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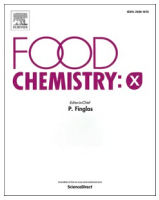

Comprehensive study of chemical and sensory profiles of hawthorn wines from China

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ABSTRACT

Hawthorn wine has gained increasing popularity in China, but comprehensive research on its sensory and chemical characteristics is still limited. This study established a sensory lexicon using Pivot Profile to describe and differentiate Chinese hawthorn wines. Based on the sensory data, 13 hawthorn wines presented three different styles, namely 'Sweet', 'Fruity' and 'Alcohol'. A total of 129 volatile compounds were identified and quantified in all hawthorn wines using headspace solid-phase microextraction (HS-SPME) combined with both gas chromatography-Orbitrap mass spectrometry (GC-Orbitrap-MS) and gas chromatography-Quadrupole mass spectrometry (GC-Quadrupole-MS). Partial least-squares regression revealed that 'sweet', 'hawthorn', and 'honey' attributes were positively correlated with several terpenes, volatile phenols, lactones, ethyl cinnamate, nonanal and phenylacetaldehyde, as well as sugar content, while negatively correlated with alcohol content. Furthermore, a salting-out effect of certain terpenes and volatile phenols was observed with increasing sucrose concentration, potentially enhancing the perceived intensity of these above attributes.

1. Introduction

Hawthorn fruit is widely grown in the temperate regions of Asia, Europe, and North America. In China, hawthorn has been approved as "medicine food homology" and can be used as both medicinal and food ingredients (Lou et al., 2022). The health benefit of hawthorn can be attributed to its abundant bioactive compounds, such as phenolic compounds and triterpenoid acids, which have antioxidant, anti-inflammatory, anti-cancer, anti-cardiovascular, and stimulating digestion activities (Lou et al., 2022; Yin et al., 2020). However, the strong sour taste of hawthorn fresh fruits limits their utilization and economic value, prompting food industries to produce derivative products. Nowadays, hawthorn wine has gained increasing popularity among Chinese consumers due to its nutritional health functions and attractive flavor (Han et al., 2021). According to the production process, hawthorn wine can typically be categorized into fermentation type and soaking type (QB/T 5476.2–2021). A few studies have been conducted on the chemical composition and health benefits of hawthorn wine (Han et al., 2021; Liu et al., 2018; Tian et al., 2024). The aroma of hawthorn wine is an important aspect in determining sensory quality and consumer acceptance, and has received increasing attention from researchers in recent years. Previous studies have mainly investigated the influence of *Saccharomyces* and non-*Saccharomyces* yeasts, fermentation stage, pulp contact, as well as pectase treatment on the concentrations of various

Abbreviations: HS-SPME, headspace solid-phase microextraction; GC-Orbitrap-MS, gas chromatography-Orbitrap mass spectrometry; GC-Quadrupole-MS, gas chromatography-Quadrupole mass spectrometry; PP, Pivot Profile; QDA, quantitative descriptive analysis; PLS, partial least-squares; $CuSO_4$, copper sulfate; NaOH, sodium hydroxide; NaCl, sodium chloride; CA, correspondence analysis; DVB/CAR/PDMS, divinylbenzene/carboxen/polydimethylsiloxane; ANOVA, analysis of variance; PCA, principal component analysis; AHC, agglomerative hierarchical clustering; VP, volatile phenol; OAV, odor activity value.

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volatile compounds (Han et al., 2021; Tian et al., 2024; Yin et al., 2020; Zhang et al., 2017). However, the aroma profile of Chinese hawthorn wine has not been fully characterized yet.

Sensory evaluation serves as a critical tool for assessing the aroma quality of foods and beverages. Among various sensory evaluation methodologies, descriptive analysis is the most mature method for characterizing products based on their perceived attributes and intensities. This method has been extensively applied in evaluating a wide range of food products, including wines and beverages (Lan et al., 2019; Lee & Chambers, 2010; Qian et al., 2024). Sensory lexicon is defined as a "standardized vocabulary", which is an essential prerequisite for conducting a descriptive sensory analysis (Suwonsichon, 2019). A sensory lexicon plays an essential role as an effective communication tool among diverse audiences, including sensory panels, sensory scientists, product developers, and marketing professionals (Suwonsichon, 2019). It allows different people to describe food product using a lexicon with well-defined and referenced descriptors and standardized evaluation procedures. Sensory lexicons have been developed for a variety of alcoholic beverages, such as wine and whiskey (Arnold et al., 2019; Souza Gonzaga et al., 2019). To our knowledge, no detailed research has been carried out on the aroma profile of hawthorn wine. It is therefore essential to develop a sensory lexicon for hawthorn wine in order to establish a standardized sensory evaluation system for hawthorn wine products.

Previous studies on the aroma of hawthorn wine have typically employed headspace solid-phase microextraction (HS-SPME) coupled with chromatography-mass spectrometry (GC–MS) techniques for the identification and quantification of aroma compounds (Han et al., 2021; Tian et al., 2024; Yin et al., 2020). However, the limited resolution and sensitivity of GC-Quadrupole-MS have posed a challenge in identifying some impact aroma compounds with low concentrations (Liu et al., 2022; Qian et al., 2020). This may have implications for the comprehensive study of hawthorn wine aroma. In recent years, the high-resolution gas chromatography-Orbitrap mass spectrometry (GC-Orbitrap-MS) has emerged as a promising alternative to GC–MS, providing high sensitivity, high mass resolving power (120,000 full width at half maximum (m/z 200) and high mass accuracy (<3 ppm). This instrument has recently been applied to the quantitative analysis of some trace volatile compounds in various food products, such as fruit wine (Liu et al., 2022), goji berry pulp (Wang et al., 2024), and infant formula (Li et al., 2024). Thus, GC-Orbitrap-MS has the potential to be a powerful tool for analyzing various trace volatile compounds in hawthorn wine.

Hawthorn wine is a complex alcoholic beverage, containing both volatile (ethanol) and non-volatile components including sugars, organic acids, polyphenols, and proteins. Numerous studies have demonstrated that wine matrix compounds can interact with aroma compounds, affecting their volatility and ultimately altering aroma perception (Villamor & Ross, 2013). Currently, the commercial hawthorn wines on the Chinese market have widely varying levels of residual sugar (ranging from 5 to 150 g/L) and ethanol (ranging from 4 % to 14 % v/v). It is therefore crucial to take into account the variable matrix components, such as alcohol and sugar, when analyzing the aroma profile of hawthorn wine. Several studies have indicated that the elevated ethanol concentrations significantly reduced the activity and partition coefficients of volatile compounds, and greatly increased the odor detection thresholds (Grosch, 2001; Villamor & Ross, 2013). However, the impact of sugar on wine aroma has been less frequently investigated. Few studies have offered inconsistent data on the effect of sugar, either enhancing or suppressing the release of volatile compounds (Hansson et al., 2001; Rabe et al., 2003; Rodriguez-Bencomo et al., 2011; Tsitlakidou et al., 2019).

Given the growing popularity of hawthorn wines, it was crucial from both industry and scientific perspectives to understand the aroma profile of such wines produced in China. Chinese hawthorn wine would develop a variety of flavor styles, which could be influenced by different matrix components such as alcohol and sugar, but there is still a lack of experimental verification. The main objectives of this study were as follows: (1) establish an aroma lexicon for hawthorn wine using Pivot Profile; (2) characterize the aroma profile of a range of commercial hawthorn wines by quantitative descriptive analysis (QDA); (3) accurately quantitate the volatile compounds in low-sugar and high-sugar matrices using HS-SPME combined with both GC-Quadrupole-MS and GC-Orbitrap-MS; and (4) explore the relationships between chemical compositions and sensory profiles by partial least-squares (PLS) regression. It is expected that the findings of this research will provide a better understanding of the general flavor chemistry of Chinese hawthorn wines.

2. Materials and methods

2.1. Hawthorn wine samples

Thirteen commercial hawthorn wines produced in different regions of China, including Shanxi, Jilin, Liaoning, Jiangsu, Hebei, Shandong, and Guangxi were used in this study (Table S1). These samples were carefully selected from 25 wines with the assistance of five wine experts to ensure that they all had the typical hawthorn wine flavor. Based on their residual sugar content, these samples were categorized into one dry (SDB-GH), four semi-dry (LRJ, SDB-BG, SDB-XM and SBL-J), two semi-sweet (ZG and SDB-BT) and six sweet wines (CBS, SPR, SZQY, LT, HGS and FS) according to the National Light Industry Standards of the People's Republic of China (QB/T 5476.2–2021). The alcohol content of these samples varied widely, ranging from 4 to 14 %v/v (Table S1). The FS sample was a formulated type with the highest alcohol content of 14 % v/v, while the other 12 samples were fermented hawthorn wine. In addition, only LRJ wine was made from *Malus doumeri* (Bois) Chev fruits, which had a high pH value of 4.59, significantly higher than other wines made from *Crataegus pinnatifida* Bge fruits.

2.2. Basic wine composition

The pH values of hawthorn wines were measured using a pH meter (HI2221, HANNA Instruments, Italy). The residual sugar and titratable acidity content of hawthorn wines were determined according to the National Standards of the People's Republic of China (GB/T 15038–2006). The detailed information on alcohol, pH, residual sugar and titratable acidity of thirteen hawthorn wines was shown in Table S1.

2.3. Chemicals

Analytical reagent grade D-glucose, tartaric acid, anhydrous copper sulfate ($CuSO_4$), sodium hydroxide (NaOH), potassium sodium tartrate, and propylene glycol were purchased from Beijing Chemical Works (Beijing, China). Sodium chloride (NaCl) and n-butanol were purchased from Shanghai Macklin Biochemical Co., Ltd. (Shanghai, China). Ethanol and dichloromethane (GC grade) were supplied by Honeywell (Marris Township, NJ, USA). The C7-C40 n-alkanes chemical reagents were obtained from Sigma-Aldrich (St. Louis, MO, USA). A total of 88 standards of aroma compounds with purities ≥97 % used for qualitative and quantitative analysis were supplied by Sigma-Aldrich (St. Louis, MO, USA), Shanghai Macklin Biochemical Co., Ltd. (Shanghai, China), Aladdin Bio-Chem Technology (Shanghai, China), Adamas Reagent, Ltd. (Shanghai, China), and CNW Technologies (Düsseldorf, Germany), as detailed in Table S2.

2.4. Sensory lab environment

All the sensory experiments were conducted in our sensory laboratory under the standard environment with controlled light and constant temperature (20 °C). The tests were carried out in individual booths and participants were supervised and required not to communicate with each other throughout the evaluation process. Each wine sample (25

mL) was prepared in an International Standards Organization (ISO) wine tasting glass, coded with three-digit random numbers and presented to panelists in random order. Ethical approval for the involvement of human subjects in this study was granted by Beijing Forestry University Ethics Committee. All participants were informed that their participation in these studies was entirely voluntary and free, and their informed consent form was signed before participating.

2.5. Development of an aroma lexicon

2.5.1. Pivot profile

The sensory panel 1 consisting of 12 judges (four males and eight females, aged 22–44) took part in the Pivot Profile (PP) experiments. All panelists were trained sensory assessors with more than three years of experience in wine sensory analysis. The FS wine was chosen as the pivot sample. Twelve sets of samples (one of twelve commercial hawthorn wines paired with the pivot sample in one set) were provided for panelists. During the evaluation process, assessors were asked to first smell the pivot and then another sample in each set. Assessors needed to use the sensory attributes freely avoiding hedonic words (such as "pleasant" or "unpleasant") and negative terms (such as "not strong") to describe the perceived samples when comparing two samples in each set. Meanwhile, they considered the intensity of those attributes of the sample in pair with the pivot sample to select either "MORE" (more intensity than the pivot) or "LESS" (less intensity than the pivot) options in the provided response sheet. All twelve sets of samples were evaluated in duplicate by panelists over four sessions, with six sets assessed per session. A 60 s rest between each sample set and a 10 min rest between each session was taken. After evaluation, data from 12 panelists were collected and subsequently subjected to the attribute grouping and classification process.

2.5.2. Panel training

The panel 2 consisted of 15 trained panelists (five males and ten females, aged 20–26), which were originally selected from a pool of 36 candidates from Beijing Forestry University. Panelists were selected and recruited in accordance with the National Standards of the People's Republic of China (GB/T 16291.1–2012) and participated in an eight-month training period, with three one-hour training sessions per week. The training sessions covered a range of sensory evaluation techniques and methods, including aroma recognition, detection/discrimination tests, ranking test, attribute generation, consensus on reference standards and rating test for intensity. The panelists were trained to use a calibrated n-butanol scale for scaling odor intensities (Table S3), as described by Xiao et al. (2019). After training and tests, all panelists showed good performance in aroma recognition and discrimination, as well as consistency and reproducibility for intensity rating. Panel 2 was involved in the subsequent lexicon development process and descriptive analysis.

2.5.3. Selection and definition of the aroma attributes

The generated sensory descriptors from PP were reviewed and rationalized by the panel 2. The aroma descriptors with the same or similar meanings were merged and redundant terms were eliminated. Only consensus descriptors related to the hawthorn samples directly were included in the preliminary descriptors list ($n = 72$). Following the method by Thuillier et al. (2015), the PP data were then transformed and subjected to correspondence analysis (CA) with the filtering option ($Cos2 > 0.1$) to select the descriptors with larger contributions. In order to facilitate practical applications, the number of aroma descriptors was greatly reduced by eliminating the descriptors with low frequencies (below nine) used by panel 1. The reduced list of descriptors was obtained by consensus of the trained panel 2. Subsequently, the intensity of each aroma attribute was scored by panel 2 using a 9 cm linear scale from 0 (very weak) to 9 (very intense). The final data were calculated from a mixture of intensity and frequency of detection using the following formula:

$$(M =)\sqrt{F(\%)*I(\%)}$$

F% is the average frequency of the referred attributes by the panel, and I% is the average intensity of the referred attributes in the group expressed as the percentage of maximum intensity.

Based on the above results, the descriptors with low M values were further eliminated, which indicated that they contributed less to the aroma of the samples. Once a complete list of attributes was finalized, reference standards for each attribute were introduced and modified until the panel reached a consensus that the standard effectively represented the attribute, as previously described in our study (Wang et al., 2024). The odor intensity of each reference sample was calibrated in accordance with the n-butanol scale.

2.4. Quantitative descriptive analysis for lexicon validation

The lexicon was validated through the quantitative descriptive analysis for thirteen hawthorn wines, thereby confirming whether the attributes facilitate the description and differentiation of the product. During formal sessions, the wine samples were prepared in ISO wine tasting glasses labeled with a random three-digit code and presented to the 15 panelists (panel 2) in a random order. Panelists were required to score the intensity of all attributes for each sample using a 9 cm linear scale with the left and right anchors labeled "0 (very weak)" and "9 (very intense)", respectively. All thirteen hawthorn wines were evaluated in duplicate by panel 2 over six sessions spanning two days. Four sessions comprised four samples each, while the remaining two sessions included five samples each. A rest of 60 s between each sample and a 10 min rest between each session was taken.

2.6. Volatile compounds analysis

2.6.1. Sample preparation

Two methods, HS-SPME combined with GC-Quadrupole-MS or GC-Orbitrap-MS were involved in aroma quantitation. The alcohol content of all wine samples was normalized to 14 % (*v*/v) by the addition of chromatographic grade absolute ethanol. Then, 5 mL of each hawthorn wine sample was added to a 20 mL vial and spiked with 1.0 g of NaCl and 10 μL of 4-methyl-2-pentanol (1.0086 g/L) as internal standard. Each sample was prepared in three replicates and used for the subsequent analysis.

2.6.2. HS-SPME-GC-quadrupole-MS

Volatile compounds were extracted by headspace solid-phase microextraction and quantitated by GC-Quadrupole-MS according to Lan et al. (2019). Automatic HS-SPME was performed on a CTC CombiPAL autosampler (CTC Analytics, Zwingen, Switzerland) with a 2 cm DVB/CAR/PDMS 50/30 μm SPME fiber (Supelco, Inc., Bellefonte, PA). The SPME fiber was activated at 250 °C prior to sample extraction. Following equilibration at 40 °C for 30 min, the previously prepared sample was extracted with a SPME fiber at 40 °C for 30 min while stirring at 500 rpm. After the extraction, the fiber was immediately thermally desorbed by inserting into the GC injection port for 8 min.

GC–MS analysis was performed using an Agilent 7890 GC coupled with an Agilent 5795C MS. The samples were analyzed on a HP-INNOWAX column (60 m × 0.25 mm × 0.25 μm, J&W Scientific, Folsom, CA, USA). High-purity helium gas (≥99.999 %) was used as the carrier gas with a flow rate of 1.0 mL/min. The splitless mode (0.75 min) was used for injection. The oven temperature was initially held at 50 °C for 1 min, then raised to 220 °C at a rate of 3 °C/min, and held for 5 min. The temperatures of the injector, ion source, quadrupole, and MSD transfer line were held at 250 °C, 230 °C, 150 °C and 280 °C, respectively. The mass detector was operated in electron ionization (EI) mode at 70 eV with full scan (*m/z* 30–350).

2.6.3. HS-SPME-GC-Orbitrap-MS

The analysis of volatile compounds using headspace solid-phase microextraction combined with GC-Orbitrap-MS was conducted as described by our previous research (Liu et al., 2022). Automatic HS-SPME was carried out on a TriPlus RSH autosampler (Thermo Fisher Scientific, Bremen, Germany) with a 2 cm DVB/CAR/PDMS 50/30 μm SPME fiber (Supelco, Inc., Bellefonte, PA). The prepared sample vial was equilibrated at 60 °C for 30 min. Subsequently, the pre-activated SPME fiber was exposed to the headspace for 30 min while maintaining at 60 °C with stirring. Finally, the fiber was thermally desorbed in splitless mode and held in the GC inlet for 8 min.

GC-Orbitrap-MS analysis was carried out using a Thermo Scientific Trace 1300 gas chromatograph combined with a Thermo Scientific Q-Exactive Orbitrap mass spectrometer (GC-Orbitrap-MS, Thermo Scientific, Bremen, Germany). A DB-WAX column (30 m × 0.25 mm × 0.25 μm, J&W Scientific, Folsom, CA, USA) was used to separate volatile compounds with high-purity helium gas (≥99.999 %) as the carrier gas at a flow rate of 1.2 mL/min. The oven temperature started at 40 °C, held for 5 min, then raised to 180 °C at a rate of 3 °C/min, and finally raised to 250 °C at 30 °C/min, followed by a hold for 10 min. The temperatures of the injector, ion source, and MSD transfer line were held at 250 °C, 280 °C, and 230 °C, respectively. Positive ion electron ionization was set at 70 eV in the Orbitrap mass spectrometer, and the scan masses from *m/z* 33 to 300 were recorded.

2.6.4. Identification and quantification of volatile compounds

The identification of volatile compounds was achieved by comparison of their mass spectra and retention indices (RIs) with those of reference standards and compounds in the NIST 20 MS database. RIs of volatile compounds were calculated from the retention times of n-alkanes (C7-C40). An index database previously established by our research group (Food Flavor Laboratory, http://foodflavorlab.cn/) was also used for the identification of volatile compounds. The detailed identification information for each compound can be found in Table S2.

The quantification of volatile compounds was carried out in two hawthorn wine matrices with different sugar content. The low sugar matrix was obtained by blending two commercial hawthorn wines, with a final sugar content of 8.4 g/L. Subsequently, a certain amount of sucrose was added to the mixed hawthorn wine to obtain a high sugar matrix with a sugar content of 82.4 g/L. The two hawthorn wine matrices were treated with a sonicator (KQ-800KDE, Kunshan Ultrasonic Instrument Co., Ltd.) at ultrasonic frequency of 40 kHz and power of 200 W for 15 min to reduce the concentration of aroma compounds. After ultrasonic treatment, the two hawthorn wine matrices were adjusted to a pH of 3.65 using NaOH and an alcohol content of 14 % (*v/v*). The calibration solutions were prepared in duplicate in both low and high sugar wine matrices at 12 concentration levels for various aroma compounds, containing a fixed level (2017.2 μg/L) of 4-methyl-2-pentanol (internal standard). According to the sugar content in the wines, the volatile compounds in seven wine samples (ZG, SDB-BT, LRJ, SDB-BG, SDB-XM, SDB-GH and SBL-J) were quantified using the low sugar matrix calibration curve, while those in six sweet wine samples (LT, CBS, HGS, SPR, SZQY and FS) were quantified using the high sugar matrix calibration curve.

2.7. Statistical analysis

Discrimination and repeatability of sensory panel were evaluated by Panel Check software (version 1.4.2). Analysis of variance (ANOVA) and independent samples *t*-test were used for the wine chemical compounds and sensory scores to evaluate the differences between the samples and carried out using Duncan's test by the SPSS software (version 22.0, IBM, Chicago, Ill, USA). WordCloud and Bubble plot of aroma descriptors was conducted on HiPlot (https://hiplot.com.cn/home/index.html) and ChiPlot (https://www.chiplot.online/), respectively. Heatmap with hierarchical cluster analysis was conducted using the "ComplexHeatmap" package in the R environment (4.2.2). Principal component analysis (PCA), correspondence analysis (CA), agglomerative hierarchical clustering (AHC), and partial least-squares (PLS) regression were performed with XLSTAT statistical software (2019, Addinsoft, Paris, France).

3. Results and discussion

3.1. Establishment of hawthorn wine aroma lexicon

The 13 hawthorn wine samples, encompassing the full range of possible sensory attributes, were evaluated to develop a comprehensive lexicon for describing the aromas of hawthorn wines. In the initial phase of the lexicon study, the expert judges generated a total of 72 aroma descriptors by Pivot Profile tests (Fig. 1a and Table S4). These aroma descriptors were classified into nine categories, including fruity, chemical, microbial, herbal, caramel, woody, earth, floral, nutty, and spice. Among them, the fruity aroma descriptors, such as 'hawthorn', 'preserved fruit', 'jam', 'apricot' and 'orange peel', were the most frequently cited. The PP data were then transformed and analyzed using correspondence analysis (CA) with the filtering option (Cos2 > 0.1) to obtain the 54 descriptors with larger contributions (Fig. 1b). After further elimination of descriptors with a frequency below nine, 23 aroma attributes retained (Fig. 1c and Table S5). The 23 descriptors were depicted in a two-tiered wheel (Fig. 1c). The descriptors that constitute the outer tier are the specific attributes, while the secondary descriptors that group a certain type of attributes are the second tier.

To further determine the most important descriptors, the trained panel assessed the strength of each aroma attribute and calculated its M-value (Fig. 1d). Only the aroma attributes with M-values more than 0.08 were included in the aroma lexicon. Following the screening process, a total of 12 aroma descriptors were retained, including 'hawthorn' (0.561, M-value), 'alcohol' (0.441), 'sweet' (0.284), 'honey' (0.249), 'fruity' (0.218), 'herb' (0.215), 'vinegar' (0.158), 'sauce' (0.149), 'liquorice' (0.100), 'smoky' (0.100), 'beer' (0.092), and 'floral' (0.083). Finally, the 12 aroma attributes with their definitions, references, as well as odor intensities were developed for the hawthorn wine aroma lexicon. The final list is shown in Table 1.

3.2. Descriptive sensory analysis of hawthorn wines

Once the aroma lexicon of hawthorn wine had been established, a quantitative descriptive analysis (QDA) was conducted to validate the lexicon by a well-trained panel. The panelists assessed the 12 aroma attributes of the 13 hawthorn wines. The mean scores for each attribute and significant differences among the 13 wines were detailed in Table S6. Except for 'vinegar' ($p < 0.05$), all other aroma attributes showed highly significant differences ($p < 0.001$) among the hawthorn wines. This result demonstrates that the established aroma lexicon is capable of describing and differentiating hawthorn wine products.

All the significantly different attributes were subjected to PCA, and the first two principal components accounted for 57.37 % of the variation (Fig. 2a). The first principal component separated samples which were associated with sweet, hawthorn and honey aromas from those that exhibited alcohol, sauce, vinegar, smoky, and beer characters. Samples with fruity attribute were differentiated along PC2 from those that were herb, sweet, sauces characters. Wines were relatively dispersed within the PCA score plot (Fig. 2a), indicating variation in terms of both the sensory profiles and wine styles covered within this sample set, although some clustering could also be observed.

The hawthorn wine samples were further subjected to agglomerative hierarchical clustering (AHC) analysis based on the QDA results for all aroma attributes (Fig. 2b). The detailed aroma profile of hawthorn wines was illustrated in Fig. 2c-e. According to AHC analysis, the 13 hawthorn wines could be classified into three distinct groups (Fig. 2b). CBS, SPR, FS, HGS, LT and ZG were among the first group (namely 'Sweet' type) that perceived to be higher in 'sweet', 'hawthorn' and 'honey' aromas

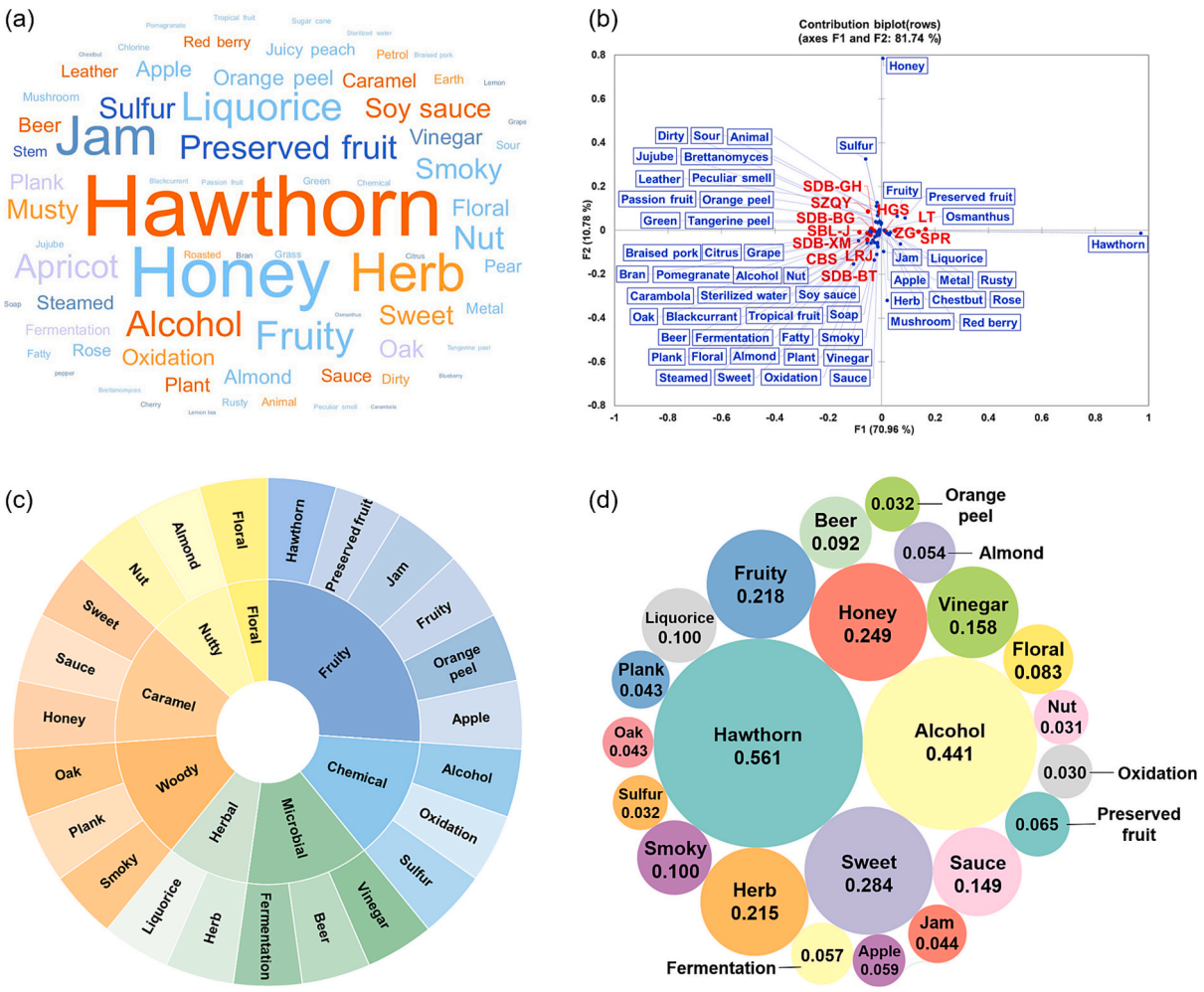

Fig. 1. (a) Wordcloud of the initial 72 aroma descriptors by Pivot Profile; (b) Correspondence analysis biplot of the 13 hawthorn wines; (c) Aroma wheel based on the selected aroma descriptors with frequencies above 9 in hawthorn wine; (d) Calculated M values of aroma descriptors.

(Fig. 2c). The second group namely 'Fruity' type, which included SDB-BG, SDB-GH and SBL-J, was characterized by more vital 'fruity' attribute and weaker 'sweet' attribute (Fig. 2d). The third group namely 'Alcohol' type, including SDB-BT, SDB-XM, SZQY and LRJ, displayed 'alcohol', 'vinegar', and 'sauce' aromas (Fig. 2e).

Interestingly, except for FS with an alcohol content of 14.0 % *v*/v, the other five 'Sweet' type wines had low average alcohol content (6.2 % *v*/v) and high average sugar content (97.0 g/L) (Table S1 and Fig. 2). In contrast, both 'Fruity' and 'Alcohol' type wines were in high alcohol content and low in sugar content, with average alcohol content of 12.3 % v/v and 11.0 % v/v, and average sugar content of 10.1 and 28.6 g/L, respectively. Our results revealed that hawthorn wines with low-alcohol and high-sugar matrices tended to exhibit prominent 'sweet', 'honey' and 'hawthorn' aroma characteristics when compared to the high-alcohol and low-sugar wines. As illustrated by Fig. 2d and e, both 'Alcohol' type and 'Fruity' type wines displayed a more pronounced 'alcohol' aroma than 'Sweet' type wines. This could be attributed to their relatively high alcohol content, ranging from 8 to 13 % v/v. In addition, some characters, such as 'fruity' and 'vinegar' aromas in these wine samples were usually classified as secondary aromas produced by fermentation (Villamor & Ross, 2013). By combining the physico-chemical parameters of hawthorn wines, we tentatively speculated that the sensory characteristics of hawthorn wines may have a close relationship with their alcohol and sugar content.

3.3. Analysis of volatile compounds by GC-Orbitrap-MS and GC-quadrupole-MS

In order to conduct a comprehensive analysis of the aroma compounds, both GC-Orbitrap-MS and GC-Quadrupole-MS were employed in the analysis of 13 hawthorn wines. A total of 129 compounds were identified across 13 hawthorn wines, including 46 esters, 22 terpenes, 13 higher alcohols, 10 aldehydes, 10 volatile phenols (VPs), 8 acids, 5 lactones, 3 norisoprenoids, 2 pyrazines, and 10 other compounds (detailed data in Supporting information, Table S2). A comparison of the two approaches revealed that 105 compounds were detected by GC-Orbitrap-MS, while 50 compounds were identified by GC-Quadrupole-MS (Fig. 3a and b). Generally, GC-Orbitrap-MS had the advantage for the analysis of a greater number of terpenes, pyrazines, esters, volatile phenols and lactones, which were typically detected at very low concentrations in wines (Fontana et al., 2017; Liu et al., 2022; Qian et al., 2020; Yang et al., 2019). The results of this study confirmed that GC-Orbitrap-MS was a powerful tool for the detection of trace compounds in fruit wines, as previously proposed in our research (Liu et al., 2022). In addition, 24 compounds could only be identified by GC-Quadrupole-MS. Thus, the combination of GC-Orbitrap-MS and GC-Quadrupole-MS proved an effective approach for a comprehensive analysis of the aroma compounds in hawthorn wine.

3.3.1. Effect of sugar content on wine volatiles release

To better understand the effect of sugar content on the aroma release,

Table 1
Hawthorn wine sensory attributes, reference, and definitions.

Attributes	Reference Sample[1]	Definition	Intensity[2]
Hawthorn	Haw jelly	The sweet and sour aroma of hawthorn.	9.0
Floral	2-Phenylethanol (5 mg/L) and linalool (300 μg/L) dissolved in distilled water	The aroma of flowers.	7.0
Sweet	Dissolved brown sugar in hot water	The aroma produced by sweet substances such as marshmallows, caramel, vanilla, or sweet fruit flavored sweets.	10 g/100 g = 8.0
Honey	Honey dissolved in warm water	Sweet aroma reminiscent of sugar or honey.	10 g/100 g = 5.0
Fruity	Mix apple juice and pear juice in a ratio of 1:1[a]	The aroma of a mixture of different fruits.	9.0
Alcohol	40 % vol absolute vodka	The aroma associated with ethanol.	8.0
Vinegar	Apple cider vinegar[b]	The aroma of a sour fruit vinegar.	10.0
Herb	Mixed Chinese medicine soup	The aroma of Chinese traditional medicine.	9.0
Sauce	Thick broad-bean sauce[c]	The aroma of a sweet soy sauce.	10.0
Smoky	Guaiacol (100 μg/L) dissolved in 1 % propylene glycol	The aroma of smoke.	7.0
Liquorice	Compound liquorice tablet[d]	The aroma of licorice tablets.	6.0
Beery	1:1 diluted Harbin Wheat King Beer	The aroma of yeast in the beer.	7.0

[1] Selected physical reference samples: [a] HuiYuan® Apple juice and Pear juice, [b] HaiTian® Apple Cider Vinegar,
[c] Xin He® Thick broad-bean sauce, [d] Shen Wei® Compound Liquorice Tablet.
[2] Sensory attribute intensity on a 9 cm linear scale, aqueous solutions of 1-butanol at different concentrations as odor intensity referencing scales.

Fig. 3c shows the peak areas of volatile compounds in high and low sugar matrices. Overall, the results show a significant decrease in the release of several esters, including isoamyl acetate, ethyl 2-methylbutanoate, ethyl 3-methylbutanoate, ethyl hexanoate, ethyl octanoate, and ethyl decanoate, by increasing the sucrose concentration from 8.4 to 82.4 g/L (Fig. 3c). This effect was more evident in the case of isoamyl acetate and ethyl hexanoate, with an average reduction of 26 % and 18 %, respectively. This reduction could be attributed to a retention effect of certain volatiles by the non-volatile wine matrix (Rodriguez-Bencomo et al., 2011). On the contrary, an increase in sucrose concentration was also shown to significantly increase the release of some terpenes and volatile phenols, such as linalool, rose oxide, guaiacol, 4-methylguaiacol, and eugenol (Fig. 3c). In particular, the effect was more pronounced for guaiacol and linalool, with an average increase of 27 % and 25 %, respectively. This effect suggested an increase in the volatility of some compounds in the presence of certain non-volatile compounds such as sucrose, which was also called a “salting-out” effect (Hansson et al., 2001; Rabe et al., 2003). Previous studies have offered inconsistent data that the addition of sucrose either enhanced or suppressed the release of volatile compounds (Hansson et al., 2001; Rabe et al., 2003; Rodriguez-Bencomo et al., 2011; Tsitlakidou et al., 2019), which is probably due to differences in sucrose concentration, polarities of compounds, or interaction with other non-volatile compounds. According to the results in this study, it is of interest to understand how sucrose concentration influences the aroma release and subsequent aroma perception. It could be postulated that the reduced volatility of esters, resulting from high sugar content, may influence the perception of fruit aroma in sweet hawthorn wines. Conversely, the floral and sweet aromas contributed by terpenes and volatile phenols may be enhanced in sweet hawthorn wines due to the presence of high sugar.

3.3.2. Quantitation of volatile compounds in hawthorn wine

The identified aroma compounds were quantified by HS-SPME coupled with GC-Orbitrap-MS and GC-Quadrupole-MS. The concentration levels and calculated odor activity values (OAVs) of each aroma compound were shown in Table 2. Ethyl and acetate esters were the most diverse class of the determined compounds, typically associated with fruity and floral aromas in wine (Niu et al., 2019). Among the esters, ethyl acetate, isoamyl acetate, ethyl butanoate, ethyl 2-methylbutanoate, ethyl 3-methylbutanoate, ethyl hexanoate, ethyl octanoate, and ethyl cinnamate had relatively high average OAVs above 1. Several authors had already reported that ethyl acetate, isoamyl acetate, ethyl octanoate, and ethyl hexanoate had high concentrations and OAVs in hawthorn wine (Tian et al., 2024). Meanwhile, these esters were also identified as important aroma compounds in other wines (Niu et al., 2019; Qian et al., 2024; Wang et al., 2023). A number of higher alcohols were identified, in particular isopentanol and 2-phenylethanol, which impart solvent and rose notes, respectively (Lan et al., 2019). The high OAVs of these two alcohols were in accordance with the previous findings in hawthorn wines (Han et al., 2021; Tian et al., 2024). Several volatile fatty acids were also detected, and they tend to contribute to the complexity of wine aroma, imparting fatty, rancid or green notes (Cai et al., 2014). Only octanoic acid and hexanoic acid were found with high OAV, which was similar to the previous results in hawthorn wine (Han et al., 2021).

Terpenes and norisoprenoids are mainly responsible for floral and fruity aromas, and they are considered to play a vital role in determining the varietal typicality of wine (Wang et al., 2023). The terpenes with the highest OAVs were (*Z*)-rose oxide and (*E*)-rose oxide, followed by *β*-ocimene and linalool, which was in agreement with the results previously reported by Han et al. (2021). The concentration of *β*-damascenone ranged from 0.2 to 33 μg/L, in accordance with previous wine studies (Qian et al., 2024; Wang et al., 2016). Generally the concentration of *β*-damascenone in the hawthorn wines studied (mean of 2.9 μg/L) was much higher than its odor threshold of 0.05 μg/L, thus it had the highest average OAV of 58. *β*-Damascenone was characterized as having honey, sweet, and cooked apple aromas, and has been identified as key odor-active compound in various wines (Lan et al., 2019; Ma et al., 2017; Mayr et al., 2014). Nevertheless, there is a lack of research examining the concentration of *β*-damascenone in hawthorn wines (Han et al., 2021; Tian et al., 2024; Yin et al., 2020).

Phenylacetaldehyde, which contributes floral and honey aromas to wine, exhibited high OAVs in the hawthorn wines studied (Table 2). This compound has been identified as one of key odorants in oxidized wines, originating from Strecker degradation of phenylalanine and direct oxidation of phenylethanol (Lan et al., 2019; Sarrazin et al., 2007). A number of volatile phenols were quantified (Table 2), particularly eugenol and 4-ethylguaiacol, which were reminiscent of spice, clove, and smoky odors (Lan et al., 2019). Five lactones were also detected, which are related to coconut, peach, apricot, and fatty aromas (Qian et al., 2020). Although low concentrations of these compounds were found in hawthorn wines, they have been demonstrated to exert a significant influence on wine aroma through additive effects (Allamy et al., 2018; Qian et al., 2024).

Principal component analysis (PCA) was conducted on the normalized data of volatile compounds to investigate the variations among different hawthorn wines. As illustrated by Fig. 4a and b, the first two components accounted for 51.91 % of the variance. As with the PCA score plot, wine samples were spread across the plot, but wine LRJ was isolated in the top right quadrant, well separated from the other wines (Fig. 4a). This may be attributed to the unique variety of hawthorn (*Malus doumeri* (Bois) Chev) used by LRJ wine, which was different from other hawthorn wines. According to the loading plot, the first PC was mainly driven by several esters, such as methyl decanoate (E42), ethyl 3-methylbutanoate (E8), and diethyl succinate (E33), some higher alcohols, such as 2,3-butanediol (H2), isopentanol (H4) and 2-phenylethanol (H11), as well as maltol (O1) and myrtenol (T16) in the positive

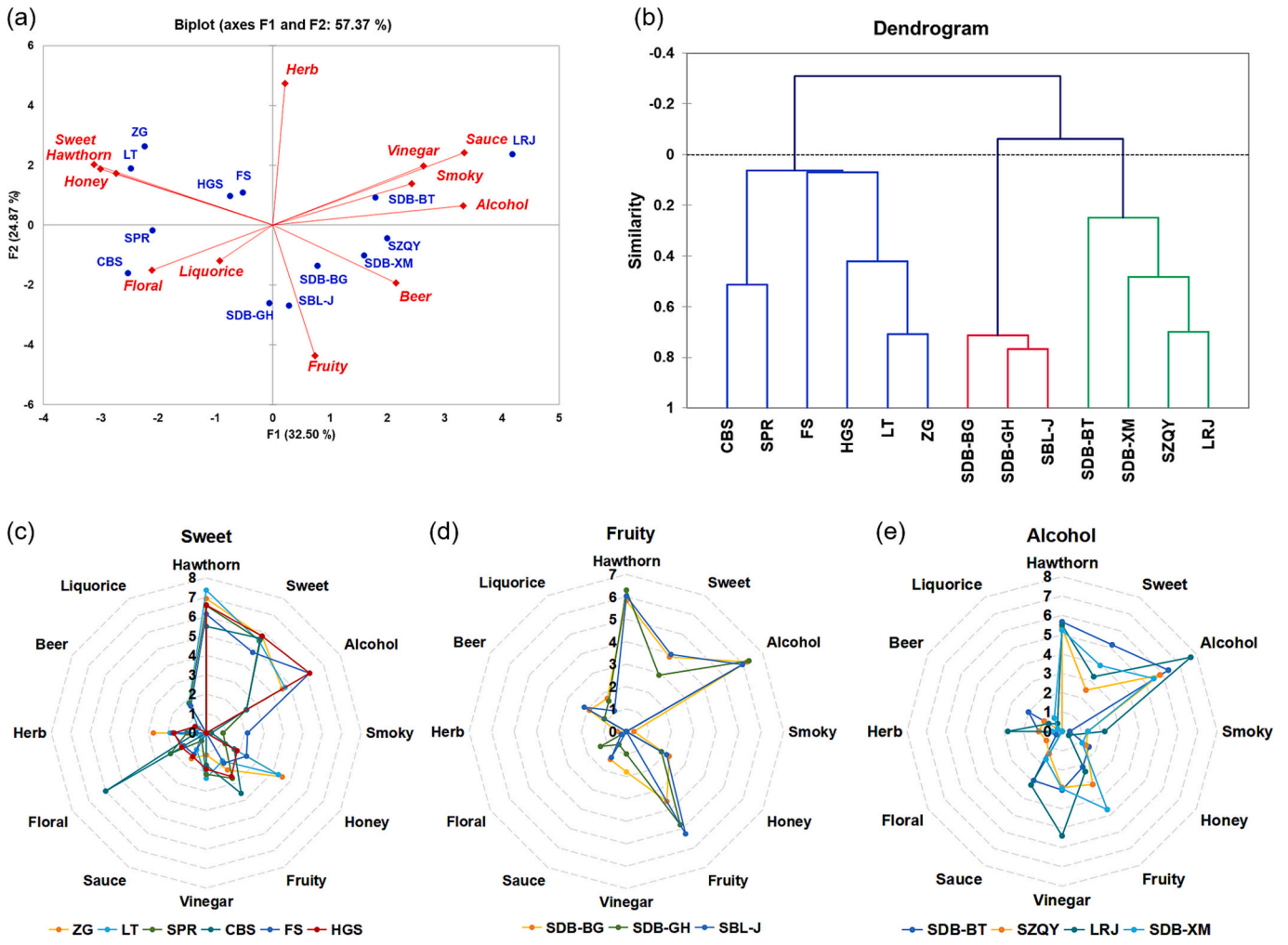

Fig. 2. (a) PCA-scores and -loadings biplot of the mean aroma sensory ratings for the 13 hawthorn wines; (b) AHC results of sensory data for 13 hawthorn wines; (c) Aroma profile analysis of six 'Sweet' type hawthorn wines; (d) Aroma profile analysis of three 'Fruity' type hawthorn wines; (e) Aroma profile analysis of four 'Alcohol' type hawthorn wines.

direction, and by ethyl salicylate (E29), ethyl cinnamate (E25), diethyl propanedioate (E36), naphthalene (O6), γ-nonalactone (L3), whiskey lactone (L2), and isoeugenol (V9) in the negative direction on PC1. Benzyl alcohol (H10), benzyl acetate (E3), ethyl nonanoate (E24), ethyl myristate (E38), and 2-furanmethanol (H12) had a large loading on PC2 in the positive direction, whereas the negative direction of PC2 was mainly driven by 2-methyl-pentanal (AD1), methyl octanoate (E41), citronellyl acetate (T20), and octanol (H8).

It was interesting that six 'Sweet' type wines were clustered on the left side, while three 'Fruity' type wines were located together on the opposite direction (Fig. 4a). However, four 'Alcohol' type samples were dispersed across the different quadrants. Six 'Sweet' type wines (FS, LT, CBS, HGS, SPR and ZG), together with SZQY, were located on the left side (Fig. 4a), being relatively high in a lot of terpenes, esters (such as ethyl cinnamate (E25) and ethyl hydrocinnamate (E26)), aldehydes, lactones, as well as volatile phenols. Three 'Fruity' type wines (SBL-J, SDB-GH and SDB-BG), together with SBD-XM and SBD-BT, which were in the bottom right quadrant, having greater amounts of several esters (such as ethyl octanoate (E21), ethyl decanoate (E22) and ethyl hexanoate (E15)), higher alcohols, acids, as well as 6-methyl-5-heptene-2-one (N1). Wine LRJ was characterized by some esters (such as benzyl acetate (E3), ethyl lactate (E31) and ethyl acetate (E1)), benzyl alcohol (H10), isobutanol (H1), furfural (AD8), as well as isobutanoic acid (A2).

The potent volatile compounds with OAVs higher than 0.1 were further subjected to the heatmap analysis to better exhibit their variations among different hawthorn wines (Fig. 4c and Table S7). It was observed that 13 hawthorn wines were clustered into two main groups. Six 'Sweet' type wines were gathered into the first group, the second group consisted of four 'Alcohol' type wines and three 'Fruity' type wines. Volatile compounds that behaved similarly across samples could be grouped into three main clusters (Fig. 4c). In general, Cluster 1 was characterized by the highest concentrations observed in LRJ wine, which included some esters (ethyl acetate (E1), phenylethyl acetate (E4), ethyl benzeneacetate (E28), ethyl 2-methylbutanoate (E7), and ethyl lactate (E31)), acetic acid (A1), isobutanoic acid (A2), isobutanol (H1), and 2-phenylethanol (H11). Cluster 2 was mainly composed of several esters (ethyl 3-methylbutanoate (E8), ethyl hexanoate (E15), ethyl octanoate (E21), ethyl decanoate (E22), and isoamyl acetate (E2)), higher alcohols (2,3-butanediol (H2), isopentanol (H4), and methionol (H13)), as well as acids (octanoic acid (A6), hexanoic acid (A3), decanoic acid (A7)). Higher concentrations of these volatiles in Cluster 2 were observed in the second group wines, which comprised three 'Fruity' and four 'Alcohol' type wines. Consistent with our results, several studies have demonstrated that several fatty acid ethyl esters and fatty acids were associated with diverse 'fruity' aroma attributes (Ferreira et al., 2016; Niu et al., 2019). Meanwhile, the 'alcohol' aroma attribute may be due to the presence of several higher alcohols, which have been reported to impart solvent, alcohol and fusel-like odors (Cai et al., 2014). Cluster 3 generally showed higher concentrations in the first group wines (six 'Sweet' type wines), and this cluster consisted of

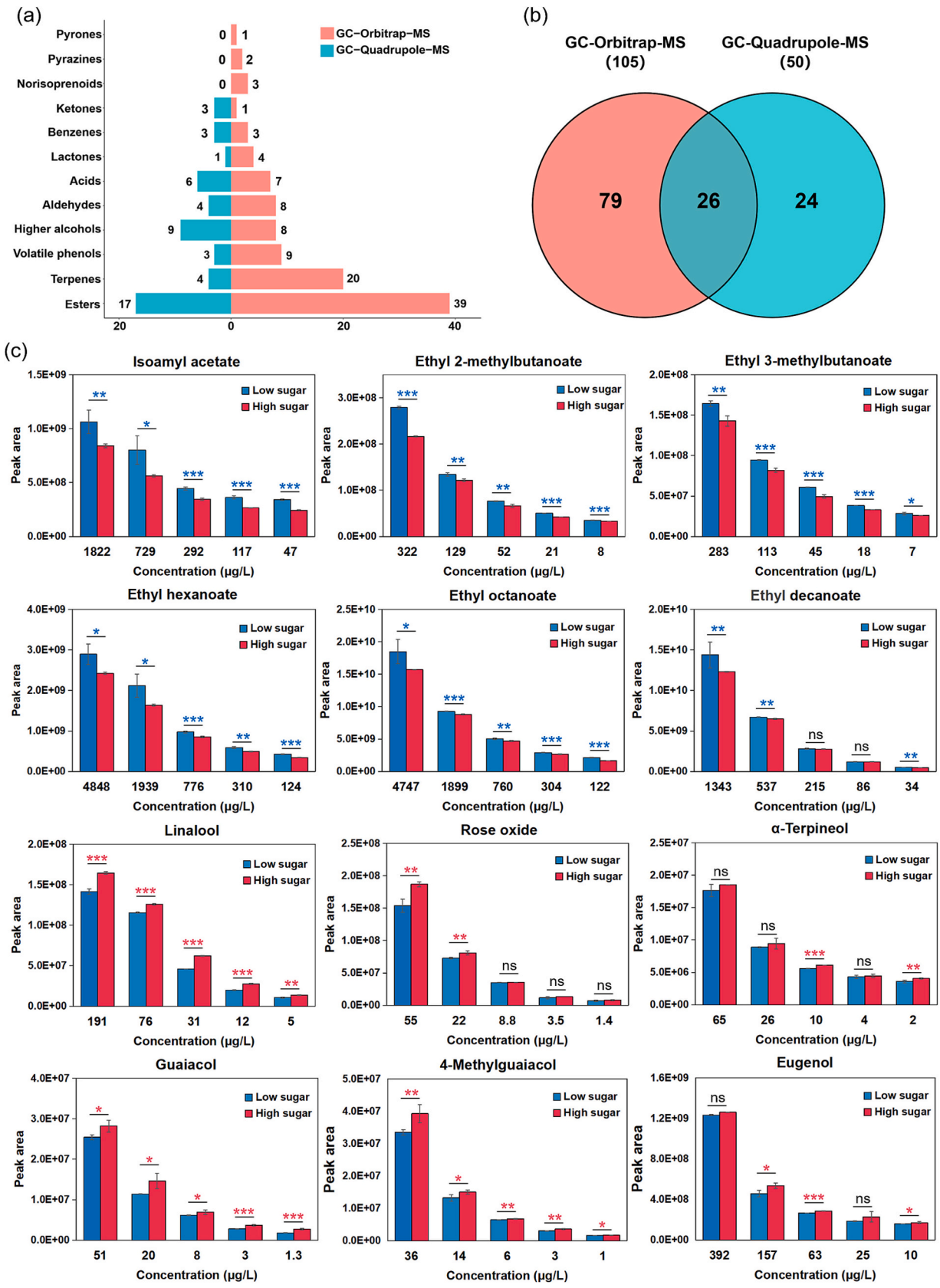

Fig. 3. (a) The kinds of aroma compounds in hawthorn wines detected by GC-Orbitrap-MS and GC-Quadrupole-MS; (b) Venn diagram of the total number of compounds detected by two instruments; (c) Effect of sugar content on the release of volatile compounds at different concentration levels in hawthorn wine. Low sugar: 8.4 g/L, and high sugar: 82.4 g/L. Effect significant at: * $p < 0.05$; ** $p < 0.01$; *** $p < 0.001$; ns: not significant. Significances in red represent the higher values in high sugar samples than low sugar samples, and significances in blue represent the higher values in low sugar samples than high sugar samples. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Table 2
Concentrations (μg/L) of volatile compounds determined in the13 hawthorn wines.

Compound	No.	CAS	Minimum	Maximum	Median	Mean (OAV)	SD[a]
Esters							
Ethyl acetate	E1	141–78-6	2204	108,568	25,989	33,736 (4.5)	25,794
Isoamyl acetate	E2	123–92-2	45	1310	64	201 (6.7)	346
Benzyl acetate	E3	140–11-4	0.4	65	1.0	6.6 (–)	18
Phenylethyl acetate	E4	103–45-7	1.8	63	5.5	18 (0.1)	22
Ethyl propanoate	E5	105–37-3	11	19	14	14 (<0.01)	4.8
Ethyl butanoate	E6	105–54-4	43	184	67	82 (4.1)	43
Ethyl 2-methylbutanoate	E7	7452–79-1	4.4	54	20	25 (25)	16
Ethyl 3-methylbutanoate	E8	108–64-5	7.4	34	20	19 (6.4)	12
Ethyl (*E*)-2-butenoate	E9	623–70-1	0.7	71	12	21 (–)	21
Ethyl tiglate	E10	5837-78-5	0.9	6.4	1.3	2.8 (–)	2.0
Ethyl carbonate	E11	105–58-8	16	1234	1189	652 (–)	613
Ethyl pentanoate	E12	539–82-2	4.1	30	6.5	10 (–)	8.4
Ethyl 2-methylpentanoate	E13	39,255–32-8	1.0	121	2.3	32 (–)	34
Ethyl 2-hydroxy-4-methylpentanoate	E14	10,348–47-7	23	719	77	148 (0.2)	190
Ethyl hexanoate	E15	123–66-0	2.5	362	110	113 (8)	97
Ethyl 2-hexenoate	E16	1552-67-6	0.3	5.8	1.2	1.8 (–)	1.6
Ethyl cyclohexanoate	E17	3289-28-9	1.3	38	2.0	7.8 (–)	10
(*E,E*)-Ethyl 2,4-hexadienoate	E18	2396-84-1	6.0	4946	58	1049 (–)	1593
Ethyl 2,4-hexadienoate	E19	110,318–09-7	3.5	5073	245	884 (–)	1390
Ethyl heptanoate	E20	106–30-9	0.1	4.0	0.3	0.7 (–)	1.0
Ethyl octanoate	E21	106–32-1	7.3	442	58	131 (26)	148
Ethyl decanoate	E22	110–38-3	2.3	30	11	13 (0.1)	9.8
Ethyl 9-decenoate	E23	67,233–91-4	1.1	21	14	12 (0.1)	7.8
Ethyl nonanoate	E24	123–29-5	1.8	20	3.3	5.5 (–)	5.6
Ethyl cinnamate	E25	103–36-6	2.0	13	9.3	6.7 (6.7)	4.3
Ethyl hydrocinnamate	E26	2021-28-5	1.2	4.5	4.1	3.1 (0.2)	1.5
Ethyl benzoate	E27	93–89-0	2.0	12	7.5	6.2 (<0.01)	4.2
Ethyl benzeneacetate	E28	101–97-3	2.7	49	18	20 (0.5)	12
Ethyl salicylate	E29	118–61-6	0.1	1.1	0.8	0.6 (–)	0.4
Ethyl (*L*)-(−)-lactate	E30	687–47-8	0.4	3452	188	521 (–)	951
Ethyl lactate	E31	97–64-3	467	54,824	3533	10,543 (0.2)	16,265
Ethyl 2-furoate	E32	614–99-3	1.5	94	9.9	20 (0.02)	26
Diethyl succinate	E33	123–25-1	1411	29,523	12,446	12,909 (0.04)	10,571
Ethyl 3-methylbutyl succinate	E34	28,024–16-0	27	56	47	46 (–)	25
Monoethyl succinate	E35	1070-34-4	99	891	131	283 (–)	258
Diethyl propanedioate	E36	105–53-3	24	1220	1190	657 (–)	609
Diethyl pentanedioate	E37	818–38-2	49	324	304	191 (–)	134
Ethyl myristate	E38	124–06-1	11	494	18	63 (–)	132
Methyl 2-methylbutanoate	E39	868–57-5	2.2	7.5	5.6	5.1 (–)	2.9
Methyl hexanoate	E40	106–70-7	1.0	9.7	3.2	4.0 (–)	3.1
Methyl octanoate	E41	111–11-5	0.1	2.2	0.8	0.9 (<0.01)	0.7
Methyl decanoate	E42	110–42-9	6.7	6.9	6.8	6.8 (–)	3.5
Methyl salicylate	E43	119–36-8	1.2	5.5	3.4	3.5 (0.05)	1.1
(*E*)-Methyl cinnamate	E44	1754-62-7	4.4	17	9.2	8.7 (–)	3.9
Monomethyl succinate	E45	3878-55-5	1.7	611	265	265 (–)	205
Methyl 2,4-hexadienoate	E46	1515-80-6	4.2	22	7.3	10 (–)	6.4
Higher alcohols							
Isobutanol	H1	78–83-1	137	17,816	2258	3124 (0.1)	4661
2,3-Butanediol	H2	513–85-9	5662	20,398	12,984	12,164 (0.1)	5501
1-Pentanol	H3	71–41-0	Tr[b]	688	151	190 (<0.01)	200
Isopentanol	H4	123–51-3	11,337	171,011	92,018	77,847 (1.9)	61,299
Hexanol	H5	111–27-3	ND[c]	793	ND	147 (0.02)	240
(*Z*)-3-Hexen-1-ol	H6	928–96-1	ND	1235	Tr	262 (0.7)	415
2-Ethyl-1-hexanol	H7	104–76-7	Tr	62	14	17 (–)	16
Octanol	H8	111–87-5	3.1	12	6.9	7.5 (<0.01)	2.3
(*Z*)-6-Nonenol	H9	35,854–86-5	5.3	94	14	31 (–)	31
Benzyl alcohol	H10	100–51-6	29	1398	78	182 (<0.01)	369
2-Phenylethanol	H11	60–12-8	2096	75,083	14,279	24,442 (1.7)	24,818
2-Furanmethanol	H12	98–00-0	Tr	873	66	126 (–)	226
Methionol	H13	505–10-2	Tr	682	101	209 (0.2)	246
Terpenes							
D-Limonene	T1	5989-27-5	Tr	59	5.3	14 (–)	18
β-Ocimene	T2	13,877–91-3	Tr	94	33	38 (2.7)	32
β-Phellandrene	T3	555–10-2	Tr	1.0	0.7	0.6 (–)	0.3
γ-Terpinene	T4	99–85-4	Tr	0.5	0.0	0.2 (–)	0.2
p-Cymene	T5	99–87-6	0.4	3.3	1.4	1.4 (–)	0.9
o-Cymene	T6	527–84-4	0.3	6.2	2.6	2.5 (–)	1.7
Terpinolene	T7	586–62-9	Tr	1.5	0.4	0.5 (–)	0.4
β-Caryophyllene	T8	87–44-5	Tr	8.0	Tr	0.9 (–)	2.2
(*Z*)-Rose oxide	T9	16,409–43-1	0.2	2.7	0.6	0.9 (4.7)	0.8
(*E*)-Rose oxide	T10	876–18-6	0.2	2.7	0.5	0.9 (4.3)	0.8
Linalool	T11	78–70-6	Tr	67	1.1	7.2 (0.3)	18
Terpinen-4-ol	T12	562–74-3	Tr	22	2.0	4.2 (0.02)	6.2

(*continued on next page*)

Table 2 (*continued*)

Compound	No.	CAS	Minimum	Maximum	Median	Mean (OAV)	SD[a]
α-Terpineol	T13	98–55-5	1.4	149	12	28 (0.1)	40
(+)-Menthol	T14	15,356–60-2	Tr	2.2	0.4	0.5 (–)	0.6
(−)-Menthol	T15	2216-51-5	Tr	0.5	0.3	0.2 (–)	0.2
Myrtenol	T16	515–00-4	0.4	2.6	1.2	1.5 (–)	0.9
Citronellol	T17	106–22-9	0.9	66	12	17 (0.02)	18
(*Z*)-Nerol	T18	106–25-2	Tr	1.1	0.2	0.5 (<0.01)	0.5
Citral	T19	5392-40-5	1.9	75	22	28 (–)	24
Citronellyl acetate	T20	150–84-5	0.1	1.4	0.7	0.7 (–)	0.3
Bornyl acetate	T21	76–49-3	Tr	11	9.0	6.8 (–)	5.0
Geranyl acetate	T22	105–87-3	0.3	1.0	0.6	0.6 (<0.01)	0.2
Norisoprenoids							
6-Methyl-5-heptene-2-one	N1	110–93-0	0.7	8.0	1.5	2.5 (–)	2.3
β-Ionone	N2	14,901–07-6	0.03	2.0	0.1	0.3 (3.4)	0.5
β-Damascenone	N3	23,696–85-7	0.2	33	0.2	2.9 (58)	9.1
Acids							
Acetic acid	A1	64–19-7	2918	20,653	5605	8121 (0.04)	6242
Isobutanoic acid	A2	79–31-2	ND	4868	Tr	764 (0.3)	1336
Hexanoic acid	A3	142–62-1	173	1852	370	541 (1.3)	468
Ethylhexanoic acid	A4	149–57-5	27	617	67	115 (–)	158
(*E,E*)-2,4-Hexadienoic acid	A5	110–44-1	369	140,873	7878	38,767 (–)	48,226
Octanoic acid	A6	124–07-2	351	3322	680	1156 (2.3)	895
Decanoic acid	A7	334–48-5	Tr	926	78	215 (0.2)	285
Benzoic acid	A8	65–85-0	85	516	172	229 (0.2)	155
Aldehydes							
2-Methylpentanal	AD1	123–15-9	Tr	1665	995	1047 (–)	557
Octanal	AD2	124–13-0	Tr	4.0	2.4	2.4 (1.0)	1.1
(*E*)-2-Octenal	AD3	2548-87-0	2.2	38	12	14 (4.6)	8.6
Nonanal	AD4	124–19-6	1.8	14	5.6	6.1 (0.4)	3.7
(*2E,4E*)-Decadienal	AD5	25,152–84-5	4.0	27	6.6	11 (–)	7.5
Benzaldehyde	AD6	100–52-7	3.4	407	44	71 (0.02)	108
Phenylacetaldehyde	AD7	122–78-1	8.5	64	23	25 (25)	16
Furfural	AD8	98–01-1	108	5732	1167	1231 (0.1)	1432
5-Methylfurfural	AD9	620–02-0	ND	Tr	ND	Tr	–
5-Hydroxymethylfurfural	AD10	67–47-0	827	2600	1649	1629 (0.02)	617
Volatile phenols							
Guaiacol	V1	90–05-1	Tr	9.7	2.4	2.9 (0.3)	2.3
4-Methylguaiacol	V2	93–51-6	Tr	2.2	0.7	1.0 (0.05)	0.6
4-Ethylguaiacol	V3	2785-89-9	Tr	186	5.8	23 (1.1)	50
4-Vinylguaiacol	V4	7786-61-0	19	1281	309	375 (0.3)	322
Phenol	V5	108–95-2	1.0	7.2	1.9	2.5 (–)	1.7
4-Ethylphenol	V6	123–07–9	2.4	1819	14	181 (0.4)	497
3-Ethylphenol	V7	620–17-7	Tr	2.9	0.9	1.5 (–)	0.9
Eugenol	V8	97–53-0	9.4	797	41	99 (17)	211
Isoeugenol	V9	97–54-1	0.3	4.6	4.0	2.4 (0.4)	2.0
2,4-Di-tert-butylphenol	V10	96–76-4	309	773	493	544 (–)	151
Lactones							
γ-Hexalactone	L1	695–06-7	Tr	32	2.1	8.8 (<0.01)	11
Whiskey lactone	L2	39,212–23-2	15.4	18	17	17 (0.1)	1.0
γ-Nonalactone	L3	104–61-0	Tr	10	10.0	5.5 (0.2)	5.1
γ-Decalactone	L4	706–14-9	ND	Tr	ND	Tr	–
δ-Hexalactone	L5	823–22-3	ND	Tr	ND	Tr	–
Others							
Maltol	O1	118–71-8	10.0	17	10	13 (<0.01)	3.4
3-Isobutyl-2-methoxypyrazine	O2	24,683–00-9	ND	Tr	ND	Tr	–
5-Ethyl-2,3-dimethylpyrazine	O3	15,707–34-3	11.7	48	17	22 (–)	10
o-Xylene	O4	95–47-6	0.4	4.2	1.7	1.6 (–)	1.3
Styrene	O5	100–42-5	0.3	6.4	0.6	1.1 (–)	1.6
Naphthalene	O6	91–20-3	0.1	0.8	0.7	0.4 (–)	0.3
1,2,4-Trimethylbenzene	O7	95–63-6	2.5	10.0	7.9	5.8 (–)	2.9
3,4-Dimethylstyrene	O8	27,831–13-6	1.6	4.5	2.4	2.9 (–)	1.1
2-Nonanone	O9	821–55-6	Tr	4.7	0.7	1.5 (–)	1.5
4-Methyl-1-pentanoylbenzene	O10	1671–77-8	Tr	29	12	16 (–)	10
2,6,8-Trimethyl-4-nonanone	O11	123–18-2	Tr	1.2	0.6	0.5 (–)	0.4
(*3E,5E*)-3,5-Heptadien-2-one	O12	18,402–90-9	ND	24	Tr	2.9 (–)	7.3

[a] Standard deviation.
[b] Tr, below LOQ.
[c] ND, not detected (below LOD).

several terpenes (*α*-terpineol (T13), (*Z*) rose oxide (T9), (*E*)-rose oxide (T10), *β*-ocimene (T2), and linalool (T11)), volatile phenols (4-vinylguaiacol (V4), 4-methylguaiacol (V2), isoeugenol (V9), and eugenol (V8)), esters (ethyl cinnamate (E25) and ethyl hydrocinnamate (E26)), aldehydes (nonanal (AD4) and phenylacetaldehyde (AD7)), as well as lactones (*γ*-nonalactone (L3) and whiskey lactone (L2)). Terpenes have been widely recognized as the main contributors to the 'floral' aroma attribute in wine (Black et al., 2015; Yang et al., 2019). In our recent study, we have also confirmed that terpenes could significantly enhance the 'honey' aroma in icewine (Qian et al., 2024). Lactones are often associated with coconut, peach, apricot, and dried fruit aromas in various wines (Allamy et al., 2018; Qian et al., 2020). In addition,

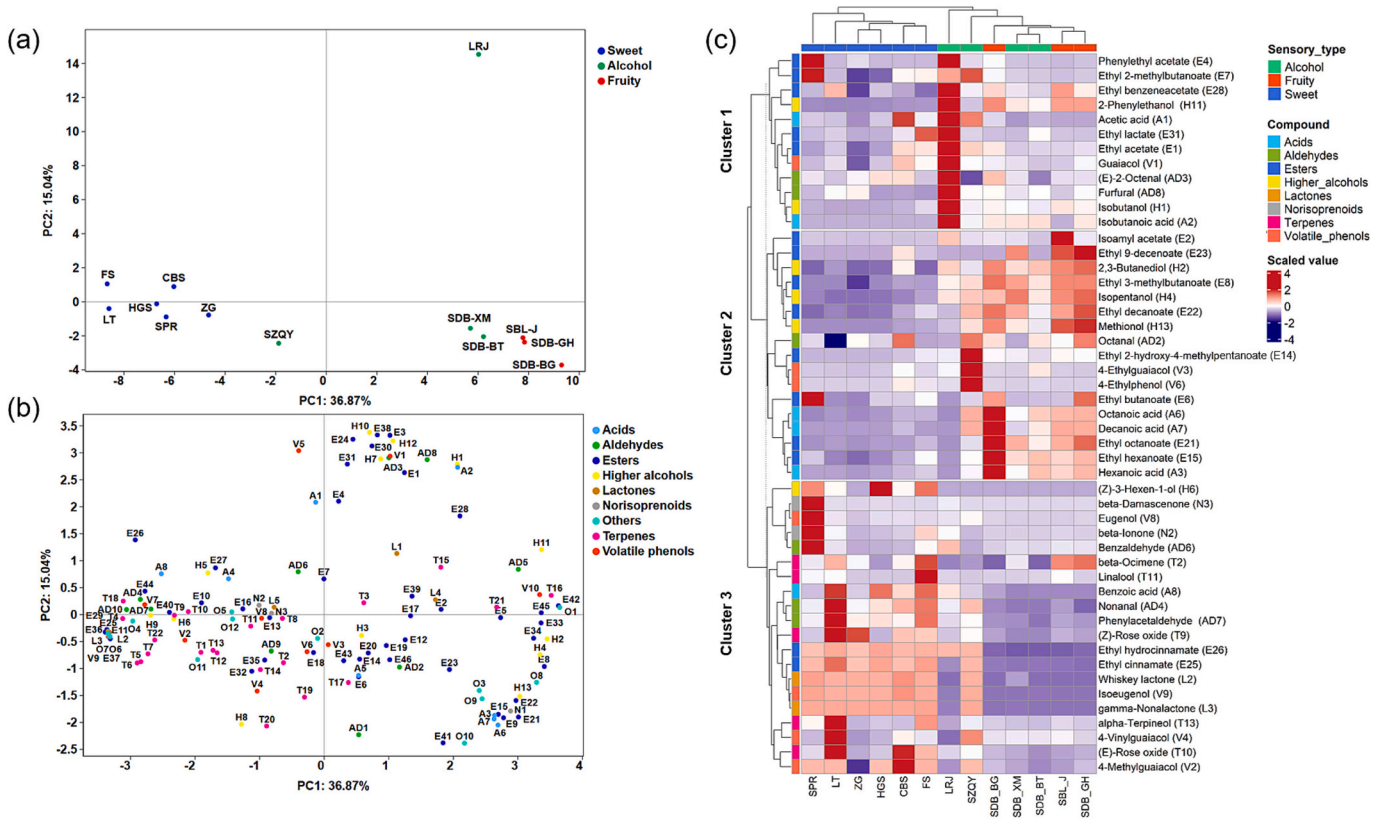

Fig. 4. PCA scores plot (a) and loadings plot (b) of volatile compounds in 13 hawthorn wines; (c) Heatmap analysis based on the concentrations of the volatile compounds with odor activity values (OAVs) higher than 0.1.

lactones, particularly γ-nonalactone, were reported to interact synergistically with furanones, significantly enhancing the sensory perception of the 'dried fruit' and 'caramel' aromas (Allamy et al., 2018; Qian et al., 2024). Several GC-O studies have revealed that phenylacetaldehyde was a potent odorant that contributed to the 'honey' aroma in some sweet wines (Allamy et al., 2018; Lan et al., 2019; Sarrazin et al., 2007). Therefore, it was speculated that these volatile compounds mentioned above may play a crucial role in the 'sweet/honey/hawthorn' attributes of hawthorn wines.

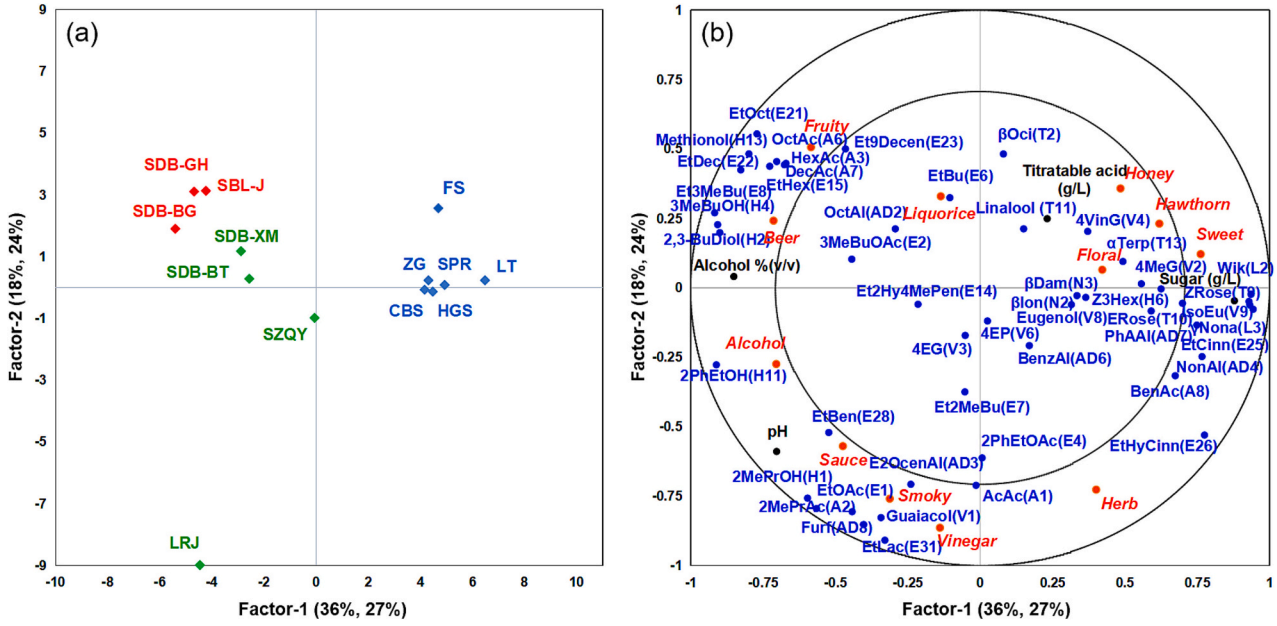

Fig. 5. Plots for partial least-squares regression for the aroma attributes using the chemical compositional data for the 13 hawthorn wines. (a) The 'Sweet' type wines are shown in blue, the 'Fruity' type wines are shown in red, and the 'Alcohol' type wines are shown in green. (b) The x loading of aroma compounds and physicochemical parameters are shown in blue and black, respectively, and the y loadings (sensory attributes) are shown in red. The abbreviation of aroma compounds are listed in Table S7. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

3.4. Relating wine composition and sensory data by PLS regression

Volatile composition, basic chemical parameters and QDA data determined for 13 hawthorn wines were analyzed through PLS regression to explore their underlying relationships. All the volatile compounds with OAVs higher than 0.1 were included in the PLS regression. Two components were chosen to build the PLS model, and the first two components explained 51 % of the variation in sensory attributes (y-variables) and 54 % of the variation in wine composition (x-variables).

Fig. 5a illustrates the distribution of the 13 hawthorn wine samples, while their corresponding sensory attributes are highlighted in red in Fig. 5b. PLS component 1 mainly contributed to the spread of samples (score plot, Fig. 5a) on the left side of the plot mainly based on the intensity of 'fruity', 'beer' and 'alcohol' attributes (correlation loadings plot, Fig. 5b), as opposed to samples on the right which generally had more 'sweet', 'hawthorn', 'honey' and 'floral' characters. Except for LRJ wine in the lower left quadrant, other wines were generally located in the upper quadrants of the plot (Fig. 5a) and could be separated into three main subsets. Six 'Sweet' type wines located in the top right quadrant (Fig. 5a) mostly exhibited more 'sweet', 'hawthorn', 'honey' and 'floral' characters, and three 'Fruity' type wines in the top left quadrant had obviously 'fruity' aroma. Those in the middle left position were mostly perceived as having 'beer' and 'alcohol' aromas. The LRJ wine in the direction of negative PLS component 2 displayed 'vinegar', 'smoky' and 'sauce' aromas.

As shown in Fig. 5b, the wine compositions that were plotted in the vicinity of an attribute were positively correlated with that attribute. The most relevant odorants in relation to 'fruity' and 'alcohol' attributes were compounds of fermentation origin (Fig. 5b). The 'fruity' attribute was strongly positively correlated with several fatty acid ethyl esters, such as ethyl hexanoate (E15), ethyl octanoate (E21) and ethyl decanoate (E22), together with linear fatty acids (octanoic acid (A6), hexanoic acid (A3), decanoic acid (A7), which was in good agreement with that reported in other wines (Niu et al., 2019; Qian et al., 2024; San-Juan et al., 2011). The 'alcohol' attribute was associated with alcohol content, as well as a number of higher alcohols, including 2,3-butanediol (H2), isopentanol (H4) and 2-phenylethanol (H11). With the exception of 2-phenylethanol, reminiscent of rose, higher alcohols are usually described as smelling of fusel, solvent, or malt (Cameleyre et al., 2015). Taken in isolation, ethanol is described as having a 'solvent-like' or 'fruity' odor (Waterhouse et al., 2016). Given the broad range of ethanol content in hawthorn wine (4–14 % *v*/v), which differs from the typical ethanol content in dry wines (11–14 % v/v), the ethanol content may also be a critical factor influencing the aroma profile of hawthorn wine. In the present study, the model also showed that 'sweet/honey/hawthorn' aromas were negatively contributed by the alcohol content, suggesting that increasing ethanol concentration may suppress the perception of these 'sweet/honey/hawthorn' aromas. Several studies have reported that increasing ethanol concentration usually decreased the partition coefficient of various volatile compounds, thereby increasing their odor detection thresholds (Grosch, 2001; Villamor & Ross, 2013). As reported by Grosch (2001), using 7 % instead of 10 % ethanol resulted in an increased overall fruity and floral aromas in model wine, and a 10–312 fold increase in thresholds was observed in model wine compared to water. The impact of alcohol content on olfactory thresholds mainly came from two aspects: one was the stimulation of alcohol on human olfactory organs, or its masking effect on the aroma characteristics of compounds, which increased the difficulty of identification; the other was the indirect change in the volatility of compounds by altering the properties of the matrix, leading to an increase or decrease in thresholds (Villamor & Ross, 2013; Waterhouse et al., 2016).

Notably for this study, 'sweet', 'hawthorn', 'honey' and 'floral' attributes were dominant in the upper right quadrant of the plot (Fig. 5b). Similar to previous studies, these 'sweet/honey/dried fruit' aromas often appeared in the opposition to the 'fruity' aroma (Lan et al., 2019; San-Juan et al., 2011). It has been reported that small amounts of honey-smelling compounds seemed to have a positive effect on the perception of 'fruit', but the presence of high amounts made the wine less fruity (San-Juan et al., 2011). The aroma attributes including 'sweet', 'hawthorn', 'honey' and 'floral' were positively associated with several terpenes, such as α-terpineol (T13), (*Z*)-rose oxide (T9) and (*E*)-rose oxide (T10), volatile phenols (isoeugenol (V9), 4-vinylguaiacol (V4), and 4-methylguaiacol (V2)), ethyl cinnamate (E25), aldehydes (nonanal (AD4) and phenylacetaldehyde (AD7)), as well as lactones (γ-nonalactone (L3) and whiskey lactone (L2)). Terpenes such as α-terpineol and rose oxide derived from the fruits can contribute to the floral character of wines (Black et al., 2015; Yang et al., 2019), and have also been demonstrated to be associated with the 'honey' aroma in icewine (Qian et al., 2024). Lactones such as γ-nonalactone and massoia lactone have been frequently reported in relation to the 'dried fruit' and 'caramel' aromas (Allamy et al., 2018; Qian et al., 2020). Phenylacetaldehyde and ethyl cinnamate have also been reported as potent odorants that contributed to the 'honey' or 'sweet' aroma in some sweet wines (Allamy et al., 2018; Lan et al., 2019; Sarrazin et al., 2007). Volatile phenols such as 4-vinylguaiacol and isoeugenol may originate from toasted oak or precursors in the fruit, usually related to smoky, clove, and sweet characters (Lan et al., 2019). The relationships between the above volatile compounds and aroma attributes were well accorded with a PLS model for 'Beibinghong' wines (Lan et al., 2019). In addition, these attributes were found to be strongly positively correlated with the sugar content (Fig. 5b). Consistent with our previous findings, a "salting-out" effect of some terpenes and volatile phenols into the headspace could be observed with increasing sucrose concentration (Fig. 3c). Similar results were obtained by Tsitlakidou et al. (2019), who revealed that as the sugar content increased, some terpenes showed an increased release, and the perceived intensity of "overripe orange" was enhanced. This study gave a preliminary understanding of the aroma profile of Chinese hawthorn wine. However, further studies are needed to verify the relationships between these chemical compositions and aroma attributes, together with their synergistic effects or other interactions.

4. Conclusions

In summary, this was the first detailed study on the chemical and aroma profile of hawthorn wines from China. This study established a sensory lexicon for describing the aromas of hawthorn wines. A total of 72 aroma descriptors were initially generated by Pivot Profile tests. Following sorting and evaluation, 12 aroma descriptors including 'hawthorn', 'alcohol', 'sweet', 'honey', 'fruity', 'herb', 'vinegar', 'sauce', 'liquorice', 'smoky', 'beer' and 'floral' formed the final lexicon list. Based on the lexicon and sensory data, the 13 hawthorn wines represented three different styles. 'Sweet' type was characterized by 'sweet', 'hawthorn' and 'honey' characters; 'Fruity' type was more 'fruity' and weaker 'sweet' characters; 'Alcohol' type had more 'alcohol', 'vinegar', and 'sauce' characters.

Regarding the composition of the volatiles, a total of 129 compounds were identified across 13 hawthorn wines using HS-SPME combined with both GC-Orbitrap-MS and GC-Quadrupole-MS. Based on OAV, volatiles such as β-damascenone, ethyl octanoate, ethyl 2-methylbutanoate, phenylacetaldehyde, and eugenol were deemed to be important. Considering the varying levels of residual sugar in Chinese hawthorn wines, the effect of sugar on the aroma release was also investigated. An increase in sucrose concentration from 8.4 to 82.4 g/L was shown to significantly decrease the release of several esters, and conversely increase the release of some terpenes and volatile phenols.

PLS regression was applied to explore the positive and negative relationships among these wine variables. It was observed that 'sweet', 'hawthorn', and 'honey' attributes were gathered together, in the opposition to the 'fruity' and 'alcohol' aromas. The 'fruity' attribute was strongly positively correlated with several fatty acid ethyl esters and fatty acids, in accord with other studies. While the 'alcohol' attribute

was associated with alcohol content and higher alcohols. The 'sweet', 'hawthorn', and 'honey' aroma attributes were positively correlated with several terpenes, volatile phenols, γ-nonalactone, whiskey lactone, ethyl cinnamate, nonanal and phenylacetaldehyde, together with sugar content. In contrast, these aroma attributes were negatively contributed by alcohol content, suggesting that increasing ethanol concentration may suppress the perception of these 'sweet/honey/hawthorn' aromas. These findings can serve as a foundational reference for evaluating the aroma quality of hawthorn wine products, and provide critical theoretical support for the modulation of aroma profiles and enhancement of product quality. Further work will focus on investigating the regulatory mechanisms of these key aroma compounds during fermentation, such as hawthorn varieties, microbial strains, and other fermentation conditions.

CRediT authorship contribution statement

Xinyue Zhang: Writing – original draft, Visualization, Formal analysis. **Yixin Chen:** Methodology, Formal analysis. **Jiani Liu:** Investigation, Formal analysis. **Yibin Lan:** Writing – review & editing, Project administration. **Xu Qian:** Writing – review & editing, Supervision, Funding acquisition, Conceptualization. **Baoqing Zhu:** Writing – review & editing, Supervision, Project administration, Funding acquisition.

Declaration of competing interest

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

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fochx.2025.102277.

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