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Identification of Key Aroma-Active Compounds in Plum, Jujube, and Grape Extracts via GC×GC-O-TOF-MS, GC-MS, Aroma Recombination and Omission Tests

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Abstract

Fruity natural flavors are widely used in food, cosmetics, and tobacco products, and their flavor profiles directly affect the sensory quality of the final products. However, the volatile organic compound systems in these flavors are highly complex, and severe co-elution among volatile components makes effective component resolution a persistent analytical challenge. In this study, comprehensive two-dimensional gas chromatography–olfactometry–time-of-flight mass spectrometry (GC×GC-O-TOF-MS) was employed to separate and identify volatile odor compounds in three natural fruit flavor extracts: plum, jujube, and grape. A total of 113, 103, and 85 volatile odor compounds were identified from the plum, jujube, and grape extracts, respectively. By progressively adjusting the split ratio, aroma extract dilution analysis was performed, leading to the screening of 14, 11, and 32 aroma-active compounds from the three extracts, respectively. Compounds with high flavor dilution factors were subjected to quantitative analysis, and their odor activity values were calculated. Subsequently, omission and recombination experiments were conducted to confirm the key aroma-active compounds in each extract. The results showed that octanal was a key aroma compound shared by the plum and jujube extracts, while ethyl cinnamate was common to both the plum and grape extracts. Through a molecular sensory science approach and utilizing GC×GC-O-TOF-MS chromatographic separation technology, this study provides a reliable analytical platform for aroma characterization in complex samples. It also establishes a critical theoretical and data foundation for precisely identifying key aroma-active components and implementing targeted aroma modulation to enhance the natural flavor quality of fruits.

Keywords: fruity natural flavors; GC×GC-O-TOF-MS; molecular sensory science; separation; identification

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1. Introduction

Flavors are substances that release specific aromatic components, perceived olfactorily to evoke pleasant sensations, and are classified as natural and synthetic flavors. Natural

flavors are derived from animals and plants, such as flowers, leaves, fruits, resins, or animal secretions. Several plant-derived natural flavors are widely used in foods, beverages, confectionery, as well as in personal care products such as soaps, toothpaste, shampoos [1], and cosmetics (e.g., perfumes) to enhance appeal or mask undesirable tastes or odors. Moreover, plant-based natural flavors are applied as dietary supplements or added to functional foods to enhance nutritional value [2–5]. Natural flavor extracts, concentrated from plant-based aromatic materials and rich in complex volatile and nonvolatile components, serve as crucial, intensely flavored base materials for premium fragrances, cosmetics, and functional foods. The sensory quality of these extracts directly influences the grade, characteristics, and overall quality of downstream products.

The flavor profile serves as a key criterion for assessing the quality of flavor products. Although several studies have focused on the aroma profiles of various fruits, studies specifically addressing the flavor characteristics of fruit extracts remain limited. Plums, grapes, and jujubes are fruits characterized by sweet, sour, and fruity flavors. Hu et al. [6] identified 495 and 466 flavor compounds in the plum cultivars 'Fengtangli' and 'Siyueli,' respectively, and reported that 'Fengtangli' plums exhibited significantly higher relative contents of hydrocarbons, alcohols, ketones, and esters than 'Siyueli' plums. Additionally, 'Fengtangli' plums exhibited more pronounced sensory attributes, including sweet, citrus, herbal, floral, and fruity aromas. H. C. Liu et al. [7] examined plum flavor compounds across three maturity stages, identifying eight shared volatile odor compounds, including benzaldehyde, ethyl acetate, and ethyl propionate. Yue et al. [8] employed gas chromatography–mass spectrometry (GC-MS) to analyze flavor compounds in jujube at different developmental stages and found that aldehydes were the primary volatile aroma compounds, with (*E*)-2-hexenal being particularly abundant. Similarly, R. J. Liu [9] employed GC-MS combined with OAV (odor activity value) analysis and identified aldehydes, ketones, and esters as the main volatile aroma compounds in jujube fruit. The study revealed that the aroma profile was primarily characterized by fruity and fatty notes, with β-damascenone contributing to fruity characteristics and (*E*)-2-oct-en-1-al and nonanal significantly influencing the fatty aromas. Wu et al. [10] analyzed the volatile aroma compounds in three grape varieties with distinct aroma profiles, highlighting esters and aldehydes as key contributors to their fruity notes. Wang et al. [11] analyzed the odor profile of the widely cultivated grape variety 'Queen Nina', identifying benzeneacetaldehyde, 2,5-dimethyl-4-hydroxy-3(2H)-furanone, ethyl octanoate, ethyl valerate, and (*E*,*Z*)-2,4-dodecyl formate as the key volatile compounds associated with fruit ripening. Notably, the chemical composition of fruit extracts is significantly more complex than that of fresh fruit or juice. During concentration, extracts develop a complex matrix in which compounds that are insignificant in fresh fruit may become key contributors to aroma. Therefore, the direct application of analytical approaches and conclusions derived from fresh fruit studies to fruit extracts has inherent limitations.

However, most existing studies rely on conventional one-dimensional gas chromatography techniques, such as gas chromatography–olfactometry–mass spectrometry (GC-O-MS) or gas chromatography–ion mobility spectrometry (GC-IMS), which offer limited separation capacity for complex aroma systems and often result in insufficient data resolution. In contrast, comprehensive two-dimensional gas chromatography–olfactometry–time-of-flight mass spectrometry enables effective separation of complex mixtures by employing two chromatographic columns with different polarities, allowing analytes with similar physicochemical properties to be further resolved based on additional interactions [5]. Owing to its superior separation capabilities, the comprehensive two-dimensional gas chromatography–time-of-flight mass spectrometry (GC×GC-TOF-MS) has proven highly effective for flavor analysis in traditional Chinese medicines [12] and plant essential oils [13]. By separating analytes sequentially across two chromatographic columns with different

polarities, GC×GC-TOF-MS achieves enhanced resolution of compounds with similar physical or chemical properties [14]. This study marks the first application of GC×GC-O-TOF-MS within a comprehensive molecular sensory science workflow to the identification of key aroma compounds in three types of natural fruit-flavored flavorings, enabling high-resolution analysis of complex volatile mixtures.

Although several volatile compounds are present in aroma systems, only a limited subset significantly influences characteristic aromas and determines the overall profile. These compounds are known as key aroma compounds. Studies on fruit-flavored fragrances have predominantly focused on characterizing volatile flavor compounds, frequently overlooking the identification of the aroma-active components. The screening of key aroma compounds necessitates the application of molecular sensory science approaches. Molecular sensory science integrates qualitative, quantitative, and descriptive molecular-level analyses, enabling the accurate reconstruction of flavors and the precise identification of compounds that contribute to aroma. Through continued methodological development, molecular sensory science has become one of the most advanced and systematic analytical frameworks for evaluating the flavor characteristics of fruits and fruit-derived products [15]. The application of molecular sensory science in identifying key aroma compounds provides significant practical benefits for enhancing the flavor profiles of natural fruit-flavored fragrances. Additionally, it establishes a robust scientific foundation to guide both product development and consumer-focused applications.

Therefore, the objectives of the present study were to: (1) screen aroma-active compounds in plum, jujube, and grape extracts using GC×GC-O-TOF-MS combined with FD factor analysis; (2) quantitatively analyze the identified aroma-active compounds in the three extracts using GC-MS; and (3) identify the key aroma-active compounds in plum, jujube, and grape extracts through omission and recombination experiments.

2. Materials and Methods

2.1. Materials and Reagents

Plum, jujube, and grape extracts were sourced from commercial suppliers. Chromatographic-grade methanol (purity $\geq$ 99%) used for the extraction and separation of flavor compounds was obtained from Beijing Mairuida Technology Co., Ltd. (Beijing, China). The internal standard quinoline (98%) was provided by Tokyo Chemical Industry. (Shanghai, China). Chromatographic-grade n-alkanes (C7-C40) for retention index determination were purchased from Sigma-Aldrich (St. Louis, MO, USA). The following reference standards were obtained from Shanghai Yuanye Biotechnology Co., Ltd. (Shanghai, China): octanal (99%), α-angelica lactone (97%), furfural (99%), benzaldehyde (99%), linalool (98%), 5-methyl-2-furaldehyde (98%), benzeneacetaldehyde (98%), 5-methyl-2-furanmethanol (95%), methyl phenylacetate (99%), guaiacol (98%), phenethyl alcohol (99%), pyrrole-2-carboxaldehyde (98%), ethyl cinnamate (98%), eugenol (98%), hexanal propylene glycol acetal (98%), isovaleraldehyde propylene glycol acetal (98%), ethyl hexanoate (99%), 1-octen-3-ol (98%), estragole (98%), geranylacetone (98%), 2-acetylpyrrole (99%), 4-ketoisophorone (98%), γ-nonalactone (98%), and γ-decalactone (99%).

2.2. Sensory Evaluation

The sensory evaluation was performed according to a previously described method [16] with slight modifications. The sensory panel was recruited from the Molecular Sensory Science Laboratory at Beijing Technology and Business University. The panel consisted of 12 members (6 male and 6 female) aged 22–30 years. Panelists underwent training in sensory evaluation techniques and descriptive quantitative analysis before the assessments. And all panelists signed an informed consent form, which was approved by the University

Scientific Research Ethics Committee of BTBU (2025-50). During the evaluation, panelists first described the odors of the natural flavor raw materials. Based on a subsequent panel discussion, the six most frequently cited sensory descriptors were selected, including "sweet," "fruity," "caramel," "floral," "licorice," and "sour." Sensory intensity was assessed using a 5-point scale, where "0" indicated an imperceptible aroma, and "5" indicated the highest intensity. The final results were calculated as the average scores for all sensory descriptors and presented as a radar chart.

2.3. Solid-Phase Microextraction (SPME)

A 1 g sample portion was placed into a 20 mL headspace vial, and 1 mL of saturated sodium chloride solution was added to facilitate the complete release of volatile aromas. Then, 1 μL of quinoline (1.093 μg/μL) was added as an internal standard. The vial was immediately sealed and equilibrated in a 60 °C water bath for 20 min. After equilibration, the SPME fiber was inserted into the headspace vial, positioned above the sample surface without touching the liquid, and allowed to adsorb volatile compounds at 60 °C for 40 min. The fiber was then retracted and inserted into the GC×GC-O-TOF-MS injection port, where desorption occurred at 230 °C for 5 min.

2.4. Comprehensive Two-Dimensional Gas Chromatography–Olfactometry–Time-of-Flight Mass Spectrometry (GC×GC-O-TOF-MS) Analysis

Volatile compounds in the three extracts were analyzed using an Agilent 8890A-5977B GC×GC-O-TOF-MS system (Agilent Technologies Co., Ltd., Shanghai, China) coupled with a Sniffer 9100 olfactometer (Gerstel Co., Ltd., Shanghai, China). The analysis was performed under the following conditions:

GC conditions: The system was equipped with two columns: a first-dimension DB-WAX polar column (60 m × 0.25 mm × 0.25 μm) and a second-dimension DB-17 ms mid-polarity column (1.85 m × 0.18 mm × 0.18 μm). An SSM1800 comprehensive (Xuejing Electronic Technology Co., Ltd., Shanghai, China) two-dimensional solid-state modulator controlled the heating and cooling stages between the columns, with the cold zone set at −51 °C and a modulation period of 5 s. The oven temperature program was as follows: an initial temperature of 40 °C held for 1 min, followed by a ramp of 2 °C/min to 230 °C, which was then held for 3 min. Samples were injected in splitless mode with helium as the carrier gas at a constant flow rate of 1 mL/min.

MS conditions: Mass spectrometry was performed using an electron impact (EI) ion source with 70 eV electron energy. The ion source temperature was 230 °C, the transfer line temperature was 280 °C, and the quadrupole was maintained at 150 °C. A solvent delay of 5 min was applied. Full-scan mode was used, with a mass range of m/z 35–450.

Olfactometry conditions: The transfer line to the olfactometry port was maintained at 200 °C. Humidified air was supplied to the sniffing port by bubbling nitrogen through a vial of ultrapure water. Olfactometry analysis was performed by three trained panelists, who recorded the perceived aroma attributes, intensities, and corresponding retention times.

2.5. Aroma Extract Dilution Analysis (AEDA)

Sample dilution was performed by adjusting the split ratio at the GC injector, with aromas diluted according to a geometric progression of 3^n. Three experienced sensory panelists assessed the aromas at the olfactometry port, recording the retention time, perceived aroma characteristics, and intensity for each compound. An aroma-active compound was considered present in the sample if it was detected by at least two evaluators. The FD factor of a compound was defined as the highest dilution at which its aroma could still be perceived. A higher FD factor indicates a greater contribution of the compound to the overall flavor profile of the sample.

2.6. Qualitative Analysis

Volatile aroma compounds were identified by integrating three complementary approaches: mass spectral matching, comparison of retention indices (RIs), and olfactometry (O) detection.

2.7. Quantitative Analysis

2.7.1. Semi-Quantitative Analysis

A semi-quantitative method using an internal standard was employed, with quinoline serving as the internal standard. The concentration of each target compound (Cx) was calculated using the following formula:

$$Cx = \frac{Ax}{Ai} \times f \times Ci$$

where Cx represents the concentration of the target compound, Ci represents the concentration of the internal standard, Ax represents the peak area of the target compound, Ai represents the peak area of the internal standard, and f represents the correction factor.

2.7.2. Precise Quantitative Analysis

An internal standard calibration curve method was employed for the accurate quantification of aroma-active compounds with FD $\geq$ 3 in the three extracts. Mixed standard stock solutions of the aroma compounds were prepared at different concentration levels, with quinoline added as the internal standard for correction. Based on the selected ion monitoring (SIM) mode of the GC-MS system, standard curves were constructed by plotting the peak area ratio of the target compound to the internal standard (Y-axis) against the corresponding concentration ratio (X-axis) [17].

2.8. Calculation of Odor Activity Values (OAVs)

The OAV quantifies the contribution of an aroma-active compound to the overall flavor [18]. The OAV is defined as the ratio of the concentration of the compound in the sample to its odor threshold. Compounds with an OAV $\geq$ 1 are considered potential contributors to the aroma profile, and the relative contribution is proportional to the OAV [19]. The OAV was calculated using the following formula:

$$OAV = \frac{C}{OT}$$

where C represents the concentration of the aroma compound in the sample, and OT represents the odor threshold of the compound.

2.9. Aroma Recombination and Omission Experiments

Recombination models were prepared by mixing aroma-active compounds with OAV $\geq$ 1 into a 1,2-propanediol matrix at their accurately determined concentrations. Due to its high solubility and chemical stability, 1,2-propanediol was selected, allowing the aroma-active compounds to mix uniformly and form a stable system. Sensory evaluations were performed on the three extracts and their corresponding recombination models.

For the omission experiments, mixture models were prepared by systematically omitting one aroma-active compound at a time from the complete set of identified compounds in each extract. Differences between the omission models and the complete recombination model were evaluated using triangle tests in accordance with the Chinese National Standard GB/T 12311-2012 [20]. Specifically, if 8, 9, or 10 out of 12 sensory panelists correctly identified a difference between the samples, the result was considered significant ($p \leq 0.05$),

highly significant ($p \leq 0.01$), or extremely significant ($p \leq 0.001$), respectively. If fewer than 8 panelists detected a difference, the result was considered non-significant.

2.10. Data Analysis

All experiments were performed in triplicate, and results are expressed as mean ± standard deviation. Statistical analyses were performed using Microsoft Excel 2023, and graphs were generated using Origin 2023.

3. Results and Discussion

3.1. Sensory Evaluation Analysis

The sensory profiles of the plum, jujube, and grape extracts were evaluated, and the results are shown in Figure 1. The plum extract exhibited pronounced sweet and sour characteristics. The jujube extract was characterized primarily by sweet and fruity notes. The grape extract also showed significant sweet and fruity notes. Among the three extracts, the grape extract exhibited the most pronounced fruity characteristics.

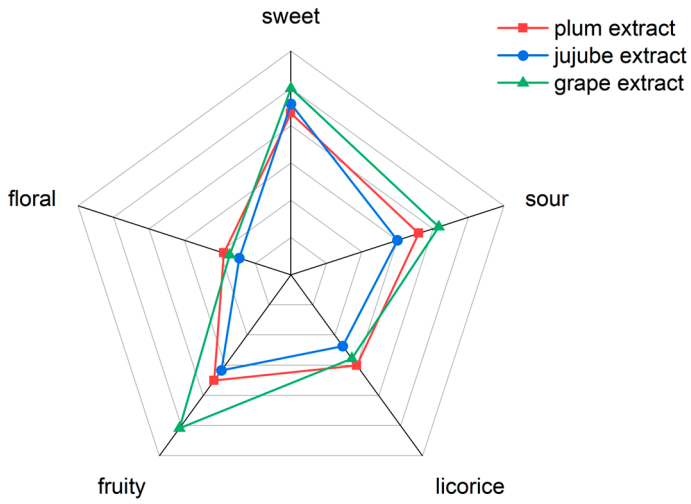


Figure 1. Sensory evaluation scores of three fruity natural flavor extracts.

3.2. Analysis of Volatile Aroma Compounds in the Three Extracts

A total of 188 volatile odor compounds were identified in the plum, jujube, and grape extracts using GC×GC-O-TOF-MS (Table A1 and Figure 2), including 44 esters, 35 aldehydes, 29 alcohols, 29 ketones, 22 heterocyclic compounds, 7 acids, 6 phenols, 5 alkenes, and 11 other compounds. Among these, 113 volatile odor compounds were identified in the plum extract, with alcohols, aldehydes, and esters being the dominant classes. Although alcohols and aldehydes were present in similar numbers, aldehydes exhibited significantly higher concentrations than alcohols. Notably, 5-methyl-2-furfural exhibited the highest concentration (111.099 μg/g), followed by 5-hydroxymethylfurfural (40.559 μg/g). A total of 103 volatile odor compounds were identified in the jujube extract, predominantly comprising aldehydes, esters, and heterocyclic compounds. The grape extract contained 85 volatile aroma compounds, mainly esters, aldehydes, and ketones. Esters were the most abundant class in the grape extract, with ethyl palmitate exhibiting the highest concentration (34.961 μg/g), which contributed to its characteristic fruity aroma.

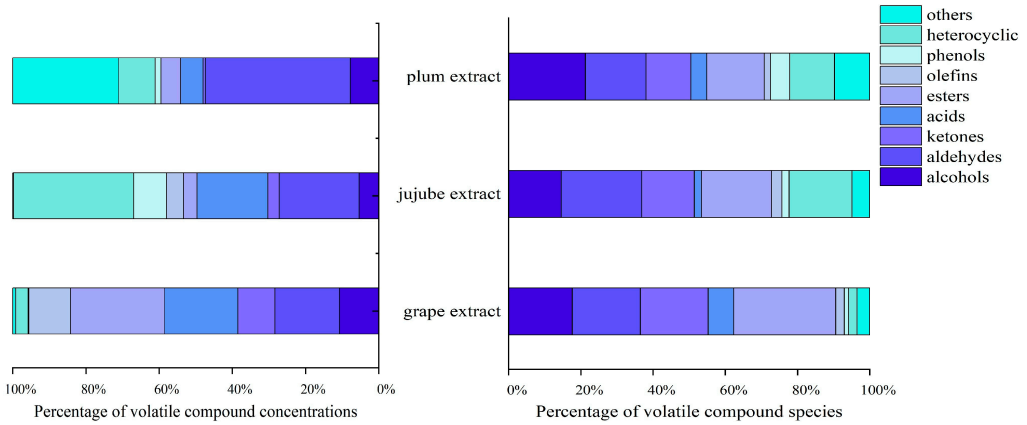


Figure 2. Comparison of volatile compound concentrations (**left**) and species (**right**) in three extracts.

3.3. Analysis of Aroma-Active Compounds in the Three Extracts

Aroma-active compounds directly influence the characteristic flavor profile of food products [21]. AEDA is widely used to evaluate the contribution of individual aroma compounds to the overall aroma, with higher FD values indicating a greater contribution of a given compound to the aroma intensity of the sample [22]. In total, 51 aroma-active compounds were identified across the three extracts (Table A2), comprising 15 aldehydes, 11 alcohols, 11 esters, 5 ketones, 4 acids, 2 phenols, 2 heterocyclic compounds, and 1 compound classified as other. Among the extracts, 14, 11, and 32 aroma-active compounds were identified in the plum, jujube, and grape extracts, respectively, indicating that the grape extract exhibited a more diverse profile.

3.3.1. Aldehyde

In the plum extract, two aldehydes with relatively high FD factors were identified: pyrrole-2-carboxaldehyde (FD = 27) and benzeneacetaldehyde (FD = 9). The compound pyrrole-2-carboxaldehyde is associated with a musty odor, whereas benzeneacetaldehyde contributes a sweet aroma. In the jujube extract, two aldehydes with high FD factors were identified: benzeneacetaldehyde (FD = 9) and octanal (FD = 3). Octanal is characterized by a waxy odor. Jia et al. [23] reported that octanal is a key aroma-active compound in dried jujube. In the grape extract, isovaleraldehyde propylene glycol acetal exhibited a significantly high FD factor of 729 and was identified as a major contributor to the fruity note.

3.3.2. Alcohols

Three alcohols with relatively high FD factors (FD = 9) were identified in the plum extract: linalool, 5-methyl-2-furanmethanol, and phenylethyl alcohol. Both linalool and phenylethyl alcohol are associated with rosy and floral notes, whereas 5-methyl-2-furanmethanol contributes a sweet aroma to the plum extract. Linalool has previously been confirmed as an aroma-active compound in plum [24]. In the jujube extract, alcohols with higher FD factors also include 5-methyl-2-furanmethanol. In the grape extract, 1-octen-3-ol exhibited a high FD factor (FD = 9) and is characterized by mushroom-like and earthy notes. It is generally formed via the lipoxygenase-mediated oxidation of linoleic acid [25].

3.3.3. Esters

The ester with the highest FD factor identified in both the plum and grape extracts was ethyl cinnamate (FD = 729). Ethyl cinnamate, which is characterized by a sweet aroma, was the dominant aroma-active compound in both extracts, consistent with previous findings [26]. In the jujube extract, the ester compounds with high FD factors were

γ-decalactone (FD = 27), γ-nonanolactone (FD = 9) and coconut aldehyde (FD = 9). γ-Decalactone imparts a pronounced fruity aroma and was the major contributor to the overall aroma of the jujube extract, whereas γ-nonanolactone and coconut aldehyde contributed sweet notes to the aroma profile.

3.3.4. Heterocyclic Compounds

No heterocyclic compounds with high FD factors were detected in the plum and jujube extracts. In contrast, a high-FD heterocyclic compound, 2-acetylpyrrole (FD = 243), was identified in the grape extract and is characterized by a musty odor.

3.3.5. Phenols

Two phenolic compounds, guaiacol and eugenol, were identified in the plum extract, each exhibiting a high FD factor of 243. Guaiacol is characterized by a woody aroma, while eugenol has a sweet note. Guaiacol has been previously identified in plum [27], and eugenol has been confirmed as the primary aroma-active compound in plum, exhibiting an OAV greater than 1 [24]. In contrast, no phenolic compounds with high FD factors were detected in the jujube or grape extracts.

3.3.6. Other Compounds

No aroma-active compounds with high FD factors in the "Other" category were detected in the plum or jujube extracts. In contrast, estragole exhibited a high FD factor (FD = 729). Estragole, characterized by a sweet aroma, was a major contributor to the overall aroma profile of the grape extract, and its presence in grape skin has been previously reported [28].

3.4. Quantification of Aroma-Active Compounds and Calculation of OAVs

Aroma-active compounds with FD $\geq$ 3 in the three extracts were accurately quantified using the internal standard calibration curve method, and their OAVs were calculated (Table A3). The OAV represents the contribution of an aroma-active compound to the overall flavor rather than its concentration alone [29]. Compounds with OAV $\geq$ 1 are considered significant contributors to the aroma of the three extracts.

In the plum extract, 14 aroma-active compounds had OAV $\geq$ 1. Among them, methyl phenylacetate exhibited the highest OAV (502,747), followed by 5-methyl-2-furanmethanol (16,749), both of which primarily contributed sweet notes. Methyl phenylacetate has been previously detected in fruit pomace at concentrations exceeding its odor threshold [30]. In the jujube extract, six aroma-active compounds showed OAV $\geq$ 1, with 5-methyl-2-furanmethanol exhibiting the highest OAV (39,958). The compound 5-methyl-2-furanmethanol, which is formed during heating, was a major contributor to the sweet aroma of the jujube extract. In the grape extract, eight aroma-active compounds had OAV $\geq$ 1. Isovaleraldehyde propylene glycol acetal had the highest OAV (57,564,641), followed by 4-methyl-2-pentyl-1,3-dioxolane (355,960), both of which enhance the characteristic fruitiness of the grape extract.

3.5. Verification by Aroma Recombination and Omission Experiments

3.5.1. Aroma Recombination Experiment

Aroma recombination experiments are commonly used to validate the accuracy of identified key aroma-active compounds [31]. Recombination models were prepared by mixing compounds with OAV $\geq$ 1. The concentrations of aroma-recomposed compounds are presented in Table 1. As shown in Figure 3, the aroma profiles of the recombination models closely resembled those of the original extracts, indicating that the recombination models successfully simulated the overall flavor characteristics of the extracts.

Table 1. Concentration of aroma-recomposed compounds.

No.	Compounds [a]	Concentration (μg/g)
Plum extract		
1	Octanal	17.917 ± 1.327
2	Angelica lactone	454.138 ± 36.520
3	Furfural	297,076.482 ± 24,444.384
4	Benzaldehyde	1815.849 ± 218.245
5	Linalool	672.494 ± 73.571
6	5-Methyl-2-furanmethanol	147.397 ± 18.844
7	Benzeneacetaldehyde	521.623 ± 63.830
8	5-Methyl-2-furfural	47,857.922 ± 5856.231
9	Methyl phenylacetate	221.712 ± 22.081
10	Guaiacol	542.220 ± 72.182
11	Phenethyl alcohol	986.834 ± 59.631
12	Pyrrole-2-carboxaldehyde	2468.591 ± 225.528
13	Ethyl cinnamate	55.293 ± 8.569
14	Eugenol	28.572 ± 4.464
Jujube extract		
1	Octanal	90.921 ± 6.82
2	Benzeneacetaldehyde	440.280 ± 46.822
3	γ-Decalactone	65.479 ± 8.303
4	γ-Nonanolactone	289.821 ± 39.777
5	Ketoisophorone	146.435 ± 16.183
6	5-Methyl-2-furanmethanol	351.631 ± 30.605
Grape extract		
1	4-Methyl-2-pentyl-1,3-dioxolane	3210.760 ± 259.337
2	Isovaleraldehyde propylene glycol acetal	9958.683 ± 700.445
3	Ethyl caproate	2225.610 ± 181.657
4	Octanal	72.586 ± 10.384
5	1-Octen-3-ol	1882.864 ± 238.500
6	Estragole	337.489 ± 31.578
7	Geranylacetone	2064.986 ± 169.471
8	Ethyl cinnamate	728.362 ± 160.313
9	Artemisia ketone	337.489 ± 31.578

[a]: aroma-active compounds with OAV greater than 1.

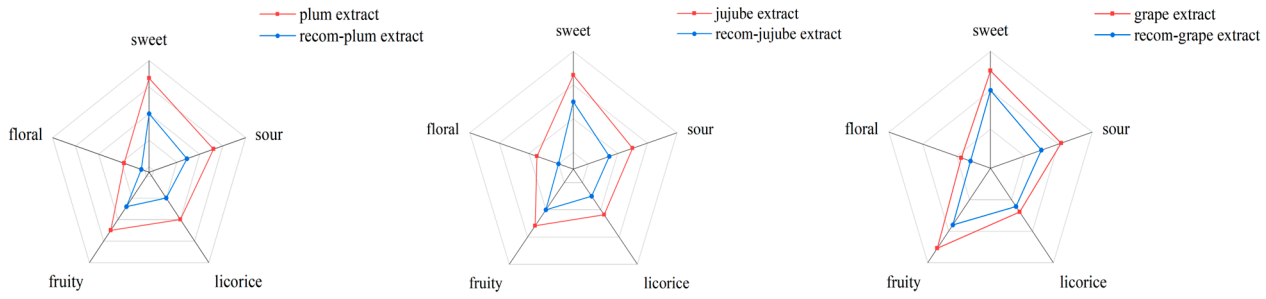


Figure 3. Sensory evaluation scores of three fruity natural flavor extracts and their aroma recombination model.

3.5.2. Aroma Omission Experiment

Aroma omission experiments were conducted to evaluate the contribution of major aroma-active compounds to the overall odor profile [19]. The results of the omission experiments are presented in Table 2.

Table 2. Triangle test results by omission experiments.

No.	Compounds [a]	*n* [b]	Significant [c]
Plum extract			
1	Octanal	8	*
2	Angelica lactone	8	*
3	Furfural	4	-
4	Benzaldehyde	3	-
5	Linalool	8	*
6	5-Methyl-2-furfural	8	*
7	Benzeneacetaldehyde	2	-
8	5-Methyl-2-furanmethanol	3	-
9	Methyl phenylacetate	9	**
10	Guaiacol	4	-
11	Phenethyl alcohol	1	-
12	Pyrrole-2-carboxaldehyde	8	*
13	Ethyl cinnamate	8	*
14	Eugenol	4	-
Jujube extract			
1	Octanal	8	*
2	Benzeneacetaldehyde	9	**
3	γ-Decalactone	8	*
4	γ-Nonanolactone	8	*
5	Ketoisophorone	8	*
6	5-Methyl-2-furanmethanol	10	***
Grape extract			
1	4-Methyl-2-pentyl-1,3-dioxolane	8	*
2	Isovaleraldehyde propylene glycol acetal	8	*
3	Ethyl caproate	2	-
4	Octanal	2	-
5	1-Octen-3-ol	9	**
6	Estragole	9	**
7	Geranylacetone	8	*
8	Ethyl cinnamate	8	*

[a]: aroma-active compounds with OAV greater than 1; [b]: number of correct judgements from 12 assessors by triangle test; [c]: significance: *, significant ($p \leq 0.05$); **, highly significant ($p \leq 0.01$); ***, very highly significant ($p \leq 0.001$); -, nonsignificant.

In the plum extract, methyl phenylacetate ($p \leq 0.01$), octanal ($p \leq 0.05$), angelica lactone ($p \leq 0.05$), linalool ($p \leq 0.05$), 5-methyl-2-furfural ($p \leq 0.05$), pyrrole-2-carboxaldehyde ($p \leq 0.05$), and ethyl cinnamate ($p \leq 0.05$) were identified as key aroma compounds. Among these, methyl phenylacetate, angelica lactone, linalool, and 5-methyl-2-furfural primarily contributed to the sweet notes of the plum extract.

In the jujube extract, 5-methyl-2-furanmethanol ($p \leq 0.001$), benzeneacetaldehyde ($p \leq 0.01$), octanal ($p \leq 0.01$), γ-decalactone ($p \leq 0.05$), γ-nonanolactone ($p \leq 0.05$), and ketoisophorone ($p \leq 0.05$) were identified as key aroma compounds. Among these, 5-methyl-2-furanmethanol, benzeneacetaldehyde, γ-nonanolactone, and ketoisophorone contributed to the sweet notes of the jujube extract. Notably, octanal was a key compound shared by both plum and jujube extracts. The octanal has a C_8 linear aldehyde structure; the carbonyl oxygen forms a hydrogen bond with the receptor, while the hydrophobic chain enters the receptor's hydrophobic pocket, activating specific olfactory receptors and producing a waxy sensation. Its FD factor is 3 in both plum and jujube extracts, with OAVs of 31 and 155, respectively. Although it does not dominate the aroma, it is an indispensable aromatic component in both. Meanwhile, octanal is a food fragrance used in the preparation of fruit flavors.

In the grape extract, 1-octen-3-ol ($p \leq 0.01$), estragole ($p \leq 0.01$), 4-methyl-2-pentyl-1,3-dioxolane ($p \leq 0.05$), isovaleraldehyde propylene glycol acetal ($p \leq 0.05$), geranylacetone ($p \leq 0.05$), and ethyl cinnamate ($p \leq 0.05$) were identified as key aroma-active compounds. Among these, estragole, 4-methyl-2-pentyl-1,3-dioxolane, isovaleraldehyde propylene glycol acetal, and ethyl cinnamate contributed primarily to the sweet notes. Geranylacetone was responsible for the sweet–fruity character of the grape extract. Ethyl cinnamate was also identified as a key aroma compound common to both the plum and grape extracts. In plant metabolism, phenylalanine serves as the primary volatile precursor for ethyl cinnamate [32]. The molecule of ethyl cinnamate consists of a benzene ring, a double bond, and an ester group; the absence of a p-methoxy group is key to its intense fruity and sweet aroma. The conjugated structure of ethyl cinnamate confers moderate lipophilicity and volatility, enabling it to easily penetrate the olfactory epithelium; once it reaches the receptors, π-π stacking, hydrogen bonding, and hydrophobic interactions work in concert to produce a strong and persistent sweet aroma even at extremely low concentrations. Its FD factor is 729 in both plum and grape extracts, with OAVs of 38 and 502, respectively, making it a key contributor to the sweet aroma. It is worth noting that ethyl cinnamate is classified as a nature-identical flavor ingredient. By adding ethyl cinnamate as a sweet aroma enhancer, it is possible to develop low-sugar foods and eco-friendly products free of artificial flavors and sweeteners. Additionally, it can be incorporated into cosmetics and personal care products to provide a fragrant experience. When added to tobacco, ethyl cinnamate helps neutralize the bitterness and pungency of the tobacco.

To verify the accuracy of the quantitative results, we selected two key aroma-active compounds with medium to high concentrations from each extract and conducted spiking recovery experiments. The recovery rates for the high-concentration compound phenethyl aldehyde and the medium-concentration compound thearubigone in the jujube extract both reached 89%, the recovery rates for the high-concentration compound furfural and the medium-concentration compound thearubigone in the plum extract reached 104% and 115%, respectively, and the recovery rates for isovaleraldehyde propylene glycol acetal and hexanal propylene glycol acetal in the grape extract were 105% and 119%, respectively, indicating that this method is accurate and reliable. The recovery rates of the selected representative compounds all fell within the acceptable range of 80–120%, indicating that this method is accurate and reliable.

Based on the identified key aroma compounds, these can also be used to develop natural fruit flavor formulations for plums, grapes, and jujubes. The process involves selecting aroma compounds with a high OAV as the primary components and using those with a low OAV to supplement and adjust the aroma profile. The ratios are evaluated and fine-tuned using scent strips to ultimately formulate the fruit flavors. Once the base formulation for the fruit flavor has been established, specialized secondary formulations must be developed for different product forms (food and beverages, cosmetics, perfumes, and tobacco products) and application scenarios.

In the food industry, high-OAV compounds can be used to replace natural extracts, enabling low-cost, standardized production. At the same time, by adjusting the ratios in flavor formulations, it is possible to develop fruit-flavored products with a variety of distinct flavors. In the cosmetics industry, these compounds are used in perfumes, creams, lipsticks, and other products to add depth to the fragrance; In the tobacco industry, fruit flavorings are primarily used to release sweet, fruity aromas that neutralize the harshness and bitterness of tobacco, mask irritation, modify the aftertaste, and enhance smoking comfort.

4. Conclusions

In this study, GC×GC-O-TOF-MS combined with a molecular sensory science approach was employed to separate and identify volatile aroma compounds in three natural fruit flavor extracts from plum, jujube, and grape. A total of 188 volatile compounds were identified, with 113, 103, and 85 compounds in plum, jujube, and grape extracts, respectively. Aroma extract dilution analysis revealed 14, 11, and 32 aroma-active compounds with flavor dilution factors ≥ 3, respectively. Quantification using internal standard calibration curves and calculation of odor activity values determined the relative contribution of each compound to the overall aroma. Octanal was identified as a key aroma-active compound shared by plum and jujube extracts, while ethyl cinnamate was common to both plum and grape extracts. Key aromatic compounds in plum extract, including angelica lactone, linalool, 5-methyl-2-furamethanol, methyl phenylacetate and ethyl cinnamate, contribute to its sweet flavor characteristics. In the jujube extract, 5-methyl-2-furfural, benzeneacetaldehyde, γ-nonanolactone, and ketoisophorone were the primary contributors to sweetness. In the grape extract, estragole, 4-methyl-2-pentyl-1,3-dioxolane, isovaleraldehyde propylene glycol acetal, and ethyl cinnamate were the main contributors to sweetness, while geranylacetone predominantly imparted fruity notes. A comprehensive two-dimensional gas chromatography–olfactometry–time-of-flight mass spectrometry (GC×GC–O–TOF–MS) method was established for the separation and identification of key aroma-active compounds in three complex natural fruit flavor extracts from plum, jujube, and grape. Through a molecular sensory science approach, this study deepens the molecular-level understanding of the aroma composition of these extracts, providing a theoretical basis for precise blending and flavor design of natural fragrances. The identified key aroma compounds can directly enhance the aroma quality of end products, facilitating the development of high-end fragrances, cosmetics, functional foods, and flavored beverages. However, flavor adjustments will still need to be made based on the product lineup. Furthermore, the specific processing methods for the three fruit-flavored natural flavor extracts are unknown, making it impossible to establish a link between the processing methods and the aromas, which presents certain limitations. Notwithstanding these limitations, the key aroma compounds identified here warrant further investigation into their biosynthetic pathways and regulatory mechanisms. Future research should focus on the biosynthetic pathways and regulatory mechanisms of these key aroma substances, promoting a systematic transition from structural elucidation to flavor innovation.

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Data Availability Statement: The original contributions presented in this study are included in the article. Further inquiries can be directed to the corresponding author.

Conflicts of Interest: The authors declare no conflicts of interest.

Appendix A

Table A1. The volatile odor compounds in extracts of three fruit flavors: plum, jujube, and grape.

	Volatile Compounds	CAS	RI	Odor Description	Identification	Relative Concentration		
						Plum Extract	Jujube Extract	Grape Extract
Esters (45)								
1	Methyl acetate	79-20-9	-	sweet, fruity	MS/RI	4.948 ± 1.941	0.983 ± 0.5	-
2	Ethyl acetate	141-78-6	844	fruity, sweet	MS/RI	-	-	28.042 ± 24.599
3	Methyl propionate	554-12-1	865	fruity	MS/RI	-	0.013 ± 0.004	-
4	Methyl butyrate	623-42-7	951	fruity, sweet	MS/RI	-	0.041 ± 0.014	-
5	Butyl acetate	123-86-4	1038	fruity	MS/RI	-	0.006 ± 0.003	-
6	Butyl butyrate	109-21-7	1190	fruity	MS/RI	-	-	0.376 ± 0.48
7	Ethyl caproate	123-66-0	1205	sweet, fruity	MS/RI/O	0.227 ± 0.086	-	13.431 ± 4.537
8	Ethyl pyruvate	617-35-6	1229	fruity, sweet	MS/RI	-	-	0.536 ± 0.272
9	Hexyl acetate	142-92-7	1242	fruity, green	MS/RI	-	0.007 ± 0.005	0.399 ± 0.572
10	Isoamyl 2-methylbutyrate	27625-35-0	1268	sweet, fruity	MS/RI	0.003 ± 0.001	-	-
11	Ethyl heptanoate	106-30-9	1302	fruity	MS/RI	-	0.057 ± 0.049	1.346 ± 0.274
12	Methyl octanoate	111-11-5	1358	waxy, sweet	MS/RI	0.047 ± 0.039	0.23 ± 0.023	-
13	Butyl hexanoate	626-82-4	1384	fruity	MS/RI	-	0.001 ± 0	-
14	Angelica lactone	591-12-8	1390	sweet	MS/RI/O	0.119 ± 0.003	0.02 ± 0.008	-
15	Ethyl caprylate	106-32-1	1406	fruity	MS/RI	0.017 ± 0.007	-	5.304 ± 0.678
16	Ethyl ester-2,4-dimethyl-1,3-dioxolane-2-acetic acid	6290-17-1	1444	fruity	MS/RI	-	-	0.535 ± 0.812
17	Furfuryl acetate	623-17-6	1489	sweet, fruity	MS/RI	0.107 ± 0.013	0.012 ± 0.003	-
18	Ethyl nonanoate	123-29-5	1502	fruity, rose	MS/RI/O	-	-	11.517 ± 1.179
19	3,7-Dimethyl-1,6-octadien-3-ol formate	115-99-1	1503	citrus, green	MS/RI	-	0.046 ± 0.004	-
20	Bornyl acetate	76-49-3	1549	woody	MS/RI	-	-	0.145 ± 0.043
21	Methyl ester decanoic acid	110-42-9	1562	fruity, floral	MS/RI	-	0.107 ± 0.043	-
22	Methyl benzoate	93-58-3	1579	floral	MS/RI	0.246 ± 0.044	-	-
23	γ-Butyrolactone	96-48-0	1582	caramel	MS/RI	0.674 ± 0.137	-	-
24	Hexyl hexanoate	6378-65-0	1582	fruity, herbal	MS/RI/O	-	0.005 ± 0.002	0.282 ± 0.322
25	Ethyl caprate	110-38-3	1608	sweet, waxy	MS/RI	-	-	1.386 ± 0.069
26	Ethyl benzoate	93-89-0	1626	fruity	MS/RI/O	-	-	1.313 ± 0.197
27	Benzyl acetate	140-11-4	1682	sweet, floral	MS/RI	0.54 ± 0.027	0.048 ± 0.014	-
28	Ethyl ester undecanoic acid	627-90-7	1709	waxy	MS/RI	-	-	0.815 ± 0.106
29	Methyl phenylacetate	101-41-7	1711	sweet, floral	MS/RI/O	0.82 ± 0.047	-	-
30	Ethyl 3-hydroxyhexanoate	2305-25-1	1722	fruity	MS/RI	-	-	0.393 ± 0.337
31	Methyl salicylate	119-36-8	1728	mint	MS/RI	1.433 ± 0.017	-	5.45 ± 4.959
32	Ethyl phenylacetate	101-97-3	1739	sweet, floral	MS/RI	-	-	0.749 ± 0.193

Table A1. *Cont.*

	Volatile Compounds	CAS	RI	Odor Description	Identification	Relative Concentration		
						Plum Extract	Jujube Extract	Grape Extract
33	Diisopropyl adipate	6938-94-9	1829	sour	MS/RI	-	-	2.997 ± 4.92
34	5-Butyldihydro-2(3H)-furanone	104-50-7	1867	sweet, coconut	MS/RI	0.106 ± 0.012	-	-
35	γ-Nonanolactone	104-61-0	1975	sweet, waxy	MS/RI/O	-	0.856 ± 0.244	-
36	Isopropyl myristate	110-27-0	2003	fatty	MS/RI	-	0.035 ± 0.053	0.762 ± 1.228
37	δ-Nonalactone	3301-94-8	2028	sweet	MS/RI	0.05 ± 0.008	-	-
38	Ethyl cinnamate	103-36-6	2077	sweet, fruity	MS/RI/O	5.439 ± 0.887	-	3.521 ± 0.528
39	γ-Decalactone	706-14-9	2092	waxy	MS/RI/O	-	0.212 ± 0.029	0.371 ± 0.209
40	Ethylpentadecanoate	41114-00-5	2118	sweet	MS/RI/O	-	-	0.218 ± 0.175
41	Ethyl palmitate	628-97-7	2216	fruity, waxy	MS/RI/O	0.07 ± 0.032	-	34.961 ± 7.252
42	5,6,7,7a-Tetrahydro-4,4,7a-trimethyl-2(4H)-benzofuranone	15356-74-8	2286	fruit, woody	MS/RI	0.44 ± 0.045	0.133 ± 0.055	-
43	Triethyl citrate	77-93-0	2395	fruity, wine	MS/RI	-	0.049 ± 0.023	-
44	Benzyl Benzoate	120-51-4	-	sweet	MS/RI	11.748 ± 0.732	0.038 ± 0.03	-
45	Ethyl formate	109-94-4	-	green, rose	MS/RI	-	-	5.272 ± 1.173
Aldehydes (33)								
1	Acetaldehyde	75-07-0	-	fruity	MS/RI	0.125 ± 0.051	0.04 ± 0.021	0.752 ± 0.729
2	Glutaraldehyde	111-30-8	830	fruity	MS/RI	0.626 ± 0.051	0.012 ± 0.007	-
3	Butyraldehyde	123-72-8	831	cocoa, musty	MS/RI	0.023 ± 0.001	-	2.065 ± 2.889
4	3-Methylbutyraldehyde	590-86-3	879	chocolate	MS/RI	2.005 ± 0.481	0.991 ± 0.553	6.424 ± 0.844
5	4-Methyl-2-pentyl-1,3-dioxolane	1599-49-1	1041	fruity, waxy	MS/RI/O	5.181 ± 0.97	-	2.063 ± 3.443
6	Hexanal	66-25-1	1047	fruity	MS/RI/O	0.048 ± 0.045	0.267 ± 0.116	8.017 ± 2.093
7	(*E*)-2-Methyl-2-butenal	497-03-0	1059	green, fruit	MS/RI	0.048 ± 0.004	0.015 ± 0.004	-
8	Isovaleraldehyde propylene glycol acetal	18433-93-7	1140	sweet, fruity	MS/RI/O	0.388 ± 0.062	-	3.904 ± 0.418
9	Heptanal	111-71-7	1151	green, herbal	MS/RI	-	0.105 ± 0.003	-
10	3-Methyl-2-butenal	107-86-8	1162	sweet, fruity	MS/RI	25.597 ± 2.891	0.002 ± 0.001	-
11	Octanal	124-13-0	1256	waxy	MS/RI/O	2.005 ± 0.481	0.199 ± 0.096	3.403 ± 0.986
12	Nonanal	124-19-6	1362	waxy, rose	MS/RI	0.808 ± 0.146	0.724 ± 0.492	-
13	(*E*)-2-Octenal	2548-87-0	1395	green, waxy	MS/RI/O	4.681 ± 0.088	-	2.763 ± 0.335
14	2-Heptyl-3-methyl-1,3-Dioxolane	74094-61-4	1397	green, woody	MS/RI	2.298 ± 0.513	-	1.973 ± 3.287
15	Furfural	98-01-1	1421	sweet, woody	MS/RI/O	0.048 ± 0.004	0.283 ± 0.011	-
16	Decanal	112-31-2	1464	sweet, waxy	MS/RI/O	2.357 ± 0.426	0.194 ± 0.029	2.984 ± 2.029
17	Undecanal	112-44-7	1472	waxy	MS/RI/O	-	0.034 ± 0.011	0.164 ± 0.071
18	Benzaldehyde	100-52-7	1478	sweet	MS/RI/O	-	3.158 ± 0.198	18.584 ± 31.923
19	5-Methyl-2-furaldehyde	620-02-0	1529	sweet, caramel	MS/RI/O	111.099 ± 65.359	7.84 ± 13.565	-
20	Pyrrole-2-carboxaldehyde	1192-58-1	1576	roasted, nutty	MS/RI/O	-	0.85 ± 0.31	-

Table A1. *Cont.*

	Volatile Compounds	CAS	RI	Odor Description	Identification	Relative Concentration		
						Plum Extract	Jujube Extract	Grape Extract
21	α,4-Dimethyl-3-cyclohexene-1-acetaldehyde	29548-14-9	1579	herbal	MS/RI	0.808 ± 0.146	-	-
22	2,6,6-Trimethyl-1-cyclohexene-1-carboxaldehyde	432-25-7	1586	herbal	MS/RI	-	0.011 ± 0.001	0.471 ± 0.218
23	Benzeneacetaldehyde	122-78-1	1596	sweet, floral	MS/RI/O	-	0.132 ± 0.049	-
24	2,3-Dihydro-2,2,6-trimethylben	116-26-7	1608	herbal	MS/RI	-	0.157 ± 0.026	-
25	Dodecanal	112-54-9	1676	waxy	MS/RI/O	-	-	0.459 ± 0.333
26	2,4-Dimethyl-benzaldehyde	15764-16-6	1765	vanilla	MS/RI	-	-	-
27	4-Methyl-2-phenyl-1,3-dioxolane	2568-25-4	1798	sweet	MS/RI	-	-	21.32 ± 30.925
28	2-Methyl-3(2-furyl)acrolein	874-66-8	1824	woody	MS/RI	-	0.086 ± 0.022	-
29	Pyrrole-2-carboxaldehyde	1003-29-8	1964	musty	MS/RI/O	-	0.543 ± 0.037	-
30	2-(Phenylmethylene)-octanal	101-86-0	2301	floral, green	MS/RI	2.357 ± 0.426	-	-
31	5-Hydroxymethylfurfural	67-47-0	-	fatty, waxy	MS/RI	40.559 ± 3.088	1.43 ± 0.348	7.117 ± 11.176
32	Propanal	123-38-6	-	earthy	MS/RI	-	0.014 ± 0.004	-
33	2-Phenyl-3-(2-furyl)-propenal	57568-60-2	-	spice	MS/RI	-	0.011 ± 0.002	-
Alcohols (29)								
1	2-Methyl-2-propanol	75-65-0	861	camphor	MS/RI/O	7.902 ± 11.067	0.233 ± 0.185	2.082 ± 0.408
2	Ethanol	64-17-5	899	alcoholic	MS/RI/O	0.39 ± 0.011	0.092 ± 0.058	0.882 ± 1.362
3	1-Propanol	71-23-8	1007	alcoholic, fermented	MS/RI	0.414 ± 0.161	-	-
4	2-Methyl-3-buten-2-ol	115-18-4	1008	herbal, earthy	MS/RI	0.005 ± 0.004	-	0.089 ± 0.077
5	2-Methyl-1-propanol	78-83-1	1057	ethereal, winey	MS/RI/O	0.088 ± 0.035	0.035 ± 0.007	-
6	2-Propen-1-ol	107-18-6	1075	mustard	MS/RI	0.092 ± 0.01	-	-
7	1-Butanol	71-36-3	1110	sweet	MS/RI	0.148 ± 0.019	0.022 ± 0.008	1.341 ± 1.66
8	3-Methyl-1-butanol	123-51-3	1173	fruity	MS/RI	0.139 ± 0.021	-	-
9	1-Pentanol	71-41-0	1215	sweet	MS/RI	1.037 ± 0.025	0.347 ± 0.117	-
10	Prenol	556-82-1	1283	fruity	MS/RI	0.007 ± 0.001	0.004 ± 0.002	-
11	1-Hexanol	111-27-3	1316	fruity	MS/RI	6.661 ± 0.326	-	-
12	5-Hexen-1-ol	821-41-0	1372	green	MS/RI	0.174 ± 0.003	-	1.101 ± 0.41
13	1-Hexanol	5989-33-3	1411	floral, sweet	MS/RI	0.584 ± 0.09	0.388 ± 0.136	22.81 ± 26.51
14	1-Octen-3-ol	3391-86-4	1413	earthy, mushroom	MS/RI	1.465 ± 0.016	-	1.037 ± 0.171
15	2-Ethylhexanol	104-76-7	1450	citrus, floral	MS/RI	4.55 ± 0.604	1.275 ± 0.365	-
16	Linalool	78-70-6	1503	floral, sweet	MS/RI/O	0.588 ± 0.054	0.062 ± 0.044	2.834 ± 0.746
17	1-Octanol	111-87-5	1516	waxy, green	MS/RI	2.946 ± 0.224	-	1.574 ± 0.983
18	Isophorone	78-59-1	1556	woody, sweet	MS/RI	0.395 ± 0.088	0.181 ± 0.058	-
19	2-Furanmethanol	98-00-0	1612	sweet, caramel	MS/RI	0.065 ± 0.017	-	-
20	1-Nonanol	143-08-8	1620	floral	MS/RI/O	0.021 ± 0.004	-	-

Table A1. *Cont.*

	Volatile Compounds	CAS	RI	Odor Description	Identification	Relative Concentration		
						Plum Extract	Jujube Extract	Grape Extract
21	α,α,4-Trimethyl-(R)-3-cyclohexene-1-methanol	7785-53-7	1657	floral	MS/RI	0.177 ± 0.078	-	-
22	Borneol	507-70-0	1661	woody, camphor	MS/RI	0.138 ± 0.131	-	5.631 ± 1.736
23	5-Methyl-2-furanmethanol	3857-25-8	1672	sweet, caramellic	MS/RI/O	10.05 ± 1.718	0.767 ± 0.645	4.13 ± 5.795
24	(3R,6S)-2,2,6-Trimethyl-6-vinyltetrahydro-2H-pyran-3-ol	39028-58-5	1717	woody	MS/RI	1.227 ± 0.047	0.516 ± 0.111	-
25	1-Decanol	112-30-1	1719	waxy, floral	MS/RI	-	-	0.302 ± 0.093
26	2,5-Dimethyl-2,5-hexanediol	110-03-2	1720	camphor	MS/RI	-	-	1.241 ± 0.292
27	1,1′-Oxybis-2-propanol	110-98-5	1782	alcoholic	MS/RI	-	-	4.724 ± 1.849
28	Benzyl alcohol	100-51-6	1824	floral, rose	MS/RI/O	-	0.206 ± 0.054	0.346 ± 0.14
29	Phenylethyl alcohol	60-12-8	1861	floral, rose	MS/RI/O	-	0.021 ± 0.006	-
Ketones (29)								
1	2-Butanone	78-93-3	858	fruity	MS/RI	-	0.074 ± 0.013	3.347 ± 2.173
2	2,3-Butanedione	431-03-8	938	sweet	MS/RI/O	-	0.619 ± 0.209	1.274 ± 0.581
3	2,3-Pentanedione	600-14-6	1022	sweet	MS/RI	0.739 ± 0.111	0.215 ± 0.042	-
4	2-Heptanone	110-43-0	1149	fruity, sweet	MS/RI	-	0.017 ± 0.006	-
5	6-Methyl-2-heptanone	928-68-7	1207	camphoreous	MS/RI	0.006 ± 0.004	-	0.138 ± 0.134
6	5-Methyl-3-heptanone	541-85-5	1224	herbal, sweet	MS/RI	-	-	0.316 ± 0.123
7	Dihydro-2-methyl-3(2H)-furanone	3188-00-9	1226	sweet	MS/RI	1.328 ± 0.538	0.049 ± 0.014	0.667 ± 0.845
8	Acetoin	513-86-0	1245	sweet	MS/RI	0.029 ± 0.009	0.042 ± 0.007	0.98 ± 0.192
9	2-Octanone	111-13-7	1253	earthy, weedy	MS/RI	0.024 ± 0.006	0.02 ± 0.002	-
10	1-Hydroxy-2-propanone	116-09-6	1258	sweet	MS/RI	0.177 ± 0.07	0.107 ± 0.045	-
11	6-Methyl-5-hepten-2-one	110-93-0	1301	citrus, green	MS/RI	0.132 ± 0.02	0.043 ± 0.03	13.882 ± 2.407
12	2-Nonanone	821-55-6	1357	sweet, green	MS/RI	0.013 ± 0.007	-	0.372 ± 0.305
13	2-Decanone	693-54-9	1459	floral	MS/RI/O	-	-	1.043 ± 0.191
14	Cis-5-methyl-2-(1-methylethyl)-cyclohexanone	491-07-6	1459	sweet	MS/RI	-	-	0.284 ± 0.139
15	6-Methyl-3,5-heptadiene-2-one	1604-28-0	1550	woody, sweet	MS/RI	0.183 ± 0.01	-	-
16	2-Undecanone	112-12-9	1565	waxy, fruity	MS/RI	-	-	0.163 ± 0.014
17	Acetophenone	98-86-2	1608	sweet	MS/RI	-	0.135 ± 0.015	13.288 ± 8.505
18	2-Hydroxy-3,5,5-trimethyl-cyclohex-2-enone	4883-60-7	1622	woody	MS/RI	0.012 ± 0.003	-	-
19	Ketoisophorone	1125-21-9	1648	woody, sweet	MS/RI/O	-	0.044 ± 0.001	-
20	6,10-Dimethyl-2-undecanone	1604-34-8	1654	musty	MS/RI	-	-	0.652 ± 0.198
21	2,6,6-Trimethyl-2,4-cycloheptadien-1-one	503-93-5	1689	minty	MS/RI	0.06 ± 0.014	-	-
22	2(5H)-Furanone	497-23-4	1699	buttery	MS/RI	0.157 ± 0.071	0.123 ± 0.043	-
23	α-Ionone	127-41-3	1810	sweet, woody	MS/RI	0.054 ± 0.005	-	-
24	Geranylacetone	3796-70-1	1814	fruity, waxy	MS/RI/O	-	-	4.416 ± 7.54

Table A1. *Cont.*

	Volatile Compounds	CAS	RI	Odor Description	Identification	Relative Concentration		
						Plum Extract	Jujube Extract	Grape Extract
25	4-(2-Furanyl)-3-buten-2-one	623-15-4	1853	sweet, spicy	MS/RI	0.165 ± 0.059	-	-
26	trans-β-Ionone	79-77-6	1898	floral, woody	MS/RI	-	0.002 ± 0.001	-
27	6,10,14-Trimethyl-2-pentadecanone	502-69-2	2090	herbal	MS/RI	-	-	0.873 ± 0.986
28	5,6-Dihydro-6-pentyl-2H-pyran-2-one	54814-64-1	2176	sweet	MS/RI	-	0.023 ± 0.02	-
29	Acetone	67-64-1	-	fruity	MS/RI/O	-	0.89 ± 0.526	5.861 ± 1.452
Heterocyclic (22)								
1	Furan	110-00-9	-	ethereal	MS/RI	0.189 ± 0.192	-	-
2	2,5-Dimethylfuran	625-86-5	919	ethereal	MS/RI	-	0.021 ± 0.008	-
3	2-Ethyl-5-methyl-Furan	1703-52-2	1003	fresh	MS/RI	-	0.003 ± 0.003	-
4	2-Vinylfuran	1487-18-9	1037	coffee	MS/RI	-	0.014 ± 0.007	-
5	2-Pentyl-furan	3777-69-3	1202	fruity green	MS/RI	0.035 ± 0.004	0.015 ± 0.003	3.847 ± 2.85
6	Thiazole	288-47-1	1212	nutty	MS/RI	-	0.004 ± 0.002	-
7	Methyl-pyrazine	109-08-0	1230	nutty	MS/RI	0.04 ± 0.004	0.006 ± 0.002	-
8	2,6-Dimethyl-pyrazine	108-50-9	1293	cocoa	MS/RI	2.055 ± 0.261	0.197 ± 0.043	-
9	Ethyl-pyrazine	13925-00-3	1297	nutty	MS/RI	-	0.005 ± 0.003	-
10	2,3-Dimethyl-pyrazine	5910-89-4	1310	nutty	MS/RI	0.005 ± 0	0.004 ± 0.001	-
11	2-Ethyl-6-methyl-pyrazine	13925-03-6	1351	roasted potato	MS/RI	0.134 ± 0.004	-	-
12	Trimethyl-pyrazine	14667-55-1	1372	nutty	MS/RI	0.057 ± 0.016	0.128 ± 0.035	-
13	2,3-Dimethyl-5-ethylpyrazine	15707-34-3	1427	popcorn	MS/RI	-	0.043 ± 0.013	-
14	Tetramethyl-pyrazine	1124-11-4	1441	nutty	MS/RI	-	0.005 ± 0.001	-
15	2-Acetylfuran	1192-62-7	1459	sweet	MS/RI/O	17.64 ± 24.385	7.135 ± 0.897	11.774 ± 10.526
16	1-(2-Furanyl)-1-propanone	3194-15-8	1529	fruity	MS/RI	-	3.699 ± 0.294	-
17	2-Methyl-benzofuran	4265-25-2	1553	burnt	MS/RI	0.057 ± 0.006	-	-
18	Pyrrole-2-carboxaldehyde	2167-14-8	1566	burnt	MS/RI	4.438 ± 0.045	13.875 ± 1.34	-
19	2-Acetyl-5-methylfuran	1193-79-9	1570	nutty	MS/RI	2.795 ± 0.23	0.102 ± 0.169	-
20	Methyl 6-methyl-2-pyridyl ketone	6940-57-4	1588	potato	MS/RI	0.792 ± 0.009	0.003 ± 0.001	-
21	1-(5-Methyl-2-furanyl)-1-propanone	10599-69-6	1639	green	MS/RI	0.339 ± 0.039	-	-
22	1-(1H-pyrrol-2-yl)-ethanone	1072-83-9	1915	fruity	MS/RI/O	21.679 ± 2.327	0.479 ± 0.718	-
Acids (7)								
1	Acetic acid	64-19-7	1407	sour, vinegar	MS/RI/O	21.791 ± 0.003	8.444 ± 14.536	22.518 ± 25.017
2	2-Methyl-propanoic acid	79-31-2	1534	sour, cheese	MS/RI	0.651 ± 0.114	-	-
3	Hexanoic acid	142-62-1	1810	sour	MS/RI	3.339 ± 0.294	-	15.817 ± 7.939
4	Heptanoic acid	111-14-8	1918	sour	MS/RI/O	0.841 ± 0.561	-	1.518 ± 1.537
5	Octanoic acid	124-07-2	2013	waxy	MS/RI/O	4.469 ± 1.331	-	7.476 ± 4.187
6	Nonanoic acid	112-05-0	2114	waxy	MS/RI/O	-	6.706 ± 5.638	34.427 ± 8.719

Table A1. *Cont.*

	Volatile Compounds	CAS	RI	Odor Description	Identification	Relative Concentration		
						Plum Extract	Jujube Extract	Grape Extract
7	Benzoic acid	65-85-0	2392	balsam	MS/RI	-	-	12.381 ± 11.056
Phenols (6)								
1	2-Methoxy-phenol	90-05-1	1807	woody	MS/RI/O	0.125 ± 0.051	-	-
2	Butylated Hydroxytoluene	128-37-0	1869	camphor	MS/RI	0.626 ± 0.051	-	1.101 ± 0.567
3	Eugenol	97-53-0	2107	sweet, woody	MS/RI/O	0.023 ± 0.001	-	-
4	2-Methoxy-4-vinylphenol	7786-61-0	2133	sweet woody	MS/RI	2.005 ± 0.481	0.139 ± 0.053	-
5	2,4-Di-tert-butylphenol	96-76-4	2244	phenolic	MS/RI	5.181 ± 0.97	6.885 ± 0.785	-
6	(*Z*)-2-Methoxy-4-(1-propenyl)-phenol	5912-86-7	2278	clove	MS/RI	0.048 ± 0.045	-	-
Olefins (5)								
1	(-)-Limonene	5989-54-8	1174	herbal	MS/RI	0.152 ± 0.005	-	-
2	(4R)-1-Methyl-4-(1-methylethenyl)-cyclohexene	5989-27-5	1174	citrus	MS/RI	-	0.274 ± 0.218	52.802 ± 14.743
3	Styrene	100-42-5	1222	sweet	MS/RI	0.116 ± 0.092	3.288 ± 5.633	-
4	(*Z*)-1-Methoxy-4-(1-propenyl)-benzene	25679-28-1	1781	anise	MS/RI	-	0.028 ± 0.019	-
5	(-)-Calamenene	483-77-2	1801	herbal	MS/RI	-	-	0.506 ± 0.352
Others (11)								
1	Dimethyl disulfide	624-92-0	1037	onion	MS/RI	0.626 ± 0.051	0.004 ± 0.002	-
2	2-Butyl-4-methyl-1,3-dioxolane	74094-60-3	1136	nutty	MS/RI	0.023 ± 0.001	0.112 ± 0.184	-
3	Trimethyl-oxazol	20662-84-4	1165	nutty	MS/RI	2.005 ± 0.481	0.005 ± 0.002	-
4	2-(Methoxymethyl)-furan	13679-46-4	1202	coffee	MS/RI	5.181 ± 0.97	-	-
5	p-Cymene	99-87-6	1238	woody	MS/RI	0.048 ± 0.045	-	0.781 ± 0.27
6	Dimethyl trisulfide	3658-80-8	1344	onion	MS/RI	0.048 ± 0.004	0.003 ± 0.001	-
7	trans-Linalool oxide (furanoid)	34995-77-2	1436	floral	MS/RI	0.388 ± 0.062	-	-
8	(-)-Camphor	464-48-2	1479	camphor	MS/RI	25.597 ± 2.891	0.007 ± 0.006	-
9	Dill ether	74410-10-9	1482	dill	MS/RI	111.099 ± 65.359	-	-
10	Estragole	140-67-0	1783	sweet	MS/RI/O	1.324 ± 0.092	-	2.014 ± 3.141
11	Heptane	142-82-5	-	sweet	MS/RI	0.808 ± 0.146	-	1.224 ± 2.058

-: not detected; MS: mass spectral data; RI: retention index, the actual RI value calculated from the peak time of the series of alkanes on the DB-WAX column; O: sniff.

Table A2. The aroma-active compounds in extracts of three fruit flavors: plum, jujube, and grape.

	Volatile Compounds	CAS	Odor Description	FD Factor		
				Plum Extract	Jujube Extract	Grape Extract
Aldehydes (15)						
1	4-Methyl-2-pentyl-1,3-dioxolane	1599-49-1	fruity, waxy	-	-	3
2	Hexanal	66-25-1	fruity	-	-	1
3	Isovaleraldehyde propylene glycol acetal	18433-93-7	sweet, fruity	-	-	729
4	Octanal	124-13-0	waxy	3	3	3
5	(*E*)-2-Octenal	2548-87-0	green, waxy	-	-	1
6	Furfural	98-01-1	sweet, woody	3	1	-
7	Decanal	112-31-2	sweet, waxy	-	-	1
8	Undecanal	112-44-7	waxy	-	-	1
9	Benzaldehyde	100-52-7	sweet	3	-	-
10	5-Methyl-2-furaldehyde	620-02-0	caramel	3	-	-
11	Pyrrole-2-carboxaldehyde	1192-58-1	roasted, nutty	-	1	-
12	Benzeneacetaldehyde	122-78-1	sweet, floral	9	9	-
13	2,3-Dihydro-2,2,6-trimethylben	116-26-7	herbal	-	-	-
14	Dodecanal	112-54-9	waxy	-	-	1
15	Pyrrole-2-carboxaldehyde	1003-29-8	musty	27	-	-
Alcohols (11)						
1	2-Methyl-2-propanol	75-65-0	camphor	-	-	1
2	Ethanol	64-17-5	alcoholic	-	-	1
3	2-Methyl-3-buten-2-ol	115-18-4	herbal, earthy	-	-	-
4	2-Methyl-1-propanol	78-83-1	ethereal, winey	-	-	1
5	1-Hexanol	111-27-3	fruity	-	1	-
6	1-Octen-3-ol	3391-86-4	earthy, mushroom	-	-	9
7	Linalool	78-70-6	floral, sweet	9	-	-
8	1-Nonanol	143-08-8	floral	-	-	1
9	5-Methyl-2-furanmethanol	3857-25-8	sweet, caramellic	9	3	-
10	Benzyl alcohol	100-51-6	floral, rose	-	-	1
11	Phenylethyl alcohol	60-12-8	floral, rose	9	1	-
Esters (11)						
1	Ethyl caproate	123-66-0	sweet, fruity	-	-	3
2	Angelica lactone	591-12-8	sweet	3	-	-
3	Ethyl nonanoate	123-29-5	fruity, rose	-	-	1
4	Hexyl hexanoate	6378-65-0	fruity, herbal	-	-	1

Table A2. *Cont.*

	Volatile Compounds	CAS	Odor Description	FD Factor		
				Plum Extract	Jujube Extract	Grape Extract
5	Ethyl benzoate	93-89-0	fruity	-	-	1
6	Methyl phenylacetate	101-41-7	sweet, floral	3	-	-
7	Ethyl cinnamate	103-36-6	sweet, fruity	729	-	729
8	γ-Decalactone	706-14-9	waxy	-	27	-
9	Ethylpentadecanoate	41114-00-5	sweet	-	-	1
10	Ethyl palmitate	628-97-7	waxy, fruity	-	-	1
11	γ-Nonanolactone	104-61-0	coconut, waxy	-	9	-
Ketones (5)						
1	2,3-Butanedione	431-03-8	sweet	-	-	1
2	2-Decanone	693-54-9	floral	-	-	1
3	Ketoisophorone	1125-21-9	woody, sweet	-	3	-
4	Geranylacetone	3796-70-1	fruity, waxy	-	-	3
5	Acetone	67-64-1	fruity	-	-	1
Acids (4)						
1	Acetic acid	64-19-7	sour, vinegar	-	-	1
2	Heptanoic acid	111-14-8	sour	-	-	1
3	Octanoic acid	124-07-2	waxy	-	-	1
4	Nonanoic acid	112-05-0	waxy	-	-	1
Phenols (2)						
1	2-Methoxy-phenol	90-05-1	woody	243	-	-
2	Eugenol	97-53-0	sweet, woody	243	-	-
Heterocyclic (2)						
1	2-Acetylfuran	1192-62-7	sweet	-	-	1
2	1-(1H-pyrrol-2-yl)-ethanone	1072-83-9	fruity	-	1	243
Others (1)						
1	Estragole	140-67-0	sweet	-	-	729

-: not detected.

Table A3. Concentrations, odor thresholds, and odor activity values of aroma-active compounds in extracts of three fruit flavors: plum, grape, and jujube.

	Aroma-Active Compounds	Linear Equations	R^3	Linear Range (μg/L)	Concentration (μg/g)			LOD	LOQ	RSD (%)	Odor Threshold (ug/g)	OAV		
					Plum Extract	Jujube Extract	Grape Extract					Plum Extract	Jujube Extract	Grape Extract
Alcohols														
1	1-Octen-3-ol	y = 0.3245x − 0.000005	0.9969	130.781~8370.000	-	-	1882.864 ± 238.500	0.00066	0.0020	12.7	1.5	-	-	1255
2	Linalool	y = 0.0868x + 0.000006	0.9989	27.125~868.000	672.494 ± 73.571	-	-	0.000048	0.00015	10.9	6	112	-	-
3	5-Methyl-2-furanmethanol *	y = 0.1619x − 0.000002	0.9984	33.653~538.450	147.397 ± 18.844	351.631 ± 30.605	-	0.000029	0.000088	12.8	0.009	16,749	39,958	-
4	Phenethyl alcohol	y = 0.0595x − 0.00001	0.9918	637.500~10,200.00	986.834 ± 59.631	-	-	0.001099	0.0034	6.0	564	2	-	-
Aldehydes														
1	Nonanal	y = 1.9639x + 0.0000008	0.9974	12.800~411.000	17.917 ± 1.327	90.921 ± 6.825	72.586 ± 10.384	0.0000016	0.0000050	14.3	0.587	31	155	124
2	Furfural	y = 0.2732x + 0.0141	0.9907	36,250.000~1,160,000	297,076.482 ± 24,444.384	-	-	0.14	0.42	0.8	9562	31	-	-
3	Benzaldehyde	y = 0.9449x + 0.0003	0.9979	325.000~10,400.000	1815.849 ± 218.245	-	-	0.00060	0.0018	12.0	750.89	2	-	-
4	5-Methyl-2-furfural	y = 0.4509x − 0.004	0.9979	34,593.750~553,500.000	47,857.922 ± 5856.231	-	-	0.034	0.10	12.2	1110	43	-	-
5	Benzeneacetaldehyde	y = 0.8886x − 0.00003	0.9928	160.469~10,270.000	521.623 ± 63.830	440.280 ± 46.822	-	0.0011	0.0033	12.2	6.300	83	70	-
6	Pyrrole-2-carboxaldehyde	y = 0.2465x − 0.00006	0.9913	187.031~5985.000	2468.591 ± 225.528	-	-	0.00070	0.0021	9.1	65	38	-	-
7	4-Methyl-2-pentyl-1,3-dioxolanel *	y = 0.5658x + 0.00003	0.9949	140.672~9003.000	-	-	3210.760 ± 259.337	0.00040	0.0012	8.1	0.00902	-	-	355,960
8	Isovaleraldehyde propylene glycol acetal *	y = 0.7832x + 0.00005	0.994	279.688~17,900.000	-	-	9958.683 ± 700.445	0.0027	0.0081	7.0	0.000173	-	-	57,564,641
Ketones														
1	Ketoisophorone	y = 0.3044x + 0.000006	0.9990	40.692~1302.149	-	146.435 ± 16.183	-	0.000045	0.00014	11.1	25	-	6	-
2	Geranylacetone	y = 0.4208x − 0.00003	0.9953	272.813~4365.000	-	-	2064.986 ± 169.471	0.00040	0.0012	8.2	60	-	-	34
Esters														
1	γ-Decalactone	y = 0.6568x + 0.000006	0.9942	14.813~474.000	-	65.479 ± 8.303	-	0.000065	0.00020	12.7	1.100	-	60	-
2	Angelica lactone *	y = 0.1132x − 0.00005	0.9900	17.516~1121.000	454.138 ± 36.520	-	-	0.00015	0.00047	8.0	0.0875	5190	-	-
3	Methyl phenylacetate *	y = 0.4044x + 0.000003	0.9973	10.410~666.250	221.712 ± 22.081	-	-	0.000041	0.00012	10.0	0.000441	502,747	-	-

Table A3. *Cont.*

	Aroma-Active Compounds	Linear Equations	R^3	Linear Range (μg/L)	Concentration (μg/g)			LOD	LOQ	RSD (%)	Odor Threshold (ug/g)	OAV		
					Plum Extract	Jujube Extract	Grape Extract					Plum Extract	Jujube Extract	Grape Extract
4	Ethyl cinnamate	y = 0.506x − 0.00005	0.9991	20.860~1056.000	55.293 ± 8.569	-	728.362 ± 160.313	0.000041	0.00012	15.5	1.45	38	-	502
5	Ethyl caproate	y = 0.1349x + 0.0000004	0.9947	271.563~4345.000	-	-	2225.610 ± 181.657	0.0000052	0.00005	8.2	5	-	-	445
6	γ-Nonanolactone	y = 0.4363x − 0.00000006	0.9902	30.500~488.000	-	289.821 ± 39.777	-	0.000049	0.00015	13.7	9.700	-	30	-
Phenols														
1	Eugenol	y = 0.0645x + 0.0000003	0.9952	10.420~666.875	28.572 ± 4.464	-	-	0.000033	0.00010	15.6	0.61	47	-	-
2	Guaiacol	y = 0.231x − 0.000006	0.9919	89.207~1427.307	542.220 ± 72.182	-	-	0.00014	0.00041	13.3	10	54	-	-
Others														
1	Artemisia ketone	y = 0.6217x − 0.00009	0.9919	301.563~4825.000	-	-	337.489 ± 31.578	0.00058	0.0018	9.4	16	-	-	21

-: not found; Odor thresholds of each compound were referenced from a book named: Odour Thresholds: Compilations of Odour Threshold Values in Air, Water and Other Media. The odor thresholds for isovaleraldehyde propylene glycol acetal, methyl phenylacetate, 4-methyl-2-pentyl-1,3-dioxolane, angelic lactone, and 5-methyl-2-furanmethanol were determined through threshold experiments; *: the threshold values of these compounds were determined through threshold experiments.

References

1. Sharmeen, J.B.; Mahomoodally, F.M.; Zengin, G.; Maggi, F. Essential Oils as Natural Sources of Fragrance Compounds for Cosmetics and Cosmeceuticals. *Molecules* **2021**, *26*, 666. [CrossRef] [PubMed]
2. Bailly, C. Anticancer properties of *Prunus mume* extracts (Chinese plum, Japanese apricot). *J. Ethnopharmacol.* **2020**, *246*, 112215. [CrossRef] [PubMed]
3. Bendaali, Y.; Vaquero, C.; Escott, C.; González, C.; Morata, A. Isotonic Drinks Based on Organic Grape Juice and Naturally Flavored with Herb and Spice Extracts. *Beverages* **2023**, *9*, 49. [CrossRef]
4. Serafini, M.; Peluso, I. Functional Foods for Health: The Interrelated Antioxidant and Anti-Inflammatory Role of Fruits, Vegetables, Herbs, Spices and Cocoa in Humans. *Curr. Pharm. Des.* **2016**, *22*, 6701–6715. [CrossRef] [PubMed]
5. Prebihalo, S.E.; Berrier, K.L.; Freye, C.E.; Bahaghighat, H.D.; Moore, N.R.; Pinkerton, D.K.; Synovec, R.E. Multidimensional Gas Chromatography: Advances in Instrumentation, Chemometrics, and Applications. *Anal. Chem.* **2018**, *90*, 505–532. [CrossRef] [PubMed]
6. Hu, X.F.; Li, D.Y.; Ding, Y.; Zhang, Y.B.; Ren, C.G. Characteristic of volatile flavor compounds in 'Fengtangli' plum (*Prunus salicina* Lindl.) were explored based on GCxGC-TOF MS. *Front. Nutr.* **2025**, *12*, 1536954. [CrossRef] [PubMed]
7. Liu, H.C.; Yu, Y.S.; Zou, B.; Yu, Y.Y.; Yang, J.G.; Xu, Y.J.; Chen, X.W.; Yang, F. Evaluation of Dynamic Changes and Regularity of Volatile Flavor Compounds for Different Green Plum (*Prunus mume* Sieb. et Zucc) Varieties during the Ripening Process by HS-GC-IMS with PLS-DA. *Foods* **2023**, *12*, 551. [CrossRef] [PubMed]
8. Yue, R.R.; Zhang, Z.; Shi, Q.Q.; Duan, X.S.; Wen, C.P.; Shen, B.Q.; Li, X.A. Identification of the key genes contributing to the LOX-HPL volatile aldehyde biosynthesis pathway in jujube fruit. *Int. J. Biol. Macromol.* **2022**, *222*, 285–294. [CrossRef] [PubMed]
9. Liu, R.J.; Ma, L.; Meng, X.Y.; Zhang, S.W.; Cao, M.; Kong, D.C.; Chen, X.X.; Li, Z.Q.; Pang, X.M.; Bo, W.H. Volatile Profile Characterization of Jujube Fruit via HS-SPME-GC/MS and Sensory Evaluation. *Plants* **2024**, *13*, 1517. [CrossRef] [PubMed]
10. Wu, Y.S.; Zhang, W.W.; Yu, W.J.; Zhao, L.P.; Song, S.R.; Xu, W.P.; Zhang, C.X.; Ma, C.; Wang, L.; Wang, S.P. Study on the volatile composition of *table grapes* of three aroma types. *Lwt-Food Sci. Technol.* **2019**, *115*, 108450. [CrossRef]
11. Wang, J.Y.; Zhang, C.Y.; Li, M.; Fan, X.C.; Zhang, Y.; Jiang, J.F.; Liu, C.H.; Sun, L. Multi-omics analysis unravels the dynamics of flavor profile in 'Queen Nina' berries and the regulatory mechanism underlying volatile esters. *Food Chem. Mol. Sci.* **2025**, *10*, 100248. [CrossRef] [PubMed]
12. Yang, X.Y.; Zeng, P.P.; Wen, J.; Wang, C.L.; Yao, L.Y.; He, M. Gain deeper insights into traditional Chinese medicines using multidimensional chromatography combined with chemometric approaches. *Chin. Herb. Med.* **2024**, *16*, 27–41. [CrossRef] [PubMed]
13. Dash, M.; Singh, S.; Sahoo, S.; Kar, B. Targeted Analyte Deconvolution and Detection of *Piper longum* Leaf Volatiles Using Two-Dimensional Gas Chromatography and Time-of-Flight Mass Spectrometry. *Anal. Lett.* **2024**, *57*, 1462–1473. [CrossRef]
14. Tranchida, P.Q.; Sciarrone, D.; Dugo, P.; Mondello, L. Heart-cutting multidimensional gas chromatography: A review of recent evolution, applications, and future prospects. *Anal. Chim. Acta* **2012**, *716*, 66–75. [CrossRef] [PubMed]
15. Gou, M.; Bi, J.F.; Chen, Q.Q.; Wu, X.Y.; Fauconnier, M.L.; Qiao, Y.N. Advances and Perspectives in Fruits and Vegetables Flavor Based on Molecular Sensory Science. *Food Rev. Int.* **2023**, *39*, 3066–3079. [CrossRef]
16. Su, K.; Sun, S.; Chen, R.Y.; Ren, M.J.; Wang, Y.B.; Bi, S.; Liu, Y. Identification of key aroma-active compounds in five types of *Zanthoxylum bungeanum* Maxim. essential oils using a molecular sensory approach. *J. Food Compos. Anal.* **2025**, *145*, 107816. [CrossRef]
17. Sgorbini, B.; Cagliero, C.; Liberto, E.; Rubiolo, P.; Bicchi, C.; Cordero, C. Strategies for Accurate Quantitation of Volatiles from Foods and Plant-Origin Materials: A Challenging Task. *J. Agric. Food Chem.* **2019**, *67*, 1619–1630. [CrossRef] [PubMed]
18. Pozo-Bayón, M.A.; Guichard, E.; Cayot, N. Flavor control in baked cereal products. *Food Rev. Int.* **2006**, *22*, 335–379. [CrossRef]
19. Xu, Y.; Bi, S.; Xiong, C.H.; Dai, Y.X.; Zhou, Q.; Liu, Y. Identification of aroma active compounds in walnut oil by monolithic material adsorption extraction of RSC18 combined with gas chromatography-olfactory-mass spectrometry. *Food Chem.* **2023**, *402*, 134303. [CrossRef] [PubMed]
20. *GB/T 12311-2012*; Sensory Analysis Method-Triangle Test. Standards Press of China: Beijing, China, 2012.
21. Conti, C.; Guarino, M.; Bacenetti, J. Measurements techniques and models to assess odor annoyance: A review. *Environ. Int.* **2020**, *134*, 105261. [CrossRef] [PubMed]
22. Shi, C.H.; Yang, F.; Yan, L.C.; Wu, J.H.; Bi, S.; Liu, Y. Characterization of key aroma-active compounds in fresh and vacuum freeze-drying mulberry by molecular sensory science methods. *J. Food Compos. Anal.* **2024**, *133*, 106387. [CrossRef]
23. Jia, Y.Y.; Wang, C.; Zhang, Y.; Deng, W.K.; Ma, Y.C.; Ma, J.F.; Han, G. The Flavor Characteristics and Metabolites of Three Commercial Dried Jujube Cultivars. *Foods* **2024**, *13*, 1193. [CrossRef] [PubMed]
24. Pino, J.A.; Quijano, C.E. Study of the volatile compounds from plum (*Prunus domestica* L. cv. Horvin) and estimation of their contribution to the fruit aroma. *Cienc. E Tecnol. Aliment.* **2012**, *32*, 76–83. [CrossRef]

25. Li, P.H.; Jia, Y.L.; Cai, D.L.; Wang, X.Y.; Liu, J.H.; Zhu, R.C.; Wang, Z.T.; He, Y.; Wen, L.K. Study on the relationship between flavor components and quality of ice wine during freezing and brewing of '*beibinghong*' grapes. *Food Chem.-X* **2023**, *20*, 101016. [CrossRef] [PubMed]
26. López, R.; Ezpeleta, E.; Sánchez, I.; Cacho, J.; Ferreira, V. Analysis of the aroma intensities of volatile compounds released from mild acid hydrolysates of odourless precursors extracted from *Tempranillo* and *Grenache* grapes using gas chromatography-olfactometry. *Food Chem.* **2004**, *88*, 95–103. [CrossRef]
27. Das, G.; Nath, R.; Talukdar, A.D.; Agagündüz, D.; Yilmaz, B.; Capasso, R.; Shin, H.S.; Patra, J.K. Major Bioactive Compounds from Java Plum Seeds: An Investigation of Its Extraction Procedures and Clinical Effects. *Plants* **2023**, *12*, 1214. [CrossRef] [PubMed]
28. Zheng, W.C.; Yang, X.J.; Dang, B.; Zhang, W.A.; Zhang, J.; Feng, Y.J.; Feng, Z.S.; Zheng, J. Changes in secondary metabolites of grape skins in response to different postharvest dehydration temperatures as evaluated by UPLC-Q-TOF-MS. *J. Food Meas. Charact.* **2024**, *18*, 125–136. [CrossRef]
29. Yang, P.; Song, H.L.; Wang, L.J.; Jing, H. Characterization of Key Aroma-Active Compounds in Black Garlic by Sensory-Directed Flavor Analysis. *J. Agric. Food Chem.* **2019**, *67*, 7926–7934. [CrossRef] [PubMed]
30. Sommer, S.; Hoffmann, J.L.; Fraatz, M.A.; Zorn, H. Upcycling of black currant pomace for the production of a fermented beverage with *Wolfiporia cocos*. *J. Food Sci. Technol.-Mysore* **2023**, *60*, 1313–1322. [CrossRef] [PubMed]
31. Yang, P.; Yu, M.G.; Song, H.L.; Xu, Y.Q.; Lin, Y.P.; Granvogl, M. Characterization of Key Aroma-Active Compounds in Rough and Moderate Fire Rougui Wuyi Rock Tea (*Camellia sinensis*) by Sensory-Directed Flavor Analysis and Elucidation of the Influences of Roasting on Aroma. *J. Agric. Food Chem.* **2022**, *70*, 267–278. [CrossRef] [PubMed]
32. Dudareva, N.; Klempien, A.; Muhlemann, J.K.; Kaplan, I. Biosynthesis, function and metabolic engineering of plant volatile organic compounds. *New Phytol.* **2013**, *198*, 16–32. [CrossRef] [PubMed]

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