berries in the early development stage (E-L 34) showed differences from those at harvest in the five cultivars. A previous study on table cultivars also suggested that grapes had discriminated C_6 volatile content at the early ripening stage and commercial harvest (Qian et al., 2017), similar to our results.

3.3. Total green leaf volatile evolution

GLVs are mostly made up of C_6/C_9 short chain fatty aldehydes, alcohols, and esters, which are produced by unsaturated fatty acids catalyzed by lipoxygenase, lipohydroperoxide lyase, and alcohol dehydrogenase (Ju et al., 2021). In grapes, GLVs contribute to herbaceous, citrus, leaf, and green sensory characteristics. In addition, GLVs are direct precursors of hexyl acetate, one of the fruity components; thus, they make an important contribution to the aroma quality of wine

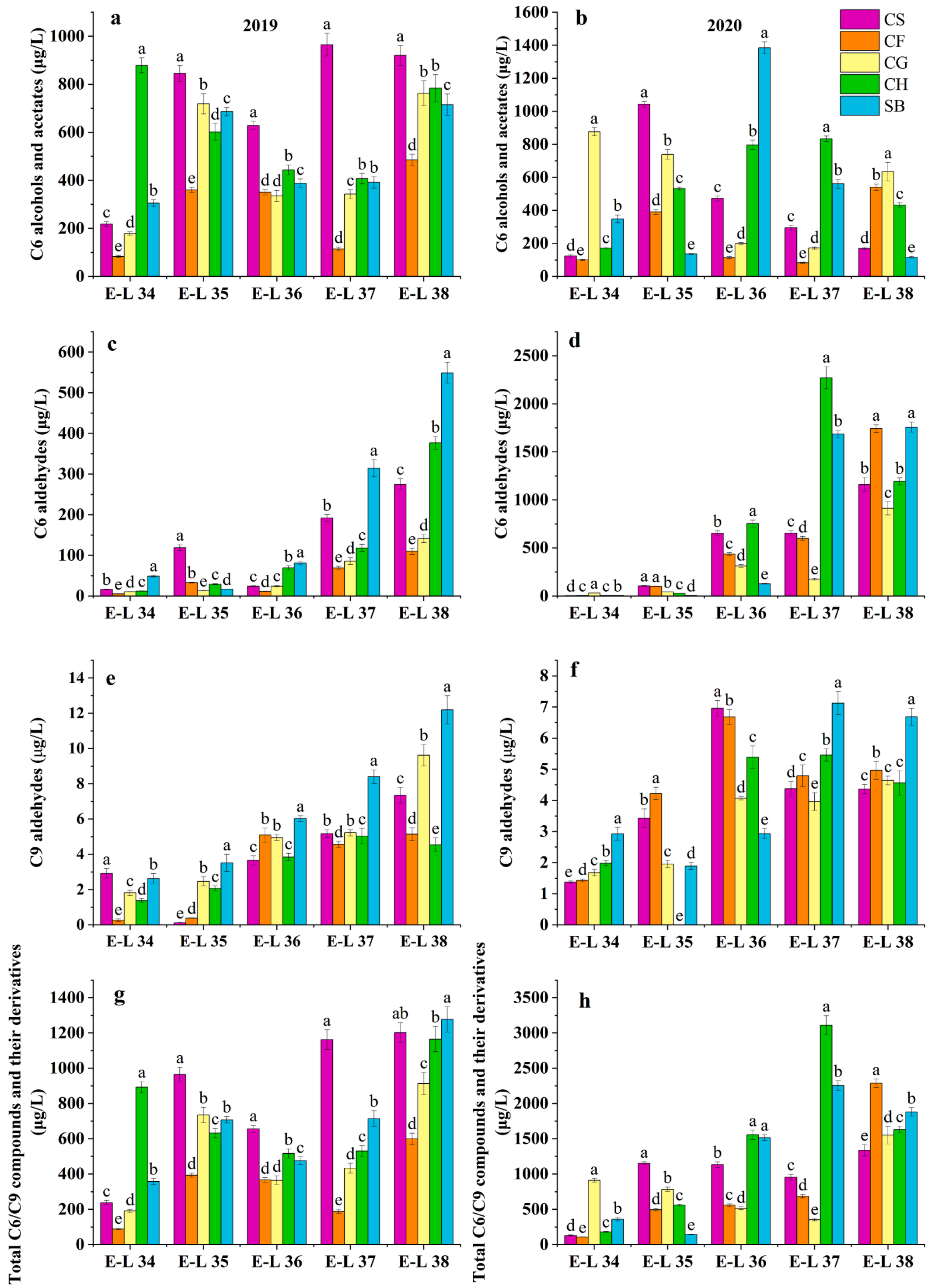

Fig. 2. Evolution of green leaf volatiles (GLVs) in five varieties of grapes in vintage 2019 and vintage 2020. CS, Cabernet Sauvignon; CF, Cabernet Franc; CG, Cabernet Gernischt; CH, Chardonnay; SB, Sauvignon Blanc. Data are expressed as mean ± standard error (n = 3). Different letters with each stage indicate the differences based on Duncan's test at $p < 0.05$. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

grapes and wine (Xu et al., 2015).

The evolution of total GLVs in the five cultivars is shown in Fig. 2. C_6 aldehydes, alcohols, and acetates are prevalent in berries due to their herbaceous and green flavors, and their accumulation patterns are visualized in Fig. 3a–d. The total C_6 alcohol and acetate content in grapes increased by 321.93% (Cabernet Sauvignon), 483.95% (Cabernet Franc), 328.04% (Cabernet Gernischt), and 134.25% (Sauvignon Blanc) during ripening in 2019, while the content decreased by 10.82% in Chardonnay grapes (Fig. 2a). The total C_6 alcohol and acetate content in Chardonnay grapes peaked at E-L 34, continued to decrease before harvest, and increased significantly at maturity. The other four varieties showed a trend of first increasing and then decreasing from E-L 34 to E-L 36. The total C_6 alcohol and acetate content in Cabernet Sauvignon grapes reached a maximum at E-L 37 (964.91 μg/L), the content of

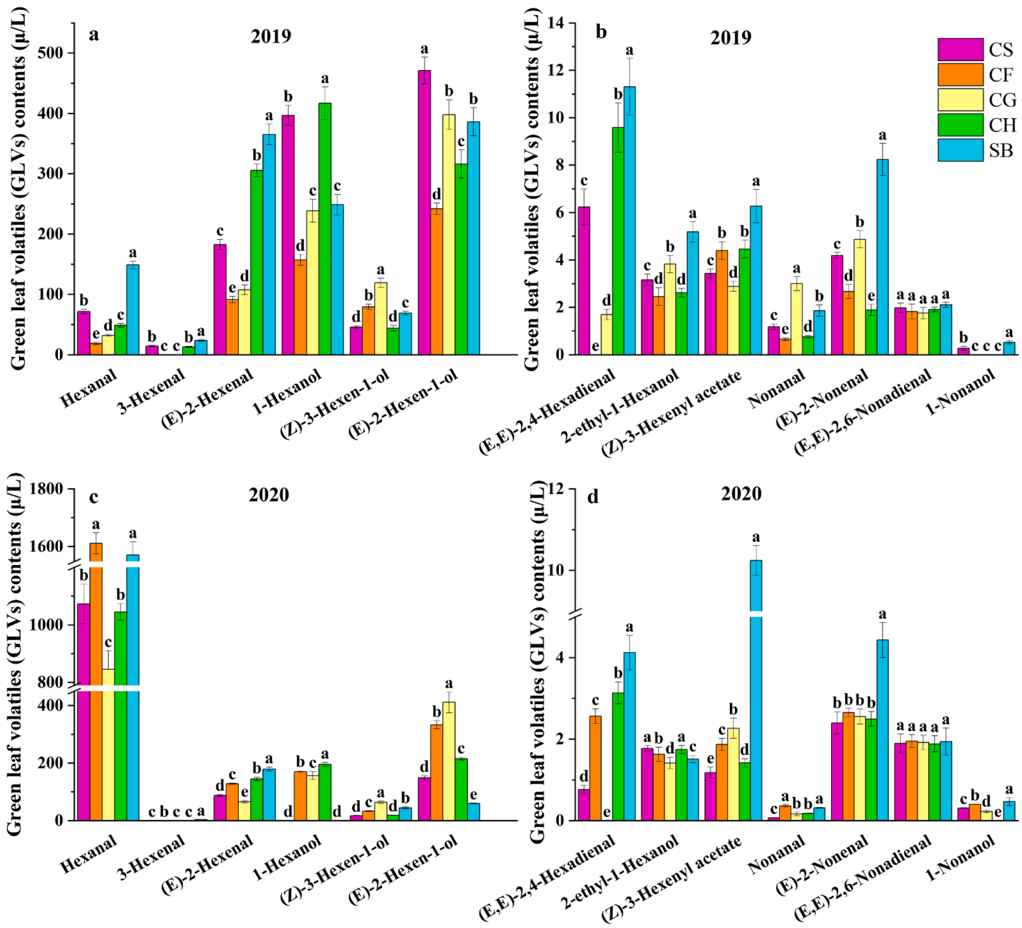

Fig. 3. Concentrations of green leaf volatiles (GLVs) in five grape cultivars at harvest in vintage 2019 and vintage 2020. CS, Cabernet Sauvignon; CF, Cabernet Franc; CG, Cabernet Gernischt; CH, Chardonnay; SB, Sauvignon Blanc. Data are expressed as mean ± standard error (n = 3). Different letters with each stage indicate the differences based on Duncan's test at $p < 0.05$. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

which peaked at harvest in the other three varieties. Except for the early development stage (E-L 34), the total C_6 alcohol and acetate content was highest in Cabernet Sauvignon grapes. Cabernet Franc grapes had a lower C_6 alcohol and acetate content during the ripening process. At harvest, there was no obvious difference in C_6 alcohol and acetate content between Cabernet Gernischt (762.32 μg/L) and Chardonnay (783.62 μg/L) berries, the content of which in Sauvignon Blanc was 715.16 μg/L (Fig. 2a). In 2020, the total C_6 alcohol and acetate content in grapes increased by 37.3% (Cabernet Sauvignon), 442.24% (Cabernet Franc), and 152.31% (Chardonnay) during ripening. Compared to the early development stage (E-L 34), the total C_6 alcohol and acetate content in Cabernet Gernischt and Sauvignon Blanc ripe berries was reduced by 27.52% and 66.72% in 2020 (Fig. 2b). The accumulation pattern of total C_6 alcohols and acetates in the five grape varieties differed from the 2019 year, which might be related to climate differences (Luo et al., 2019). The total C_6 alcohol and acetate content of Cabernet Sauvignon grapes was highest at E-L 34, and its content was significantly higher than that of other varieties during this period. At harvest, Cabernet Gernischt berries had the highest total C_6 alcohol and acetate content, followed by Cabernet Franc and Cabernet Franc grapes (Fig. 2b).

Regarding the total C_6 aldehyde content, all varieties had a low content at the early ripening stage (E-L 34) and reached a maximum at maturity in the 2019 vintage. Sauvignon Blanc grapes had the highest total C_6 aldehyde content, from E-L 36 to E-L 38, and Cabernet Franc and Cabernet Gernischt grapes had a low content during the same period (Fig. 2c). The accumulation pattern of total C_6 aldehydes in grapes of the 2020 vintage was similar to that of the 2019 vintage, whose content was low in the early development stage and accumulated gradually with maturity. At harvest, the total C_6 aldehyde content in the five grape varieties ranged from 911.54 to 1757.45 μg/L in 2020. The total C_6 aldehyde content of the grape varieties tested in 2020 was significantly higher than that in the 2019 harvest period (Fig. 2d). Consistent with our results, Wang et al. (2019) reported that the concentrations of C_6 aldehydes largely differed across seasons.

C_9 compounds mainly existed in the form of aldehydes in grapes, including (E)-2-nonanal, nonanal, and (E,Z)-2,6-nonadienal. Although the C_9 aldehyde content is low, they can contribute citrus and cucumber aromas to grapes due to low threshold (Wang et al., 2019). Cabernet Sauvignon grapes had the highest C_9 aldehyde content among all varieties at E-L 34 of the 2019 vintage, the content of which was the lowest in Cabernet Franc berries. From E-L 35 to maturity, the total C_9 aldehyde content was most abundant in Sauvignon Blanc grapes compared to other cultivars. Except for a slight reduction in Chardonnay total C_9 aldehyde content at maturity, the other cultivars peaked at harvest (Fig. 3e). Similar to the 2019 vintage, the total C_9 aldehyde content was the highest in Sauvignon Blanc grapes from E-L 37 to maturity. Cabernet Sauvignon and Cabernet Franc grapes reached the maximum total C_9 aldehyde content at E-L 36 and had a slight reduction at maturity (Fig. 2f).

In terms of total GLV content, all varieties reached the maximum at maturity in the 2019 vintage, and their content in the five cultivars ranged from 599.94 to 1276.84 μg/L. Of the grapes, Cabernet Sauvignon grapes had the highest total GLV content during the veraison stage (from E-L 35 to E-L 37), and Cabernet Franc had the lowest content during the same period. At harvest, the total GLV content was higher in the Sauvignon Blanc and Cabernet Sauvignon grapes, followed by Chardonnay, Cabernet Gernischt, and Cabernet Franc (Fig. 3g). In the 2020 vintage, the total GLV content in all berries was between 1334.90 and 2287.25 μg/L, an increase of 11.03–281.25% compared to that in the 2019

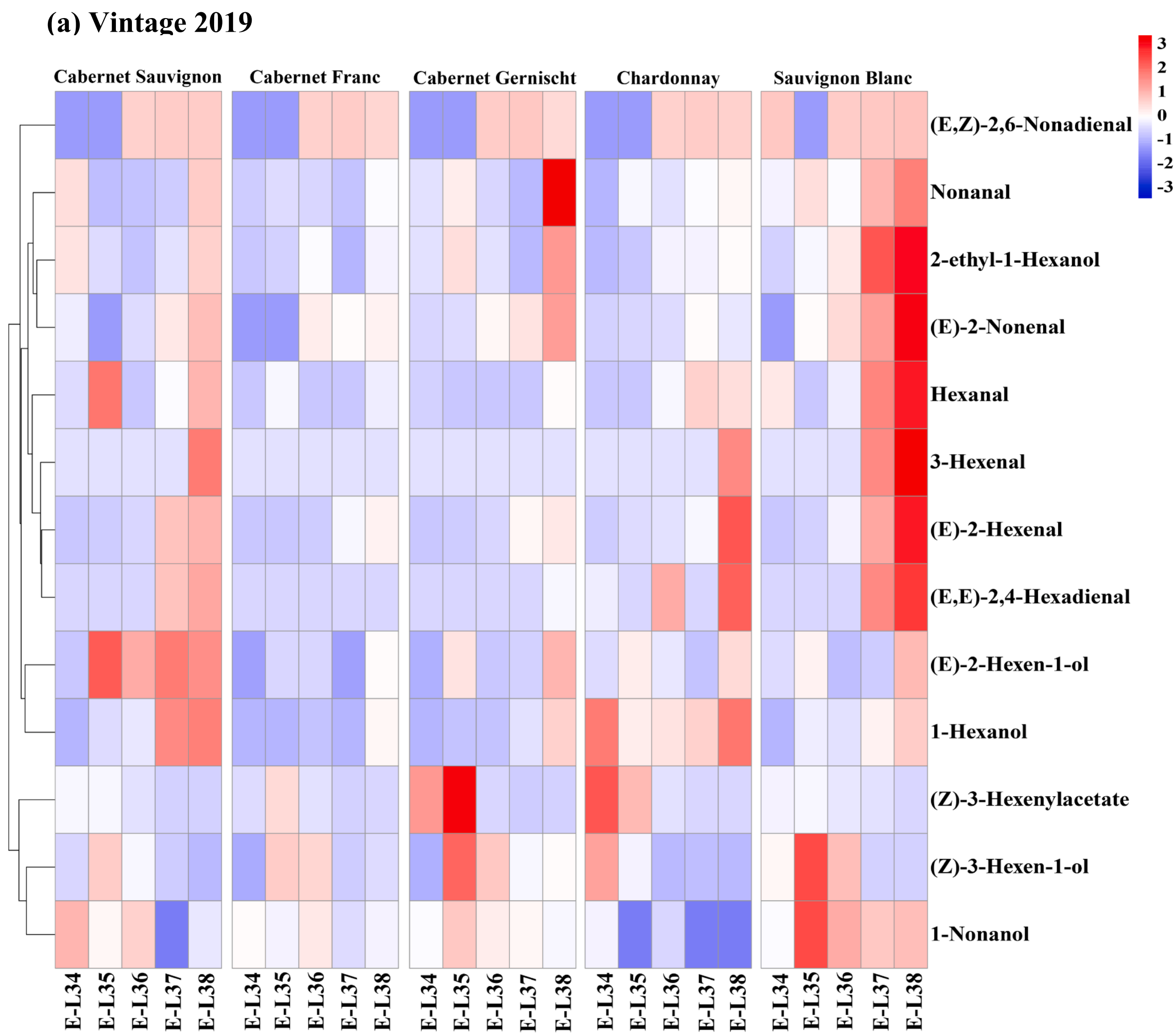

Fig. 4. Evolution heatmaps of quantified green leaf volatiles (GLVs) in five varieties of grapes in (a) vintage 2019 and (b) vintage 2020. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

vintage. Cabernet Gernischt grapes had a higher total GLV content at the early ripening stage (E-L 34 and E-L 35). The total GLV content in Chardonnay and Sauvignon Blanc berries rapidly accumulated at veraison and peaked before harvest, with an obvious reduction at maturity (Fig. 2h).

In light of the above, the pattern of total GLV evolution differed among varieties and was also affected by season. Consistent with our results, Ju et al. (2018) and Wang et al. (2019) reported that the variation patterns of GLVs were affected by the vintage. Alterations in the microclimate between two seasons, such as sunshine and rainfall, might influence the expression of genes in the GLV synthetic pathway, which further affects the activity of GLV synthesis-related enzymes (Qian et al., 2017). Moreover, variety is regarded as the key factor influencing grape aroma. Qian et al. (2017) indicated that there were significant differences in terms of GLV content among seven table grape cultivars. Ju et al. (2021) clarified that the varietal differences in GLV compounds in six Chinese wild spine grapes were consistent with the expression of some synthesis-related genes. So far, few studies have focused on the variation in GLV volatiles with grassy or green odors during the ripening of different wine grape varieties (Godshaw et al., 2019; Luo et al., 2019). Combined with previous studies, we concluded that GLV volatiles were also important components of varietal typicality.

3.4. Green leaf volatile profiles in grapes during berry development

We detected 13 GLV components in five grape cultivars at harvest, including four C_6 aldehydes, four C_6 alcohols, one C_6 esters, three C_9 aldehydes, and one C_9 alcohol (Fig. 3). Among the GLV profiles, (E)-2-hexen-1-ol and 1-hexanol were the dominant components in all mature berries from the 2019 vintage; the former contributed to 27.13–43.57% of the total GLV content, and the latter accounted for 19.48–35.76% (Fig. 3a). Of the 2020 vintage, hexanal was the most abundant individual component in all mature grapes, and its content ranged from 845.73 to 1610.95 μg/L (Fig. 3b).

Regarding the C_6 alcohols, 1-hexanol, (Z)-3-hexen-1-ol, (E)-2-hexen-1-ol, and 2-ethyl-1-hexanol existed in all grapes for two vintages (except for 1-hexanol in 2020) at harvest (Fig. 3). The total C_6 alcohol levels in 2019 mature Cabernet Sauvignon grapes (916.61 μg/L) were the highest, and the lowest was detected in the Sauvignon Blanc cultivar (105.81 μg/L) at harvest in 2020. Of the Cabernet Franc, Cabernet Gernischt, and Chardonnay varieties, Cabernet Gernischt grapes had the highest total C_6 alcohol content in both vintages. Except for Cabernet Franc, the 2020 ripened grape cultivars possessed a lower total C_6 alcohol content than those harvested in 2019. In grapes, 1-hexanol contributes to fruity, flowery, grassy, and green sensory characteristics (Wen et al., 2014). 1-Hexanol was not detected in mature Cabernet Sauvignon and Sauvignon

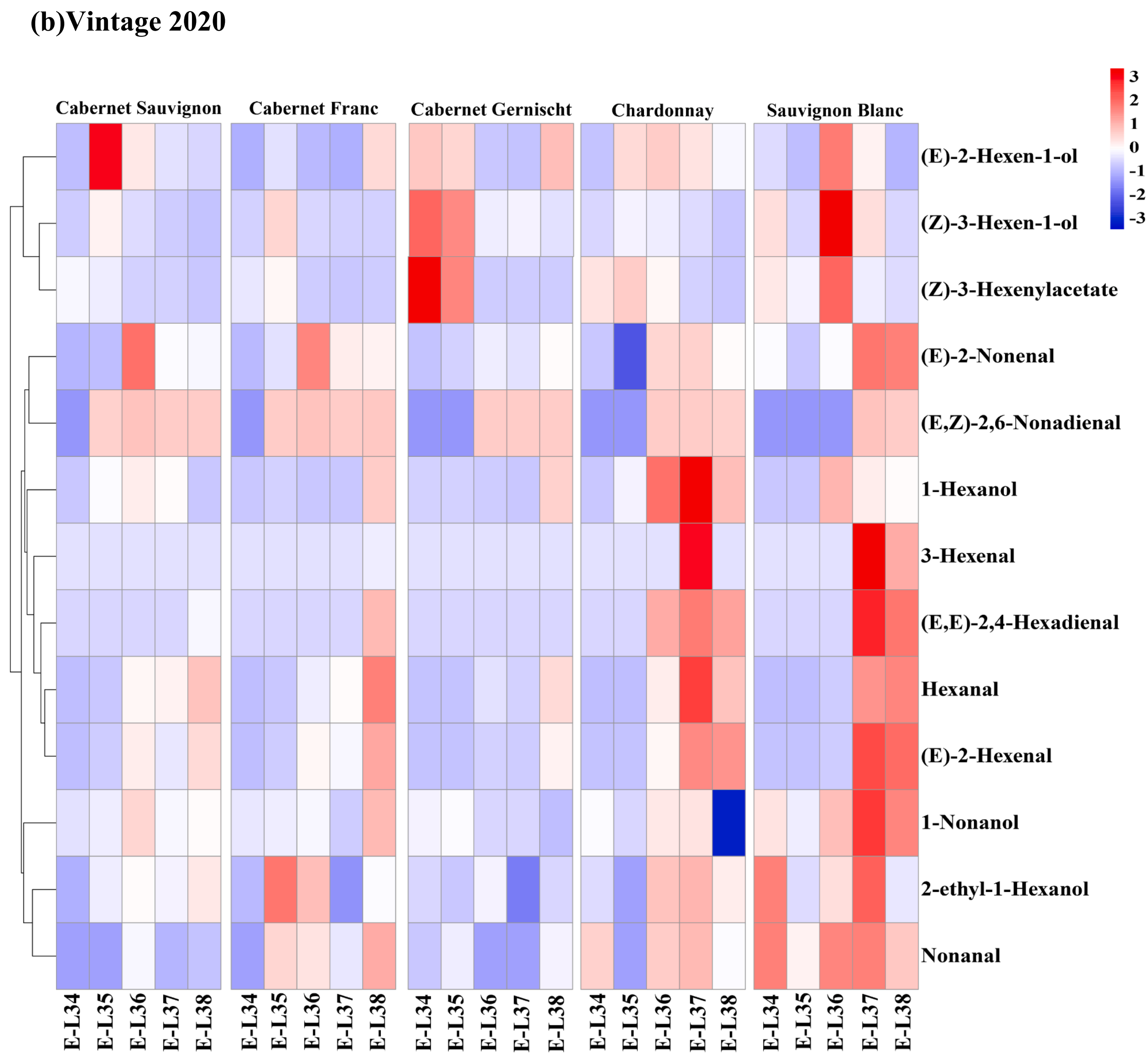

Fig. 4. (*continued*).

Blanc berries of the 2020 vintage, and Chardonnay exhibited the highest content of 1-hexanol in both vintages. (E)-2-hexen-1-ol contributes fruity and ethereal flavors to berries, which is generated by (E)-2-hexenal under the catalysis of alcohol dehydrogenase (ADH) in the LOX-HPL pathway (Kalua & Boss, 2010). Among C_6 alcohol profiles, (E)-2-hexen-1-ol accounted for 40.57–88.78% of all grapes for two vintages. Similar to total C_6 alcohols, among all cultivars, Cabernet Sauvignon grapes had the highest (E)-2-hexen-1-ol content (471.01 μg/L) in 2019, and Sauvignon Blanc berries had the lowest (E)-2-hexen-1-ol content (59.85 μg/L) in 2020 during the harvest period.

In terms of the C_6 aldehydes, we detected four individual C_6 aldehydes in mature berries: hexanal, 3-hexenal, (E)-2-hexenal, and (E,E)-2,4-hexadienal. (E)-2-hexenal and hexanal were the dominant C_6 aldehydes, and these two aldehydes were detected in all grape cultivars at harvest, consistent with the results of previous studies on table grape varieties (Qian et al., 2017). (E)-2-hexenal and hexanal affect the overall aroma of wine grapes, which contribute to green, apple-like, and fruity flavors (Wang et al., 2019). The concentration of (E)-2-hexenal in all mature berries ranged between 65.81 and 365.28 μg/L in two years, and its content was the highest in Sauvignon Blanc berries in both the 2019 and 2020 vintages, followed by Chardonnay. In all mature grapes, hexanal was more abundant in 2020 than in 2019. Consistent with our results, Xu et al. (2015) also implied that the generation of hexanal was affected by the grape-growing environment.

(Z)-3-hexenyl acetate was the only C_6 ester found in five grape cultivars at harvest and exhibited a low concentration in berries (1.18–10.24 μg/L). Except for Sauvignon Blanc (10.24 μg/L) in 2020, (Z)-3-hexenyl acetate was below that of other grape cultivars. Qian et al. (2017) also indicated that (Z)-3-hexenyl acetate contributes a limited aroma to grapes.

Regarding the C_9 compounds, nonanal, (E)-2-nonenal, and (E,E)-2,6-nonadienal were detected in all mature berries, while 1-nonanol did not exist in mature Chardonnay in the 2019 and 2020 vintages. 1-Nonanol had the highest concentrations in mature Sauvignon Blanc grapes (Fig. 3b). The (E)-2-nonenal and (E,E)-2,6-nonadienal ranged from 1.88 to 8.24 μg/L and 1.76 to 2.10 μg/L, respectively, which were clearly higher than their odor thresholds (0.08 and 0.02 μg/L, respectively) (Wen et al., 2014). These results indicate that their citrus, greasy, and cucumber flavors could significantly influence wine grapes' overall aroma (Wang et al., 2019). Among these ripened berries, Sauvignon Blanc possessed the highest level of (E)-2-nonenal in both the 2019 and 2020 vintages. There were no significant differences in the content of (E,

Table 1
Odour thresholds (OT), odour description, Odour activity values (OAVs) of green leaf volatiles (GLVs) in five varieties of grapes at different stages of the berry maturation in vintage 2019 and vintage 2020.

Compounds	OT[a] (ppb in water)	Odour description	Aromatic series	2019 Cabernet Sauvignon OAV[b]					2020 Cabernet Sauvignon OAV[b]				
				E-L 34	E-L 35	E-L 36	E-L 37	E-L 38	E-L 34	E-L 35	E-L 36	E-L 37	E-L 38
Hexanal	4.5	Green, grassy, fruity	Green, fruity	2.40	23.46	nd	6.19	15.92	nd	19.58	130.25	137.68	238.39
3-Hexenal	0.21	Green, grassy	Green	nd	nd	nd	nd	68.45	nd	nd	nd	nd	nd
(E)-2-Hexenal	17	Green, grassy	Green	0.33	0.80	1.42	9.35	10.73	0.24	0.98	3.95	2.03	5.14
1-Hexanol	150	Green, fruity, flowery, grassy	Green, fruity	nd	0.53	0.65	2.50	2.64	nd	0.55	0.73	0.59	nd
(Z)-3-Hexen-1-ol	13	Green, lettuce-like, grassy	Green	5.86	12.97	8.10	5.00	3.52	2.22	9.59	4.30	2.12	1.32
(E)-2-Hexen-1-ol	100	Fruity,ethereal	Fruity	1.21	5.76	4.13	5.19	4.71	0.74	8.18	3.02	1.73	1.49
(E,E)-2,4-Hexadienal	10	Green, sweet, fruity	Green, fruity	nd	nd	nd	0.50	0.62	nd	nd	nd	nd	0.08
(Z)-3-Hexenyl acetate	8	Green; banana-like	Fruity	2.28	2.31	1.23	0.37	0.43	2.45	1.99	0.50	0.51	0.15
2-ethyl-1-Hexanol	270	Fruity, ethereal	Fruity	0.01	0.01	0.01	0.01	0.01	nd	0.01	0.01	0.01	0.01
Nonanal	1	Aldehyde, citrus, orange peel	Citrusy	0.97	0.12	0.15	0.27	1.18	nd	nd	0.18	0.04	0.07
(E)-2-Nonenal	0.08	Cucumber, green	Green	24.27	nd	20.27	36.80	52.38	17.18	19.51	58.96	30.43	29.96
(E,Z)-2,6-Nonadienal	0.02	Green, fresh cucumber	Green, fruity	nd	nd	93.97	97.95	98.70	nd	93.29	103.20	95.18	94.77
1-Nonanol	34	Fruity, soapy	Fruity	0.02	0.01	0.01	nd	0.01	0.01	0.01	0.01	0.01	0.01

Compounds	OT[a] (ppb in water)	Odour description	Aromatic series	2019 Cabernet Franc OAV[b]					2020 Cabernet Franc OAV[b]				
				E-L 34	E-L 35	E-L 36	E-L 37	E-L 38	E-L 34	E-L 35	E-L 36	E-L 37	E-L 38
Hexanal	4.5	Green, grassy, fruity	Green, fruity	nd	5.77	nd	nd	4.16	0.00	18.92	83.88	122.02	357.99
3-Hexenal	0.21	Green, grassy	Green	nd	nd	nd	nd	nd	nd	nd	nd	nd	1.61
(E)-2-Hexenal	17	Green, grassy	Green	0.31	0.40	0.65	4.04	5.37	0.26	0.85	3.48	2.85	7.54
1-Hexanol	150	Green, fruity, flowery, grassy	Green, fruity	nd	nd	0.17	nd	1.05	nd	0.11	nd	nd	1.13
(Z)-3-Hexen-1-ol	13	Green, lettuce-like, grassy	Green	2.11	12.99	12.23	4.89	6.10	2.95	13.04	3.27	2.77	2.54
(E)-2-Hexen-1-ol	100	Fruity,ethereal	Fruity	0.46	1.50	1.53	0.46	2.42	0.46	1.76	0.66	0.44	3.33
(E,E)-2,4-Hexadienal	10	Green, sweet, fruity	Green, fruity	nd	nd	nd	nd	nd	nd	nd	nd	0.00	0.26

Compounds	OT[a] (ppb in water)	Odour description	Aromatic series	2019 Cabernet Franc OAV[b]					2020 Cabernet Franc OAV[b]				
				E-L 34	E-L 35	E-L 36	E-L 37	E-L 38	E-L 34	E-L 35	E-L 36	E-L 37	E-L 38
(Z)-3-Hexenyl acetate	8	Green; banana-like	Fruity	1.00	4.81	1.34	0.42	0.55	1.70	3.32	0.41	0.19	0.23
2-ethyl-1-Hexanol	270	Fruity, ethereal	Fruity	0.01	0.01	0.01	0.01	0.01	nd	0.01	0.01	nd	0.01
Nonanal	1	Aldehyde, citrus, orange peel	Citrusy	0.26	0.38	0.33	0.17	0.65	nd	0.28	0.24	0.14	0.36
(E)-2-Nonenal	0.08	Cucumber, green	Green	nd	nd	35.85	30.99	33.34	17.90	25.59	54.99	34.15	33.12
(E,Z)-2,6-Nonadienal	0.02	Green, fresh cucumber	Green, fruity	nd	nd	94.43	95.57	91.07	nd	94.82	101.96	95.85	97.58
1-Nonanol	34	Fruity, soapy	Fruity	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01

Compounds	OT[a] (ppb in water)	Odour description	Aromatic series	2019 Cabernet Gernischt OAV[b]					2020 Cabernet Gernischt OAV[b]				
				E-L 34	E-L 35	E-L 36	E-L 37	E-L 38	E-L 34	E-L 35	E-L 36	E-L 37	E-L 38
Hexanal	4.5	Green, grassy, fruity	Green, fruity	1.00	nd	nd	0.00	7.11	5.65	7.95	64.78	35.21	187.94
3-Hexenal	0.21	Green, grassy	Green	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
(E)-2-Hexenal	17	Green, grassy	Green	0.34	0.75	1.42	5.05	6.33	0.40	0.42	1.22	0.92	3.87
1-Hexanol	150	Green, fruity, flowery, grassy	Green, fruity	nd	0.18	0.19	0.59	1.59	0.13	0.14	0.08	nd	1.04
(Z)-3-Hexen-1-ol	13	Green, lettuce-like, grassy	Green	2.73	20.89	13.53	8.13	9.17	26.01	21.97	5.88	6.23	4.89
(E)-2-Hexen-1-ol	100	Fruity,ethereal	Fruity	0.73	2.94	1.23	1.44	3.98	3.88	3.54	1.04	0.88	4.11

(*continued on next page*)

Table 1 (*continued*)

Compounds	OT[a] (ppb in water)	Odour description	Aromatic series	2019 Cabernet Sauvignon OAV[b]					2020 Cabernet Sauvignon OAV[b]				
				E-L 34	E-L 35	E-L 36	E-L 37	E-L 38	E-L 34	E-L 35	E-L 36	E-L 37	E-L 38
(E,E)-2,4-Hexadienal	10	Green, sweet, fruity	Green, fruity	nd	nd	nd	nd	0.17	0.00	nd	nd	nd	nd
(Z)-3-Hexenyl acetate	8	Green; banana-like	Fruity	8.38	15.45	0.57	0.21	0.36	16.06	9.59	0.34	0.26	0.28
2-ethyl-1-Hexanol	270	Fruity, ethereal	Fruity	0.01	0.01	0.01	0.01	0.01	0.01	nd	0.01	nd	0.01
Nonanal	1	Aldehyde, citrus, orange peel	Citrusy	0.45	0.88	0.36	0.07	3.00	0.08	0.15	nd	nd	0.16
(E)-2-Nonenal	0.08	Cucumber, green	Green	17.13	19.88	33.04	39.05	60.83	19.96	22.50	27.26	25.86	31.92
(E,Z)-2,6-Nonadienal	0.02	Green, fresh cucumber	Green, fruity	nd	nd	96.75	101.35	87.85	0.00	0.00	94.38	94.84	96.05
1-Nonanol	34	Fruity, soapy	Fruity	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01

Compounds	OT[a] (ppb in water)	Odour description	Aromatic series	2019 Chardonnay OAV[b]					2020 Chardonnay OAV[b]				
				E-L 34	E-L 35	E-L 36	E-L 37	E-L 38	E-L 34	E-L 35	E-L 36	E-L 37	E-L 38
Hexanal	4.5	Green, grassy, fruity	Green, fruity	nd	nd	5.45	12.28	10.84	nd	4.31	153.16	468.11	232.22
3-Hexenal	0.21	Green, grassy	Green	nd	nd	nd	nd	61.88	nd	nd	nd	38.56	nd
(E)-2-Hexenal	17	Green, grassy	Green	0.64	1.70	2.28	3.69	17.97	0.32	0.40	3.66	8.96	8.52
1-Hexanol	150	Green, fruity, flowery, grassy	Green, fruity	2.70	1.15	1.32	1.57	2.78	nd	0.45	2.07	3.10	1.30
(Z)-3-Hexen-1-ol	13	Green, lettuce-like, grassy	Green	16.01	7.83	3.59	3.69	3.37	3.56	6.19	6.05	3.75	1.43
(E)-2-Hexen-1-ol	100	Fruity,ethereal	Fruity	1.69	2.70	1.87	1.17	3.16	0.89	3.35	3.79	3.12	2.15
(E,E)-2,4-Hexadienal	10	Green, sweet, fruity	Green, fruity	0.11	nd	0.60	nd	0.96	nd	nd	0.28	0.41	0.31
(Z)-3-Hexenyl acetate	8	Green; banana-like	Fruity	11.85	6.86	1.09	0.54	0.56	4.33	5.89	3.29	0.68	0.18
2-ethyl-1-Hexanol	270	Fruity, ethereal	Fruity	0.01	0.01	0.01	0.01	0.01	0.01	nd	0.01	0.01	0.01
Nonanal	1	Aldehyde, citrus, orange peel	Citrusy	0.03	0.64	0.43	0.65	0.76	0.29	nd	0.30	0.35	0.18
(E)-2-Nonenal	0.08	Cucumber, green	Green	16.83	17.74	19.35	30.47	23.54	21.11	nd	39.48	40.07	31.19
(E,Z)-2,6-Nonadienal	0.02	Green, fresh cucumber	Green, fruity	nd	nd	93.00	96.84	95.23	nd	nd	96.55	95.09	94.21
1-Nonanol	34	Fruity, soapy	Fruity	0.01	nd	0.01	nd	nd	0.01	0.01	0.01	0.01	nd

Compounds	OT[a] (ppb in water)	Odour description	Aromatic series	2019 Sauvignon Blanc OAV[b]					2020 Sauvignon Blanc OAV[b]				
				E-L 34	E-L 35	E-L 36	E-L 37	E-L 38	E-L 34	E-L 35	E-L 36	E-L 37	E-L 38
Hexanal	4.5	Green, grassy, fruity	Green, fruity	9.39	nd	4.38	21.85	33.06	nd	nd	24.61	325.88	349.04
3-Hexenal	0.21	Green, grassy	Green	nd	nd	nd	62.44	112.33	nd	nd	nd	43.04	16.78
(E)-2-Hexenal	17	Green, grassy	Green	0.39	0.99	3.60	11.48	21.49	0.33	0.30	0.95	11.98	10.54
1-Hexanol	150	Green, fruity, flowery, grassy	Green, fruity	nd	0.69	0.57	1.12	1.66	nd	nd	1.41	0.77	0.58
(Z)-3-Hexen-1-ol	13	Green, lettuce-like, grassy	Green	9.53	23.16	14.10	5.43	5.31	11.59	3.18	37.99	11.99	3.42
(E)-2-Hexen-1-ol	100	Fruity,ethereal	Fruity	1.63	2.61	1.04	1.39	3.86	1.62	0.74	5.86	2.71	0.60
(E,E)-2,4-Hexadienal	10	Green, sweet, fruity	Green, fruity	nd	nd	nd	0.76	1.13	nd	nd	nd	0.57	0.41
(Z)-3-Hexenyl acetate	8	Green; banana-like	Fruity	2.06	2.31	1.39	1.11	0.78	4.25	2.39	11.36	2.01	1.28

Compounds	OT[a] (ppb in water)	Odour description	Aromatic series	2019 Sauvignon Blanc OAV[b]					2020 Sauvignon Blanc OAV[b]				
				E-L 34	E-L 35	E-L 36	E-L 37	E-L 38	E-L 34	E-L 35	E-L 36	E-L 37	E-L 38
2-ethyl-1-Hexanol	270	Fruity, ethereal	Fruity	0.01	0.01	0.01	0.02	0.02	0.01	0.01	0.01	0.01	0.01
Nonanal	1	Aldehyde, citrus, orange peel	Citrusy	0.59	1.01	0.66	1.42	1.86	0.46	0.21	0.45	0.46	0.31
(E)-2-Nonenal	0.08	Cucumber, green	Green	nd	31.25	42.95	62.07	102.97	30.83	20.94	31.01	57.80	55.40
(E,Z)-2,6-Nonadienal	0.02	Green, fresh cucumber	Green, fruity	101.77	nd	96.67	100.69	105.06	nd	nd	nd	101.82	96.98
1-Nonanol	34	Fruity, soapy	Fruity	0.01	0.02	0.02	0.01	0.02	0.01	0.01	0.01	0.02	0.01

[a] Reported odor descriptors and odor thresholds (Wen et al., 2014; Czerny et al., 2008; Aoki & Koizumi, 1986; Czerny et al., 2008; Tamura et al., 2001; Masanetz & Grosch, 1998; Rosen et al., 1963).

[b] Odour Activity Values (OAVs), ratio of the concentration of a molecule to its odour threshold.

E)-2,6-nonadienal in the five grape varieties.

The evolution patterns of the GLV profiles during grape ripening are shown in Fig. 4a and 4b for vintages 2019 and 2020, respectively. Our results showed that the evolution of GLV profiles differed between varieties and even within the same variety in both vintages, which might be explained by variations in cultivar genetics and environmental responses (Luo et al., 2019). The dynamic changes in the GLV profiles of five grape cultivars during the berry development stages are discussed below.

Concentration heatmaps illustrated that the GLV profiles were clustered into two large groups regarding their evolution similarity in five grape cultivars in both vintages. In 2019, (Z)-3-hexen-1-ol, (Z)-3-hexenyl acetate, and 1-nonanol had similar evolution patterns in five grape cultivars and had a higher content during the veraison stage and lower levels at harvest. Among all grape cultivars, the vast majority of the remaining GLV components remained at low levels at the early development stage and reached their maximum content at the ripening stage (Fig. 4a). Some GLV profiles exist only in specific stages of typical varieties. For example, 3-hexenal was detected only in Chardonnay, Sauvignon Blanc, and Cabernet Sauvignon at harvest in the 2019 vintage. Except for Sauvignon Blanc, (E,E)-2,6-nonadienal was not detected before the veraison stage (E-L 34 and E-L 35), followed by relatively low levels at harvest. In terms of (E)-2-hexen-1-ol, Cabernet Sauvignon had the highest abundance during berry development, and Cabernet Franc had the lowest abundance (Fig. 4a).

Vintage variations in some GLV compound evolution patterns are shown in Fig. 4. (Z)-3-hexen-1-ol, (E)-2-hexen-1-ol, and (Z)-3-hexenyl acetate in five cultivars during ripening were divided into the same class in the 2020 vintage, their content in Sauvignon Blanc berries peaked at veraison (E-L 36), and their content was higher at the early stage for the other four grape cultivars (E-L 34 and E-L 35). (E)-2-nonenal peaked at veraison (E-L 36) in Cabernet Sauvignon and Cabernet Franc in the 2020 vintage, followed by a decrease at harvest (Fig. 4b). Similar to vintage 2019, (E,E)-2,6-nonadienal was detected at relatively low levels at harvest in most cultivars. 1-Nonanol, nonanal, and 2-ethyl-1-hexanol showed the same evolution patterns in the five grape cultivars of the 2020 vintage, which had a lower content throughout Cabernet Sauvignon and Cabernet Gernischt berry development in comparison to Sauvignon Blanc grapes. Most of the remaining GLV components were more abundant in Chardonnay and Sauvignon Blanc berry development in the 2020 vintage, and they reached their maximum content at E-L 37.

3.5. Analysis of aroma activity

The odor activity values (OAVs) of the GLV compounds in the five grape cultivars during their development are shown in Table 1. The OAV values are calculated using the concentration ratio to the threshold in water, and compounds with OAV values above 1 contribute to grape aroma (Wen et al., 2014). There were almost 9 and 7 GLV compounds with OAV values > 1 in all mature berries in 2019 and 2020 vintages, respectively. Two odor-active GLV compounds with OAVs higher than 30 in five mature grape cultivars were (E)-2-nonenal and (E,Z)-2,6-nonadienal in both vintages 2019 and 2020, which might significantly contribute to grape aroma. For the 2019 vintage, Sauvignon Blanc had a high OAV (112.33) for 3-hexenal at harvest, the OAV value of which was 68.45 and. 61.88 in mature Cabernet Sauvignon and Chardonnay berries, respectively. Hexanal had the highest OAV (187.94–357.99) in all mature berries of the 2020 vintage, followed by (E,Z)-2,6-nonadienal and (E)-2-nonenal. The OAV value of hexanal reached approximately 350 in mature Cabernet Franc and Sauvignon Blanc grapes, which might play a key role in aroma formation.

Descriptors are widely used to recount the odors of volatile

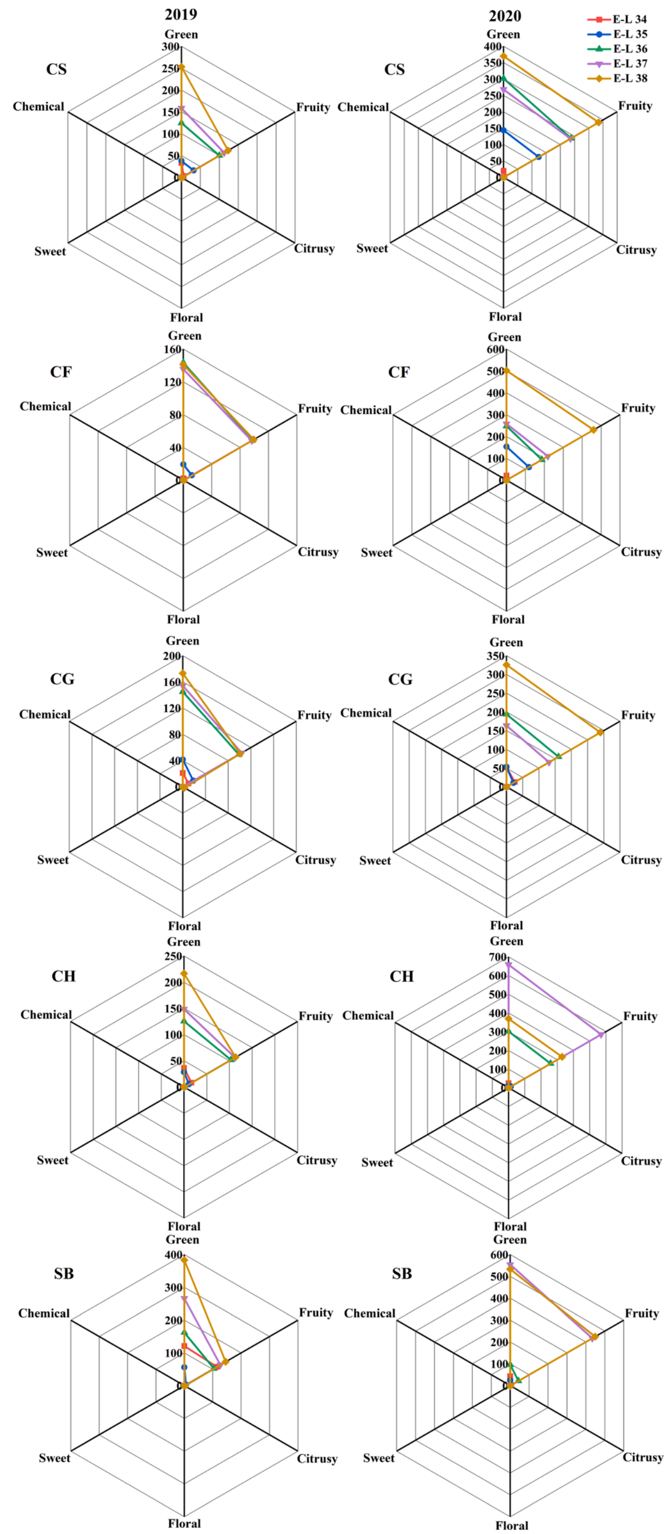

Fig. 5. Aromatics series of aroma compounds during the five grape cultivars ripening based on OAVs in the vintage 2019 and 2020. CS, Cabernet Sauvignon; CF, Cabernet Franc; CG, Cabernet Gernischt; CH, Chardonnay; SB, Sauvignon Blanc.

compounds (Yao et al., 2021). The descriptors of aroma compounds were divided into six types: green, fruity, sweet, citrusy, floral, and chemical (Wen et al., 2014). The potential odor contribution of GLV compounds in the grapes of these five cultivars is shown in Fig. 5. In vintage 2019, the odor characteristic green was significantly stronger than the other odors in all five grape varieties. The odor characteristic green varied in five grape cultivars, and the most intense green odor characteristic was found in mature Sauvignon Blanc berries due to the high concentration of 3-hexanal. Each grape variety reached its maximum odor at maturity. The evolution of the odor characteristics of berries during grape development is affected by season. In the 2020 vintage, the odor characteristics of green and fruity were relatively strong in all five grape varieties. Except for Sauvignon Blanc grapes, the odor characteristics of other grape cultivars changed regularly during ripening. For Chardonnay grapes, the most intense green and fruity odor characteristics were found at E-L 37. At harvest, Sauvignon Blanc grapes had the strongest green odor, followed by Cabernet Franc berries.

4. Conclusions

This study evaluated the varietal dependence of GLV profiles during berry ripening. A total of 13 GLV profiles were detected: four C_6 aldehydes, four C_6 alcohols, one C_6 esters, three C_9 aldehydes, and one C_9 alcohol. Among the GLV profiles in all mature berries, (E)-2-hexen-1-ol and 1-hexanol were the dominant components from the 2019 vintage, and hexanal was the most abundant individual component of the 2020 vintage. Principal component analysis showed that the five wine grape varieties had discriminating GLV profiles at different E-L stages. In terms of the total GLV content, their evolution patterns were affected by the grape cultivar and season. All varieties reached a maximum at maturity in the 2019 vintage; the total GLV content was higher in Sauvignon Blanc and Cabernet Sauvignon grapes, followed by Chardonnay, Cabernet Gernischt, and Cabernet Franc. Furthermore, Cabernet Sauvignon grapes had the highest total GLV content during the veraison stage (from E-L 34 to E-L 37), and Cabernet Franc had the lowest content during the same period. In the 2020 vintage, Cabernet Gernischt grapes had a higher total GLV content at the early ripening stage (E-L 34 and E-L 35). The total GLV content in Chardonnay and Sauvignon Blanc berries rapidly accumulated at veraison and peaked before harvest, with an obvious reduction at maturity. For specific GLV profiles, (Z)-3-hexen-1-ol and (Z)-3-hexenyl acetate had similar evolution patterns in the five grape cultivars, and they had a higher content during the veraison stage and lower levels at harvest.

The present results provided insights into the GLV profiles of different types of wine grape varieties at various ripening stages. Our findings could help winemakers create a good balance of wine aroma by selecting the optimum grape maturity and variety to improve the “green leaf” odor under the production of multi-varietal wines.

CRediT authorship contribution statement

Xiaofeng Yue: Conceptualization, Methodology, Software, Investigation, Writing – original draft. **Yanlun Ju:** Validation, Formal analysis, Visualization. **Yitong Cui:** Validation, Formal analysis, Visualization, Software. **Shichao Wei:** Validation, Formal analysis, Visualization, Software. **Huaide Xu:** Validation, Formal analysis, Visualization. **Zhenwen Zhang:** Resources, Writing – review & editing.

Declaration of Competing Interest

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

Data availability

The data that has been used is confidential.

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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.2023.100676.

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