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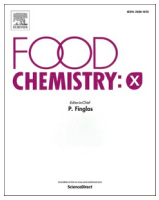

Comparative investigation on the aroma profiles of edible citrus flowers in the main organs and different developmental stages

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A R T I C L E I N F O

Chemical compounds studied in this article:
Ethyl 2-methylbutanoate (PubChem CID: 24020)
linalool (PubChem CID:6549)
β-myrcene (PubChem CID:31253)
2-butenal (PubChem CID: 447466)
D-limonene (PubChem CID:440917)
ethyl hexanoate (PubChem CID: 31265)
citral (PubChem CID: 638011)
(E,E)-2,4-heptadienal (PubChem CID: 5283321)
(E,Z)-2,6-nonadienal (PubChem CID: 643731)
and (E)-2-hexenal (PubChem CID: 5281168)
were obtained

Keywords:
Edible flowers
Different developmental stages
Volatiles
Odorants
Potential odorant marker

A B S T R A C T

Pomelo flowers emit a strong fragrance and give aromatic odors. Volatile compounds from pomelo flowers were analyzed at three developmental stages and in the main organs by molecular sensory science. A total of 134 volatiles including 25 odorants, were analyzed by gas chromatography–mass spectrometry/pulsed flame photometric detector (GC–MS/PFPD) and multidimensional GC–MS/olfactory (MDGC–MS/O). The total volatile content varied among pomelo flowers at different developmental stages (stage-III > stage-II > stage-I) and among different organs of pomelo flowers (petal > pistil > stamen). Linalool was an important odorant with a high OAV, and floral/fruity comprised the predominant aroma profile. Four odorants, ethyl 2-methylbutanoate, linalool, β-myrcene, and 2-butenal, were selected based on variable importance in projection (VIP) values and contributed mainly to the discrimination of pomelo flowers at three different developmental stages. Linalool, β-myrcene, D-limonene, and ethyl hexanoate were potential markers for evaluating flavor differences in pomelo floral organs.

1. Introduction

Citrus is the most widely cultivated fruit variety worldwide and is grown commercially in more than140 countries. There are many varieties of citrus, including orange, mandarin, pomelo, lemon, and lime etc. Pomelo (*Citrus grandis* (L.) Osbeck), also known as pummelo, shaddock, Chinese grapefruit, and *Limau Bali,* is a commercially important citrus cultivar that is characterized by a large appearance (Cheong et al., 2011). China is the largest producer of pomelo globally. According to a previous report, the production of fresh pomelo fruits in China reached 5.0 million tons, accounting for over 50% of all pomelo output worldwide (Makkumrai et al., 2021). In China, there are many commercial pomelo cultivars, such as Liangping pomelo, Guanxi pomelo, Shatian pomelo, and Wendan pomelo, that are extensively grown in Chongqing, Fujian, Guangxi, and Zhejiang city.

Pomelo flowers can be pollinated and grow into fruits. However, citrus trees normally produce many more flowers than they need. The number of flowers and flower buds can be reduced by tree regulation or thinning methods (manual, chemical and mechanical methods) during the growth process, which balances the flower capacity of trees and results in high-quality fruits (Romano et al., 2019; Stephenson, 1981). According to traditional experience, approximately 20% ~ 40% of flowers develop into fruit. As a result, more researchers are focusing on methods for obtaining valuable products through thinning flowers. Flower thinning technology can prevent excessive nutrient consumption of fruit trees, and can improve the fruit setting rate. Most pomelo flowers are discarded, resulting in a waste of resources. To fully utilize citrus flower resources, the unique aroma and rich nutritional value of citrus flowers have attracted the attention of many researchers, laying a foundation for the full utilization of flower resources. There were 180

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edible flowers worldwide, including citrus flowers (Feng et al., 2024). Citrus flowers are processed by microwave or hot air-drying technologies, citrus flowers are soaked in water or added as an ingredient to other products. Yang et al. (2024) used citrus flowers and green tea to make flower tea, and citrus green tea products are favored by consumers due to their unique citrus flavor. In addition, citrus flowers can be processed into essential oils for food, cosmetics and other industries.

Flowers emit a strong fragrance and aromatic odors. In studies on flower odorants, solid phase microextraction (SPME) (Wang et al., 2009), dynamic headspace (Ohashi et al., 2019) and solvent extraction (Darjazi, 2011) methods are commonly used to extract volatiles. Gas chromatographer (GC) matched mass spectrometry (MS), olfactory and flame ionization detector (FID) are commonly detection equipment (Darjazi, 2011; Dugo et al., 2011; Ohashi et al., 2019). Many studies have been performed on the volatiles of citrus flowers, mainly focusing on oranges and mandarin varieties. Behzad Babazadeh reported that 37 volatiles were detected in 'Page' mandarin flowers using GC − MS and GC-FID (Darjazi, 2011). Alissandrakis et al. (2003) reported that linalool was the predominant component in orange (51.6%), tangerine (75.2%) and sour orange (80.6%) flowers, and that eucalyptol was the main volatile component in lemon (35.7%). The content of volatiles in citrus flowers varied at different developmental stages and in different parts. As determined by Azam et al. (2013), the amount of volatiles was greatest in the half opened flowers of Yuhuan pomelo and Zaoxiang pomelo, and in the fully opened flowers of Ponkan, Satsuma, Qingjia, Liuben orange, Eureka, Bergamot and Huyou. In addition, it has been confirmed that linalool, limonene, β-pinene and β-ocimene are the main volatiles in different parts (flower buds, petals, filaments and anthers plus pollens) of buntan shaddock flowers (Huang et al., 2017). Citrus flowers are important spices that are widely used in the cosmetics, food, medicine and perfume industries (Mostafa et al., 2022).

However, there are very few studies on the chemical compositions and odorants of pomelo flowers at different developmental stages and in different organs. The olfactory characteristics and flavor economic value of pomelo flowers have not been explored in depth. Therefore, the main aim of this study was to identify volatile compounds, screen odorants and clarify the aroma profile of pomelo flowers at three different developmental stages and from three different organs (petal, stamen and pistil) of pomelo flowers by using molecular sensory approaches. In addition, multivariate statistical analysis was used to distinguish the flavor at three different developmental stages and in three different parts of the flowers.

2. Materials and methods

2.1. Samples preparation

Liangping pomelo is among the main pomelo varieties in China. In this study, an experiment was conducted using Liangping pomelo flowers. Approximately of 20 kg of pomelo flowers were picked from Liangping Farming, Chongqing, China, in 2021. Flower samples were harvested during full blossoming and separated into three different stages, i.e., stage-I, stage-II and stage-III (Fig. S1). Three different organs (petal, stamen and pistil) of fully blossom pomelo flowers were also collected (Fig. S1). The samples were immediately prepared These flowers and different organs of flowers were immediately frozen with liquid nitrogen, and then crushed into powder by a shredder, which was stored at −80 °C until analysis. Each sample was assessed in triplicate.

2.2. Reagents and chemicals

Cyclohexanone (99%), dimethyl sulfide (99%), ethyl methyl sulfide (96%), a-pinene (98%), β-myrcene (90%), p-cymenene(99.5%), hexanal (99%), camphene (95%), (E)-2-hexenal (98%), citronellol (95%), ethyl 2-methylbutanoate (98%), ethyl hexanoate (99%), and methyl-heptenone (98%) were obtained from Aladdin Industrial Co. (Shanghai, China). Carbon disulfide (99%) was purchased from TCI Chemical Co. (Shanghai, China). n-Alkanes (C5-C20), D-carvone (97%), D-Limonene (97%) were purchased from Sigma-Aldrich Co. (Shanghai, China). (E,Z)-2,6-Nonadienal (98%) and linalool (98%) were acquired from Shanghai Macklin Biochemical Co. (Shanghai, China). Citral (98%), and neral (98%) were purchased from Shanghai Yuanye Bio-Technology Co. (Shanghai, China). Neryl acetate (95%) was purchased from Meryer Technology Co. (Shanghai, China).

2.3. Headspace solid-phase microextraction (SPME)

The volatiles of flowers were extracted using SPME according to a previously described method (Cheng et al., 2023) with modifications. Cyclohexanone (9.423 mg/mL) and ethyl methyl sulfide (8.42 mg/L) were used as internal standards. The frozen flower powder (0.5 g) with 5 mL of saturated sodium chloride solution was placed in a 20 mL vial that was previously mixed by vortex oscillations. A total of 1 μL of ethyl methyl sulfide and 1.5 μL of cyclohexanone internal standards were added to the vial. The vial was capped with a PTFE-silicon septum. The vial with flower powder was equilibrated at 40 °C in a water bath for 20 min. A 2-cm SPME fiber (50/30 μm DVB/CAR/PDMS, Supelco, Bellefonte, PA, USA) was exposed to the headspace for 30 min with instant agitation. Afterward, the fiber was desorbed for 5 min at 230 °C in the injector of the GC.

2.4. Gas chromatography-mass spectrometry/pulsed flame photometric detector (GC–MS/PFPD)

GC–MS/PFPD analysis was performed using a 7890B GC (Agilent, Palo Alto, CA, USA) instrument equipped with an Agilent 5977 A MS detector and a 5380 PFPD (OI Analytical Co., College Station, TX, USA). Volatile compounds were separated using DB-5 (30 m × 0.25 mm id × 0.25 μm). Helium was used as carrier gas at a flow rate of 1.2 mL/min, and the GC inlet was set in spitless mode. The oven temperature was set at 35 °C, held for 6 min, and increased to 203 °C at 6 °C/min and held for 10 min, and then increased to 243 °C at 10 °C/min with a final hold for 3 min. The MS transfer line and ion source temperature were set to 280 °C and 230 °C, respectively. The electron ionization (EI) mode was implemented at 70 eV, and the scanning range was from *m/z* 33–500 amu. The temperature of the PFPD was set at 250 °C, and the sulfur gate was opened between 5 and 24 ms. The PFPD output was recorded in square root mode.

2.5. Multidimensional gas chromatography–mass spectrometry/olfactometry (MDGC-MS/O) with time-intensity

MDGC-MS/O analyses were carried out as described previously (Cheng et al., 2023) with modifications. Chromatography was performed using an Agilent 7890B MDGC equipped with a 5977B MS and an olfactometric detector (Volatile Analysis Co., Grant, USA). The odorants were separated and evaluated using a nonpolar BP-5 column (^{1}D, 30 m × 0.53 mm × 0.5 μm) and a polar SLOGEL-wax column (^{2}D, 30 m × 0.53 mm × 0.5 μm). The oven temperature was increased from 35 °C to 70 °C at 7 °C/min, raised to 100 °C at 100 °C/min, increased to 140 °C at 4 °C/min, and raised to 212 °C at 6 °C/min. Helium was the carrier gas at a flow rate of 8.7345 mL/min for the ^{1}D column and 8.7663 mL/min for the ^{2}D column. Three panelists were trained as described with some modifications from Dreher et al. (2003). Ten mixed odorant (dimethyl sulfide, methional, ethyl acetate, ethyl propionate, hexenal, α-pinene, limonene, γ-terpinene, linalool, and terpinen-4-ol) solutions were used to help familiarize the panelists with odor descriptions and time-intensity scale recording by the computer automatically. The intensity of the odorants was normalized so that each panelist was given a score of 10 for the highest intensity. A peak was considered an odorant when over half of the panel responses smelled a similar scent at the same retention time. Each panelist repeated the

experiment in triplicate, and the intensity of the odorant was the average of three panelists. In addition, this equipment also equipped with a flame ionization detector (FID). The deans switch allowed volatile compounds to elute from the ^{1}D column into an olfactometric detector and FID. In the GC-FID/O system, the parameters of the oven and sniffing port were same as the described with those in the MDGC-MS/O system. The temperature of the FID was set at 280 °C. GC-FID/O was only used to assist in the identification of odorants in MDGC-MS/O systems.

2.6. Qualitative and quantitative analysis of volatile compounds

In the GC–MS/PFPD system, PFPD was used to determine volatile sulfur compounds (VSCs). Linear retention index (LRI) values were calculated from a series of alkane standards (C5-C20). By matching the mass spectra from the mass spectral library (NIST11, W10N14), comparing the LRI values from MS or PFPD detector responses with the LRI values from the database (https://www.vcfonline.nl/VcfCompoundsearch.cfm), and comparing the retention times of volatiles and authentic standards, the volatile compounds of flowers were identified. The odorants were identified by matching the mass spectral library (NIST11, W10N14), aroma descriptors, authentic standards, and LRIs from the response of the FID detector.

Ethyl methyl sulfide was used to calculate the content of VSCs in edible citrus flowers. In addition to volatile sulfur compounds, non-sulfur volatile compounds were quantified by using internal standard cyclohexanone.

2.7. Calculation of odor activity values (OAVs)

The OAVs of the volatile compounds were measured as the ratio of the odorant concentration to the odorant threshold. The thresholds were obtained from the references. Usually, volatile compounds with OAV $\geq$ 1 indicate that the volatiles contribute to the overall flavor (Callejón et al., 2008).

2.8. Statistical analysis

The figures were plotted by using Origin 7.5 (OriginLab, Northampton, MA). Principal component analysis (PCA), partial least squares (PLS) regression analysis, and orthogonal partial least squares discriminant analysis (OPLS-DA) were carried out for multivariate statistical analysis in SIMCA 14.1 software.

3. Results and discussion

3.1. Identification of volatile compounds in different developmental stages and parts of pomelo flowers

To comprehensively detect volatile compounds in pomelo flowers, volatile compounds were separated through a polar (wax) column and tandem polar-nonpolar columns, and MS, O, FID and PFPD were used for detection. As shown in Table 1, a total of 134 volatiles were positively identified in different stages (stage-I, stage-II and stage-III) and organs (petal, stamen and pistil) of pomelo flowers by GC–MS/PFPD and MDGC–MS/O; these volatiles included 51 terpenes, 27 alcohols, 23 aldehydes, 12 ketones, 10 esters, 8 other compounds and 3 VSCs. Among these compounds, 47 common volatile compounds were determined in the different detection equipment (Fig. 1). In previous studies, 66 and 53 volatiles were detected in Yuhuan pomelo flowers and Zaoxiang pomelo flowers, respectively, at three blooming stages by using GC–MS (Azam et al., 2013). The number of volatile compounds was different in different developmental stages and parts of pomelo flowers. A total of 104, 106 and 107 volatile compounds were detected in stage-I, stage-II and stage-III, respectively. In total, 99, 77, and 93 volatiles were detected in the petal, stamen and pistil parts of pomelo flowers,

Table 1
Identification of volatile compounds in Pomelo flowers at different developmental stages and in the main organs.

Compound	RI Calculated	Odor descriptor	Identification		
			GC-MS/PFPD	GC-FID/O	MDGC-MS/O
sulfur compounds					
hydrogen sulfide	<500	–	PFPD, RI	–	–
dimethyl sulfide	519	–	PFPD, RI, Std	–	–
carbon disulfide	553	–	PFPD, RI, Std	–	–
terpenes					
a-thujene	928	–	MS, RI	–	MS
a-pinene	934	piney	MS, RI	AD, RI	MS, AD, Std
camphene	950	camphor	MS, RI	AD, RI	MS, AD, Std
sabinene	973	–	MS, RI	–	–
β-pinene	978	–	MS, RI	–	MS
β-myrcene	990	herbal	MS, RI	AD, RI	MS, AD, Std
cosmene	1006	–	MS, RI	–	–
a-terpinene	1016	–	MS, RI	–	–
D-limonene	1030	fatty, citrusy	MS, RI	AD, RI	MS, AD, Std
β-phellandrene	1031		MS, RI	–	MS
(*Z*)-β-ocimene	1038	–	MS, RI	–	MS
β-ocimene	1046	–	MS, RI	–	–
γ-terpinene	1059	–	MS, RI	–	MS
terpinolene	1087	–	MS, RI	–	–
p-cymenene	1089	petrol	MS, RI	AD, RI	MS, AD, Std
p-1,3,8-menthatriene	1112	–	MS, RI	–	–
alloocimene	1129	–	MS, RI	–	–
bornylene	1228	–	MS, RI	–	–
δ-elemene	1338	–	MS, RI	–	–
α-cubebene	1349	–	MS, RI	–	–
α-ylangene	1372	–	MS, RI	–	–
copaene	1376	–	MS, RI	–	–
(−)-β-elemene	1388	–	MS, RI	–	MS
β-elemene	1391	–	MS, RI	–	–
tetradecane	1400	–	MS, RI	–	–
γ-caryophyllene	1405	–	MS, RI	–	–
caryophyllene	1420	–	MS, RI	–	MS
γ-elemene	1430	–	MS, RI	–	–
calarene	1433	–	MS, RI	–	–
aromadendrene	1440	–	MS, RI	–	–
(*Z*)-β-farnesene	1444	–	MS, RI	–	–
α-humulene	1454	–	MS, RI	–	MS
γ-muurolene	1481	–	MS, RI	–	MS
germacrene D	1481	–	MS, RI	–	MS
1,4-cadinadiene	–	–	MS	–	–
a-farnesene	1506	–	MS, RI	–	–
(−)-β-bisabolene	1503	–	MS, RI	–	–
γ-cadinene	1513	–	MS, RI	–	MS
δ-cadinene	–	–	MS	–	–
calamenene	1525	–	MS, RI	–	MS
β-sesquiphellandrene	1522	–	MS, RI	–	MS
cubenene	1530	–	MS, RI	–	–
α-Cadinene	–	–	MS	–	–
germacrene B	1556	–	MS, RI	–	–
p-cymene	–	–	–	–	MS
4-ethyl-1,2-dimethylbenzene	–	–	–	–	MS
(4E,6Z)-alloocimene	–	–	–	–	MS
p-1,3,8-menthatriene	–	–	–	–	MS
(*E*)-β-farnesene	–	–	–	–	MS
alloaromadendrene	–	–	–	–	MS
α-selinene	–	–	–	–	MS
Aldehydes					
acetaldehyde	434	–	MS, RI	–	MS
pentanal	701	–	MS, RI	–	–
hexanal	801	grassy	MS, RI	AD, RI	MS, AD, Std

(*continued on next page*)

Table 1 (*continued*)

Compound	RI Calculated	Odor descriptor	Identification		
			GC-MS/PFPD	GC-FID/O	MDGC-MS/O
(E)-2-hexenal	851	green	MS, RI	AD, RI	MS, AD, Std
benzaldehyde	961	bitter almond	MS, RI	AD, RI	MS, AD
(E,E)-2,4-heptadienal	1007	package, plastic	MS, RI	AD, RI	MS, AD
(E)-2-octenal	1059	–	MS, RI	–	–
R-(+)-citronellal	1052	–	MS, RI	–	–
lilac aldehyde C	1156	–	MS, RI	–	MS
(E)-2-nonenal	1160	–	MS, RI	–	–
geranial	1269	–	MS, RI	–	–
p-1,8-menthadien-7-al	1274	–	MS, RI	–	–
neral	1239	fruity	MS, RI	AD, RI	MS, AD, Std
2-butenal	–	chemical	MS, RI	AD, RI	MS, AD
(E)-2-pentenal	–	–	–	–	MS
citronellal	–	–	–	–	MS
(E,*Z*)-2,6-nonadienal	–	fresh	–	AD, RI	AD
phenylacetaldehyde	–	fatty, pungent	–	AD, RI	AD
myrtenal	–	–	–	–	MS
citral	–	lemony	–	AD, RI	MS, AD, Std
tetradecanal	–	–	–	–	MS
pentadecanal	–	–	–	–	MS
(E,E)-farnesal	–	–	–	–	MS
		Alcohols			
ethanol	551	–	MS, RI	–	MS
1-pentanol	765	medicine	MS, RI	AD, RI	MS, AD
(E)-2-hexen-1-ol	860	–	MS, RI	–	–
1-hexanol	868	–	MS, RI	–	MS
trans-4-thujanol	1089	–	MS, RI	–	–
linalool	1100	floral	MS, RI	AD, RI	MS, AD, Std
cis-p-2,8-menthadien-1-ol	1136	–	MS, RI	–	–
trans-(−)-pinocarveol	1140	–	MS, RI	–	–
terpinen-4-ol	1178	–	MS, RI	–	–
myrtenol	1193	–	MS, RI	–	MS
carveol	1215	–	MS, RI	–	MS
trans-carveol	1217	–	MS, RI	–	–
nerol	1227	–	MS, RI	–	–
cis-carveol	1229	–	MS, RI	–	MS
geraniol	1256	–	MS, RI	–	MS
p-8-menthene-1,2-diol	–	–	MS	–	MS
trans-p-2,8-menthadien-1-ol	1559	herbal	MS, RI	–	MS
nerolidol	1559	–	MS, RI	–	MS
(−)-spathulenol	1571	–	MS, RI	–	MS
1-penten-3-ol	–	–	–	–	MS
isocarveol	–	–	–	–	MS
cis-verbenol	–	–	–	–	MS
a-terpineol	–	–	–	–	MS
citronellol	–	citrusy	–	AD, RI	MS, AD, Std
spathulenol	–	–	–	–	MS
farnesol	–	–	–	–	MS
(Z,E)-farnesol	–	–	–	–	MS
esters	–				
ethyl acetate	613	–	MS, RI	–	–
(Z)-3-hexenyl butanoate	1186	–	MS, RI	–	–
methyl geranate	1323	–	MS, RI	–	–
methyl anthranilate	1340	–	MS, RI	–	–
neryl acetate	1364	floral, fruity	MS, RI	AD, RI	MS, AD, Std
ethyl anthranilate	1415	–	MS, RI	–	MS

Table 1 (*continued*)

Compound	RI Calculated	Odor descriptor	Identification		
			GC-MS/PFPD	GC-FID/O	MDGC-MS/O
ethyl 2-methylbutanoate	–	fruity	–	AD, RI	MS, AD, Std
ethyl hexanoate	–	overripe fruit	–	AD, RI	MS, AD, Std
cis-sabinene hydrate	–	–	–	–	MS
geranyl acetate	–	floral	–	AD, RI	MS, AD
ketones	–				
1-penten-3-one	682	–	MS, RI	–	MS
methylheptenone	985	fruity	MS, RI	AD, RI	MS, AD, Std
(E,*E*)-3,5-octadien-2-one	1067	–	MS, RI	–	–
pinocarvone	1161	–	MS, RI	–	MS
trans-dihydrocarvone	1201	–	MS, RI	–	MS
D-carvone	1256	herbal	MS, RI	AD, RI	MS, AD, Std
4-acetyl-1-methylcyclohexene	–	green. Medicine	–	AD, RI	MS, AD
geranylacetone	–	–	–	–	MS
3,5-octadien-2-one	–	–	–	–	MS
dihydrocarvone	–	–	–	–	MS
4-isopropyl-2-cyclohexen-1-one	–	–	–	–	MS
6-methyl-3,5-heptadien-2-one	–	–	–	–	MS
other compounds	–				
limonene 1,2-oxide	1132	–	MS, RI	–	–
trans-limonene 1,2-oxide	1140	–	MS, RI	–	–
indole	1295	–	MS, RI	–	MS
caryophyllene oxide	1580	woody	MS, RI	AD, RI	MS, AD
p-xylene	–	–	–	–	MS
o-xylene	–	–	–	–	MS
(+)-(E)-limonene oxide	–	–	–	–	MS
humulene epoxide II	–	–	–	–	MS

RI of standard compounds; RI (reference) come from the library: https://www.vcf-online.nl/VcfCompoundSearch.cfm and https://www.odour.org.uk/index.htmL.

AD, aroma descriptor; std., authentic standards.

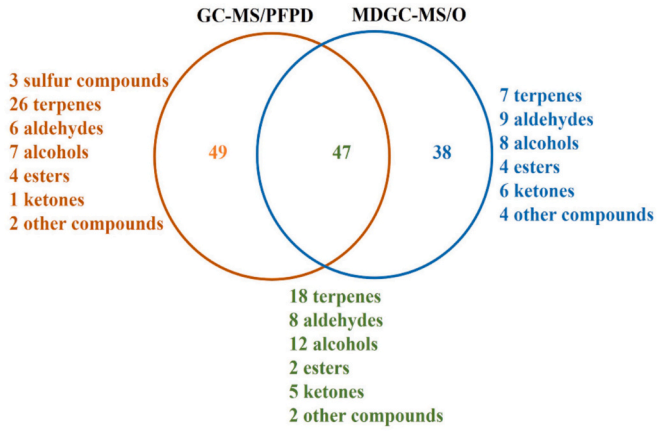

Fig. 1. Venn diagram of volatile compounds in Pomelo flowers at different developmental stages and in the main organs of flowers by GC–MS/PFPD and MDGC–MS-O.

respectively. The sulfur volatile group was first confirmed in pomelo flowers. Three VSCs, hydrogen sulfide, dimethyl sulfide and carbon disulfide, were observed in all developmental stages and organs of pomelo

flowers. These three VSCs have been reported in citrus fruits, such as mandarin juice (Cheng et al., 2023) and orange juice (Perez-Cacho et al., 2007). Among the different developmental pomelo flower stages, there were more terpenes and alcohols in stage-II and stage-III in pomelo flowers than in stage-I, while there were fewer esters and ketones than those in stage-I. Among the different parts of pomelo flowers, the number of terpenes, aldehydes and esters in petals was greater, and the number of alcohols and ketones in pistils was greater than that in other parts.

3.2. Quantification of volatile compounds in different developmental stages and parts of pomelo flowers

The volatile compounds were quantified in pomelo flowers by cyclohexanone and ethyl methyl sulfide, and the results are indicated in Table 2 and Fig. 2. In terms of pomelo flower growth, the greatest pomelo flower concentration was found in stage-III (1568.72 μg/g), followed by stage-II(1295.33 μg/g) and stage-I (1104.09 μg/g). Therefore, the total content of volatiles in pomelo flowers increased as the pomelo flowers grew. Terpenes comprised 36.98–65.69% of the total volatiles in pomelo flowers, followed by alcohols (16.39% ~ 27.32%), aldehydes (7.70% ~ 13.28%), other compounds (3.45% ~ 17.84%), ketones (2.03% ~ 5.45%), esters (0.32% ~ 0.72%) and VSCs (0.10% ~ 0.59%). The total content of volatile compounds such as aldehydes, alcohols, VSCs and other compounds increased as the pomelo flowers grew. The highest content of terpenes and the lowest content of esters and ketones were observed in pomelo flowers at stage-II. The concentration of D-limonene, the most predominant compound in pomelo flowers at stage-I (431.14 ± 24.01 μg/g) and stage-II (325.74 ± 3.18 μg/g), decreased gradually during the growth stage. Linalool, the most abundant compound in pomelo flowers at stage-III (281.35 ± 2.52 μg/g), gradually increased during the growth stage. Flamini and Cioni (2010) also reported that the content of linalool increased as grapefruit flowers bloomed, and became the most abundant volatile (37.1%) in mature grapefruit flowers.

An increasing trend was observed for the contents of 32 volatile compounds during the growth stage of pomelo flowers, and a decreasing trend was observed for the content of 20 volatile compounds. Volatile compounds were distributed differently in the petals, stamens and pistils of pomelo flowers. Petal (1603.09 μg/g) contained the most volatile compounds, followed by pistil (1008.18 μg/g) and stamen (554.43 μg/g). Compared with the other parts of the pomelo flower, the pistil part contained the highest amounts of VSCs and ketones, and the petal part contained the highest contents of terpenes, aldehydes, alcohols, esters and other compounds. Terpenes and alcohols are the main classes in the petal part of pomelo flowers, accounting for 47.29% and 31.22%, respectively, of the total emissions. Terpenes and aldehydes were the most represented classes in the pistil part of pomelo flowers, accounting for 62.47% and 17.62%, respectively. Alcohols and aldehydes were the main volatile compounds in the stamen part of pomelo flowers, accounting for 52.78% and 27.63%, respectively. The contents of 8 (linalool, β-myrcene, D-limonene, (*Z*)-β-ocimene, β-pinene, (E)-2-hexenal, nerolidol, hexanal), 2 (linalool and (E)-2-hexenal), and 7 (β-myrcene, β-ocimene, D-limonene, β-pinene, (E)-2-hexenal, hexanal and linalool) major volatiles in the petal, stamen and pistil of pomelo flowers, respectively, were >50 μg/g.

3.3. Odorants identified by MDGC–MS/O

MDGC-O with a time-intensity method (OMSE) was used to determine the relative intensities of odorants at different developmental stages and in different parts of pomelo flowers. The 25 odorants detected in the pomelo flowers were identified by odor description and mass spectra, and the odorants were not identified. Regarding three different growth stages and three main parts of pomelo flowers, 21, 21, 21, 19, 17, and 18 odorants were identified in stage-I, stage-II, and stage-III and in the petal, stamen, and pistil, respectively (Table 3). Terpenes and aldehydes were the most abundant odor-active compounds in the three developmental stages and the three parts of pomelo flowers. Among the 25 odorants, 7, 5, 11, 10, 4, and 8 odorants were classified as having at least high intensity (≥4) in stage-I, stage-II, and stage-III and the petal, stamen, and pistil of flowers, respectively. The most potent odor-active compounds in each sample were D-limonene (odor intensity = 7.1), linalool (5.5), and trans-p-2,8-menthadien-1-ol (5.3) in the flowers at stage-I; linalool (7.0), D-limonene (6.8), and (E)-2-hexenal (5.6) in the flowers at stage-II; linalool (8.5), β-myrcene (5.8) and (E)-2-hexenal (5.7) in the flowers at stage-III; linalool (8.8), hexanal (5.95), and D-limonene (5.8) in the flower petal; linalool (7.4), 2-butenal (4.65), and (E)-2-hexenal (4.65) in the flower stamen; and hexanal (6), (E)-2-hexenal (5.5), and linalool (5.5) in the flower pistil. (See Table 3.)

Among the total odorants detected across the three growth stages of pomelo flowers, there were five terpenes (a-pinene, camphene, β-myrcene, D-limonene, p-cymenene), eight aldehydes (2-butenal, hexanal, (E)-2-hexenal, (E,E)-2,4-heptadienal, benzaldehyde, phenylacetaldehyde, (E,Z)-2,6-nonadienal, neral and citral), two alcohols (linalool and trans-p-2,8-menthadien-1-ol), two esters (ethyl 2-methylbutanoate and ethyl hexanoate), one ketone (6-methyl-5-hepten-2-one) and a pyran (caryophyllene oxide). Citral with lemony odor was observed in pomelo flowers at stage-II and stage-III. The odor intensities of the 18 odorants varied with concentration during different the growth stages of the flowers. Among these odorants, the odor intensity of six odorants (β-myrcene, hexanal, (E)-2-hexenal, neral, citral, and linalool) showed an increasing trend, and four odorants (D-limonene, (E,E)-2,4-heptadienal, (E,Z)-2,6-nonadienal, ethyl 2-methylbutanoate) exhibited an opposite trend for the developmental stages of the flowers. D-limonene, with a relatively high intensity ranging from 6.8 to 7.1, was the greatest contributor to the overall flavor of stage-I and stage-II pomelo flowers. Linalool with floral was the most significant odorant in pomelo flowers at stage-III.

3.4. Forming an aroma profile using aroma categories and combined MDGC–MS/O intensity

The odorants with similar sensory descriptors in Table 3 were grouped into four general aroma categories based on their main odor character (floral/fruity, green/grassy/fresh, pungent/bitter almond, and herbal/piney/woody). Most of these sensory descriptors have been reported in previous citrus flower studies (Cheong et al., 2011; Pellizzeri et al., 2020). As shown in Fig. 3, floral/fruity aroma descriptors exhibited the highest combined aroma intensity for whole pomelo flowers and the petal and stamen of pomelo flowers, accounting for 33.67% ~ 39.56% of the total aroma intensity. Floral/fruity aromas were mainly related to twelve odorants, including linalool, geranyl acetate, (E,E)-2,4-heptadienal, D-limonene, neral, citral, citronellol, ethyl 2-methylbutanoate, ethyl hexanoate, neryl acetate, 6-methyl-5-hepten-2-one and one unidentified compound. During the growth of pomelo flowers, the floral/fruity intensity gradually increased, and there were significant differences among the three stages of pomelo flower development ($p < 0.01$). The distribution of floral/fruity aromas differed among the different parts of the pomelo flower, among which the flower petals exhibited the strongest floral/fruity aroma, followed by the stamen and pistil parts. Herbal/piney/woody aromas were the most intense for the pistils and the second most intense for the whole pomelo flower, flower petal and stamen parts. In this study, the herbal/piney/woody was composed of a-pinene, camphene, caryophyllene oxide, β-myrcene, trans-p-2,8-menthadien-1-ol, carvone and one identified compound. The third most intense aroma for the flower pistil part and pomelo flower at stage-II and stage-III was green/grassy/fresh. The compounds leading to the green/grassy/fresh odor include hexanal, (E)-2-hexenal, (E,Z)-2,6-nonadienal and one unknown fresh compound. Aldehydes greatly contribute to green/grassy/fresh aromas, which has also been reported in a previous study (Du et al., 2015). Pungent/bitter almond/garlic has

Table 2
Quantification of volatile compounds in Pomelo flowers at different developmental stages and in the main organs.

Compound	Concentration μg/g					
	Flower at different stages			Flower at different parts		
	stage-I	stage-II	stage-III	Petal	Stamen	pistil
sulfur compounds						
hydrogen sulfide	0.01 ± 0.00	0.19 ± 0.02	0.30 ± 0.04	0.09 ± 0.01	0.14 ± 0.01	0.19 ± 0.01
dimethyl sulfide	0.60 ± 0.02	1.98 ± 0.05	6.99 ± 0.62	3.86 ± 0.11	2.29 ± 0.06	5.17 ± 0.30
carbon disulfide	0.52 ± 0.01	1.07 ± 0.01	2.05 ± 0.20	0.60 ± 0.01	0.99 ± 0.02	5.22 ± 0.19
terpenes						
a-thujene	4.04 ± 0.14	6.79 ± 0.11	6.51 ± 0.12	6.67 ± 0.06	0	6.79 ± 0.08
a-pinene	12.47 ± 0.25	24.41 ± 0.56	5.64 ± 0.18	8.73 ± 0.12	2.13 ± 0.09	5.91 ± 0.49
camphene	trace	3.38 ± 0.19	1.55 ± 0.03	2.05 ± 0.10	trace	1.44 ± 0.02
sabinene	23.19 ± 0.69	33.43 ± 1.97	35.42 ± 0.21	40.64 ± 0.71	1.73 ± 0.04	31.70 ± 0.64
β-pinene	84.92 ± 2.83	105.89 ± 3.70	108.92 ± 0.23	127.30 ± 2.03	11.34 ± 0.35	98.10 ± 0.14
β-myrcene	18.42 ± 2.31	24.20 ± 0.91	218.00 ± 0.60	166.36 ± 2.76	12.91 ± 0.25	209.23 ± 0.15
cosmene	5.79 ± 0.01	4.41 ± 0.12	3.05 ± 0.00	0	1.04 ± 0.04	3.62 ± 0.08
a-terpinene	0.00	0.00	1.38 ± 0.09	1.41 ± 0.01	1.01 ± 0.03	6.02 ± 0.22
ᴅ-limonene	431.14 ± 24.01	325.74 ± 3.18	111.08 ± 2.05	151.21 ± 6.54	13.55 ± 0.09	99.47 ± 3.32
β-phellandrene	0	0	0	0	0.53 ± 0.03	0
(Z)-β-ocimene	32.58 ± 1.97	173.22 ± 8.88	2.31 ± 0.12	137.55 ± 0.24	2.93 ± 0.06	0
β-ocimene	0	0	0	0	13.14 ± 0.25	110.02 ± 14.60
γ-terpinene	5.31 ± 1.71	5.11 ± 0.83	5.24 ± 0.62	4.86 ± 0.05	1.08 ± 0.02	4.54 ± 0.08
terpinolene	3.83 ± 0.54	3.62 ± 0.61	2.69 ± 0.38	3.48 ± 0.09	0.00	2.18 ± 0.03
p-cymenene	2.65 ± 0.53	1.64 ± 0.25	10.15 ± 0.12	3.74 ± 0.11	trace	trace
p-1,3,8-menthatriene	1.89 ± 0.32	3.92 ± 0.28	6.87 ± 0.32	8.00 ± 0.02	0.00	5.42 ± 0.06
alloocimene	0.00	2.93 ± 0.56	0.00	5.08 ± 0.13	0.17 ± 0.00	12.58 ± 0.62
bornylene	0.00	1.49 ± 0.00	0.00	0.66 ± 0.01	0.00	0.00
δ-elemene	0.52 ± 0.06	11.07 ± 0.41	0.51 ± 0.01	3.14 ± 0.63	0.00	0.68 ± 0.01
α-cubebene	1.38 ± 0.13	1.66 ± 0.13	0.82 ± 0.06	1.20 ± 0.01	0.00	0.00
α-ylangene	0.53 ± 0.01	0.70 ± 0.03	0.88 ± 0.02	1.87 ± 0.03	0.54 ± 0.04	0.68 ± 0.05
copaene	0.52 ± 0.17	0.87 ± 0.06	0.53 ± 0.01	0.70 ± 0.01	0.45 ± 0.01	0.00
(−)-β-elemene	0.36 ± 0.07	0.30 ± 0.03	0.31 ± 0.01	0.35 ± 0.00	0.00	0.00
β-elemene	4.10 ± 0.43	4.17 ± 0.25	2.86 ± 0.55	3.82 ± 0.02	0.00	1.47 ± 0.12
tetradecane	0.75 ± 0.20	1.10 ± 0.07	0.78 ± 0.02	0.78 ± 0.01	0.47 ± 0.03	0.55 ± 0.05
γ-caryophyllene	0.40 ± 0.03	0.00	0.00	0.00	0.00	0.00
caryophyllene	44.02 ± 3.63	36.54 ± 2.48	10.38 ± 0.41	18.01 ± 0.33	1.02 ± 0.04	6.85 ± 0.48
γ-elemene	1.44 ± 0.14	1.67 ± 0.02	1.08 ± 0.02	1.47 ± 0.12	0.17 ± 0.01	0.54 ± 0.01
calarene	0.42 ± 0.09	0.56 ± 0.06	0.29 ± 0.00	0.41 ± 0.03	0.00	0.00
aromadendrene	0.57 ± 0.18	1.13 ± 0.04	0.99 ± 0.04	2.15 ± 0.14	0.85 ± 0.08	0.00
(Z)-β-farnesene	0.59 ± 0.16	1.44 ± 0.04	1.86 ± 0.00	3.73 ± 0.02	2.26 ± 0.02	0.49 ± 0.03
α-humulene	4.99 ± 0.69	3.94 ± 0.04	1.70 ± 0.15	2.71 ± 0.00	0.12 ± 0.00	1.03 ± 0.02
γ-muurolene	1.10 ± 0.16	1.57 ± 0.03	0.62 ± 0.05	1.11 ± 0.05	0.00	0.00
germacrene d	3.92 ± 0.17	6.47 ± 0.10	2.13 ± 0.27	5.49 ± 0.03	0.46 ± 0.06	0.74 ± 0.06
1,4-cadinadiene	0.00	1.33 ± 0.03	0.00	1.16 ± 0.04	0.00	0.00
a-farnesene	1.51 ± 0.12	5.85 ± 0.32	3.89 ± 0.30	12.55 ± 0.53	5.11 ± 0.11	1.26 ± 0.02
(−)-β-bisabolene	0.00	0.00	0.35 ± 0.03	0.70 ± 0.01	0.55 ± 0.10	0.00
γ-cadinene	0.55 ± 0.03	1.09 ± 0.01	0.34 ± 0.02	0.62 ± 0.07	0.00	0.00
δ-cadinene	0.96 ± 0.04	1.82 ± 0.04	0.65 ± 0.05	1.16 ± 0.12	0.00	0.00
calamenene	0.41 ± 0.02	0.44 ± 0.02	0.34 ± 0.03	0.64 ± 0.07	0.00	0.00
β-sesquiphellandrene	0.00	0.00	0.00	0.00	0.55 ± 0.06	0.00
cubenene	0.16 ± 0.04	0.28 ± 0.02	0.00	0.25 ± 0.01	0.00	0.00
α-cadinene	0.19 ± 0.08	0.36 ± 0.00	0.17 ± 0.00	0.37 ± 0.01	0.00	0.00
germacrene b	0.88 ± 0.23	1.42 ± 0.11	0.82 ± 0.01	0.00	0.00	0.38 ± 0.02
p-cymene	0.00	0.00	5.31 ± 0.19	0.00	0.90 ± 0.01	3.50 ± 0.23
4-ethyl-1,2-dimethylbenzene	5.33 ± 0.09	2.09 ± 0.03	2.96 ± 0.26	1.47 ± 0.10	0.00	2.63 ± 0.07
(4E,6Z)-alloocimene	1.43 ± 0.09	3.40 ± 0.03	1.43 ± 0.07	1.59 ± 0.00	0.77 ± 0.01	1.25 ± 0.15
p-1,3,8-menthatriene	14.44 ± 0.29	5.40 ± 0.05	8.21 ± 0.16	3.96 ± 0.13	1.05 ± 0.03	6.61 ± 0.13
(E)-β-farnesene	0.00	2.09 ± 0.07	2.13 ± 0.07	4.33 ± 0.07	3.94 ± 0.19	0.74 ± 0.00
alloaromadendrene	3.91 ± 0.16	5.33 ± 0.22	2.43 ± 0.27	2.79 ± 0.00	0.00	1.80 ± 0.00
α-selinene	3.59 ± 0.17	15.24 ± 0.20	6.51 ± 0.08	11.93 ± 0.22	3.63 ± 0.15	1.59 ± 0.09
aldehydes						
acetaldehyde	1.48 ± 0.32	1.84 ± 0.24	2.09 ± 0.35	1.36 ± 0.00	1.35 ± 0.09	1.89 ± 0.09
pentanal	1.29 ± 0.00	0.13 ± 0.00	0.82 ± 0.01	0.00	0.00	1.42 ± 0.00
hexanal	18.14 ± 1.09	41.32 ± 1.65	46.22 ± 0.80	53.86 ± 0.41	44.81 ± 0.74	58.44 ± 0.60
(E)-2-hexenal	21.43 ± 0.05	82.49 ± 0.15	80.62 ± 2.18	78.09 ± 3.41	67.54 ± 0.78	86.26 ± 1.72
benzaldehyde	3.58 ± 0.13	2.22 ± 0.06	3.83 ± 0.14	3.44 ± 0.20	2.91 ± 0.30	2.47 ± 0.02
(E,E)-2,4-heptadienal	6.46 ± 0.05	2.76 ± 0.13	2.14 ± 0.07	0.90 ± 0.03	1.64 ± 0.05	4.41 ± 0.28
(E)-2-octenal	0.86 ± 0.04	2.59 ± 0.17	0.46 ± 0.00	2.36 ± 0.01	2.53 ± 0.19	2.65 ± 0.02
R-(+)-citronellal	2.78 ± 0.73	1.17 ± 0.01	–	8.31 ± 0.07	1.98 ± 0.13	3.93 ± 0.03
lilac aldehyde c	trace	trace	12.30 ± 0.67	0.00	0.00	0.00
(*E*)-2-nonenal	0	0	0	0.00	1.54 ± 0.09	0.00
geranial	3.30 ± 0.07	3.80 ± 0.18	8.77 ± 0.20	34.82 ± 2.61	1.69 ± 0.11	3.45 ± 0.02
p-1,8-menthadien-7-al	0.80 ± 0.05	0.00	0.95 ± 0.05	0.11 ± 0.00	0.00	0.00
neral	3.83 ± 0.09	6.81 ± 0.29	8.08 ± 0.08	15.83 ± 0.30	3.89 ± 0.04	3.17 ± 0.01
2-butenal	2.85 ± 0.09	0.00	0.00	0.00	0.00	0.00

(*continued on next page*)

Table 2 (*continued*)

Compound	Concentration μg/g					
	Flower at different stages			Flower at different parts		
	stage-I	stage-II	stage-III	Petal	Stamen	pistil
(E)-2-pentenal	1.09 ± 0.02	0.78 ± 0.04	1.06 ± 0.07	0.09 ± 0.00	0.84 ± 0.06	0.66 ± 0.04
citronellal	3.51 ± 0.23	3.40 ± 0.06	6.81 ± 0.57	8.00 ± 0.47	3.75 ± 0.26	2.46 ± 0.10
(E,Z)-2,6-nonadienal	trace	trace	trace	trace	trace	trace
phenylacetaldehyde	trace	trace	trace	trace	trace	trace
myrtenal	7.81 ± 0.90	1.78 ± 0.13	3.85 ± 0.60	1.87 ± 0.38	0.00	1.83 ± 0.01
citral	4.24 ± 0.18	12.35 ± 0.90	25.20 ± 0.55	36.86 ± 1.45	5.86 ± 0.31	3.56 ± 0.00
tetradecanal	0.00	0.00	0.00	3.28 ± 0.01	2.18 ± 0.26	0.00
pentadecanal	0.00	0.84 ± 0.03	3.41 ± 0.01	9.42 ± 0.21	8.73 ± 0.33	1.05 ± 0.02
(e,e)-farnesal	1.58 ± 0.00	0.81 ± 0.01	1.67 ± 0.02	8.47 ± 0.23	1.95 ± 0.01	0.00
alcohols						
ethanol	0.19 ± 0.03	2.28 ± 0.08	3.39 ± 0.01	1.82 ± 0.12	3.00 ± 0.06	2.79 ± 0.11
1-pentanol	1.23 ± 0.04	0.43 ± 0.01	1.33 ± 0.01	0.00	2.78 ± 0.02	1.13 ± 0.07
(E)-2-hexen-1-ol	0.00	4.56 ± 0.14	1.71 ± 0.10	4.07 ± 0.39	5.51 ± 0.18	7.98 ± 0.32
1-hexanol	0.00	6.95 ± 0.19	4.43 ± 0.07	7.03 ± 0.38	12.24 ± 0.11	10.20 ± 0.02
trans-4-thujanol	4.74 ± 0.37	2.75 ± 0.01	9.86 ± 0.14	3.96 ± 0.004	0.00	0.00
linalool	59.44 ± 3.98	148.47 ± 8.09	281.35 ± 2.52	302.60 ± 5.33	194.61 ± 0.69	58.08 ± 3.46
cis-p-2,8-menthadien-1-ol	23.07 ± 0.65	0.00	0.00	0.00	0.00	0.00
trans-(−)-pinocarveol	6.94 ± 0.28	0.76 ± 0.08	10.15 ± 0.06	1.05 ± 0.04	0.00	4.77 ± 0.04
terpinen-4-ol	1.17 ± 0.24	1.06 ± 0.02	0.00	0.87 ± 0.08	0.00	0.00
myrtenol	15.84 ± 0.14	3.17 ± 0.17	9.13 ± 0.13	0.00	0.00	7.34 ± 0.19
carveol	4.58 ± 0.56	1.00 ± 0.00	0.00	0.00	0.00	0.00
trans-carveol	9.28 ± 0.71	1.54 ± 0.11	1.31 ± 0.04	1.50 ± 0.01	0.42 ± 0.02	2.79 ± 0.02
nerol	0.00	2.85 ± 0.11	21.45 ± 0.07	30.93 ± 1.10	5.44 ± 0.33	5.55 ± 0.28
cis-carveol	6.27 ± 0.15	1.87 ± 0.05	2.28 ± 0.02	1.50 ± 0.01	1.21 ± 0.03	2.71 ± 0.02
geraniol	0.00	2.08 ± 0.08	22.07 ± 2.29	20.29 ± 0.38	8.49 ± 0.10	4.66 ± 0.17
p-8-menthene-1,2-diol	0.00	0.00	0.62 ± 0.02	0.00	0.00	0.00
trans-p-2,8-menthadien-1-ol	12.03 ± 0.05	1.98 ± 0.08	3.40 ± 0.34	2.61 ± 0.14	1.47 ± 0.06	3.30 ± 0.10
nerolidol	0.00	3.62 ± 0.07	21.57 ± 1.18	72.99 ± 1.07	36.65 ± 2.12	2.37 ± 0.13
(−)-spathulenol	0.18 ± 0.02	0.00	0.00	0.00	0.00	0.00
1-penten-3-ol	1.25 ± 0.00	0.60 ± 0.03	1.25 ± 0.05	0.00	0.88 ± 0.01	0.96 ± 0.03
isocarveol	22.09 ± 1.30	4.17 ± 0.26	6.28 ± 0.18	4.36 ± 0.16	1.79 ± 0.03	5.42 ± 0.12
cis-verbenol	2.32 ± 0.09	0.84 ± 0.05	1.82 ± 0.16	0.00	0.00	1.24 ± 0.01
a-terpineol	6.57 ± 0.14	2.55 ± 0.28	3.72 ± 0.24	2.28 ± 0.07	1.48 ± 0.08	7.25 ± 0.01
citronellol	2.18 ± 0.03	13.29 ± 0.35	6.10 ± 0.18	6.90 ± 0.49	3.48 ± 0.11	1.27 ± 0.04
spathulenol	1.87 ± 0.02	1.19 ± 0.01	1.39 ± 0.05	0.00	0.00	1.08 ± 0.02
farnesol	1.58 ± 0.01	0.00	1.85 ± 0.00	0.00	2.64 ± 0.01	0.35 ± 0.001
(Z,E)-farnesol	1.14 ± 0.01	4.28 ± 0.02	12.06 ± 0.13	35.70 ± 0.04	10.55 ± 0.67	0.92 ± 0.00
esters						
ethyl acetate	0.20 ± 0.01	0.00	0.00	0.00	0.00	0.00
(Z)-3-hexenyl butanoate	1.21 ± 0.15	0.00	0.00	0.00	0.00	0.00
methyl geranate	0.00	0.00	1.62 ± 0.51	1.88 ± 0.01	1.57 ± 0.14	0.77 ± 0.02
methyl anthranilate	0.00	0.00	1.19 ± 0.10	4.99 ± 0.12	1.19 ± 0.01	0.00
neryl acetate	trace	0.00	0.00	0.00	0.00	2.65 ± 0.09
ethyl anthranilate	0.00	0.00	0.00	0.59 ± 0.00	0.00	0.00
ethyl 2-methylbutanoate	0.99 ± 0.05	trace	trace	0.00	0.00	0.00
ethyl hexanoate	2.94 ± 0.47	2.82 ± 0.16	3.78 ± 0.43	3.75 ± 0.00	1.65 ± 0.00	2.52 ± 0.00
cis-sabinene hydrate	2.66 ± 0.29	1.34 ± 0.02	4.09 ± 0.47	2.42 ± 0.16	0.00	2.86 ± 0.18
geranyl acetate	0.00	0.00	0.00	1.04 ± 0.06	0.00	2.63 ± 0.00
ketones						
1-penten-3-one	2.77 ± 0.01	2.59 ± 0.00	3.52 ± 0.09	0.00	0.28 ± 0.01	3.66 ± 0.03
methylheptenone	2.31 ± 0.02	1.09 ± 0.03	4.07 ± 0.24	3.18 ± 0.01	2.52 ± 0.07	1.19 ± 0.11
(E,E)-3,5-octadien-2-one	2.60 ± 0.06	0.00	0.00	0.63 ± 0.06	0.00	4.02 ± 0.02
pinocarvone	8.53 ± 0.85	1.03 ± 0.11	6.37 ± 0.04	2.94 ± 0.26	0.00	4.94 ± 0.01
trans-dihydrocarvone	4.12 ± 0.08	1.51 ± 0.08	2.40 ± 0.16	2.02 ± 0.03	1.01 ± 0.03	2.33 ± 0.11
carvone	20.00 ± 0.85	4.79 ± 0.14	9.96 ± 0.11	4.90 ± 0.01	2.78 ± 0.04	5.06 ± 0.04
4-acetyl-1-methylcyclohexene	2.19 ± 0.24	1.35 ± 0.20	4.67 ± 0.23	0.00	0.00	6.14 ± 0.09
geranylacetone	1.54 ± 0.05	2.41 ± 0.20	2.18 ± 0.11	4.17 ± 0.10	4.35 ± 0.08	1.25 ± 0.09
3,5-octadien-2-one	7.52 ± 0.19	3.08 ± 0.06	3.84 ± 0.74	0.00	0.00	2.81 ± 0.34
dihydrocarvone	2.07 ± 0.12	2.02 ± 0.00	1.36 ± 0.22	1.35 ± 0.11	0.00	0.84 ± 0.00
4-isopropyl-2-cyclohexen-1-one	2.68 ± 0.12	1.21 ± 0.06	1.58 ± 0.23	1.29 ± 0.02	0.00	1.03 ± 0.00
6-methyl-3,5-heptadien-2-one	3.83 ± 0.22	5.26 ± 0.33	11.92 ± 0.79	0.00	0.00	0.00
other compounds						
limonene 1,2-oxide	5.16 ± 0.62	2.34 ± 0.10	0.00	0.00	0.74 ± 0.05	2.76 ± 0.16
trans-limonene 1,2-oxide	0.00	14.81 ± 0.05	0.00	13.21 ± 0.07	0.00	0.00
indole	0.00	0.00	0.97 ± 0.02	18.48 ± 0.25	3.16 ± 0.06	0.00
caryophyllene oxide	12.87 ± 0.60	3.54 ± 0.40	3.84 ± 0.23	3.92 ± 0.54	trace	2.74 ± 0.02
p-xylene	1.45 ± 0.04	2.50 ± 0.01	1.26 ± 0.06	2.08 ± 0.07	1.49 ± 0.06	1.42 ± 0.06
o-xylene	0.37 ± 0.06	0.37 ± 0.01	0.61 ± 0.06	0.00	0.00	0.00
(+)-(E)-limonene oxide	19.24 ± 1.12	19.75 ± 0.77	271.99 ± 2.98	0.00	0.00	5.14 ± 0.04
humulene epoxide ii	1.52 ± 0.09	1.39 ± 0.01	1.25 ± 0.03	0.00	0.00	1.22 ± 0.00

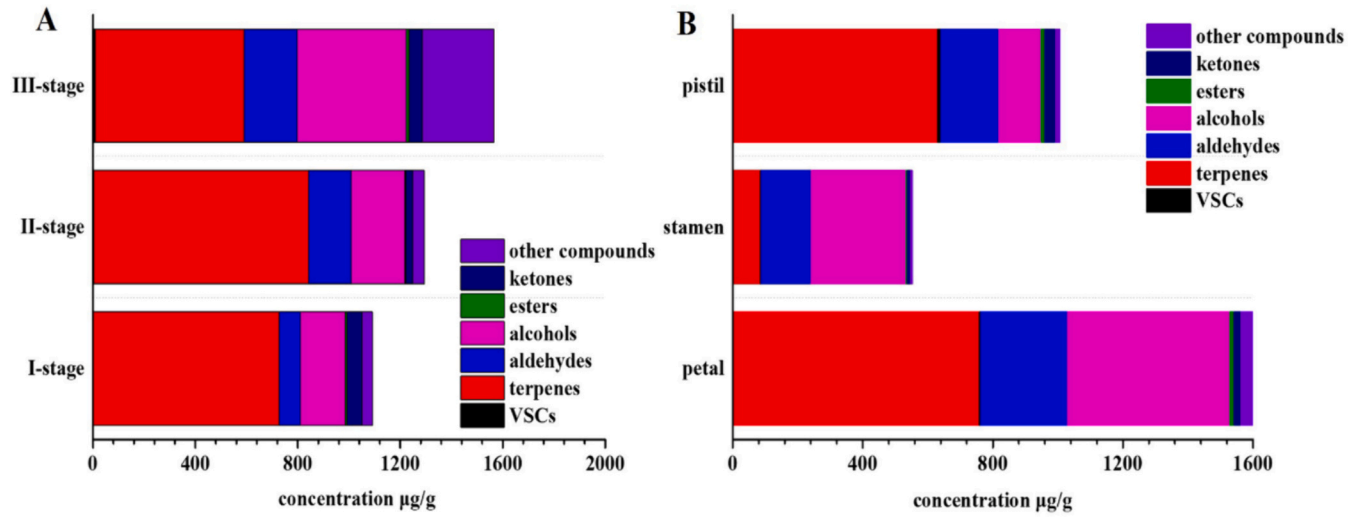

Fig. 2. Quantitative analysis of volatile compounds in Pomelo flowers at different developmental stages (A) and in the main organs of flowers (B).

Table 3
Odor intensity of aroma-active compounds in Pomelo flowers at different developmental stages and in the main organs.

compound	odor descriptor	odor intensity					
		stage-I	stage-II	stage-III	petal	stamen	pistil
Terpenes							
a-pinene	piney	3.05	3.1	2.3	2.6	2.1	2.9
camphene	camphor	2.2	4.9	4.2	4.4	2.1	4.2
β-myrcene	herbal	2.85	3.1	5.8	4.9	2.3	5.1
D-limonene	fatty, citrusy	7.1	6.8	5.5	5.8	3.4	5
p-cymenene	petrol	3.1	2.9	3.6	3.1	0	0
Aldehydes							
2-butenal	chemical	5.1	3.95	4.1	4.1	4.65	1.7
hexanal	grassy	3.25	4.3	4.35	5.95	4.3	6.0
(E)-2-hexenal	green	4	5.6	5.7	5.2	4.65	5.5
(E,E)-2,4-heptadienal	floral	3.05	2	1.9	0	0	2.75
benzaldehyde	bitter almond	2.5	1.55	2.6	2.5	1.9	1.7
phenylacetaldehyde	fatty, pungent	5.2	3.2	4.5	2.9	3.5	3.8
(E,Z)-2,6-nonadienal	fresh	4.25	4.05	3	3.4	3.35	4
neral	fruity	3.15	3.8	4	4.2	3.25	3
citral	lemony	0	3	4	4.4	0	0
Alcohols							
1-pentanol	medicine	0	0	0	0	2.5	0
linalool	floral	5.5	7	8.5	8.8	7.8	5.5
trans-p-2,8-menthadien-1-ol	herbal	5.3	3.7	4.7	4.1	3.7	4.7
citronellol	citrusy	0	2.4	0	0	0	0
Esters							
ethyl 2-methylbutanoate	fruity	3.85	3.5	1.5	0	3.3	0
ethyl hexanoate	overripe fruit	2.85	2.85	3.1	3.2	0	2.4
neryl acetate	floral, fruity	2.15	0	0	0	0	2.2
geranyl acetate	floral	0	0	0	2.1	0	0
Ketones							
6-methyl-3,5-hepten-2-one	fruity	3.1	2.05	3.5	3.3	3.2	1.5
carvone	herbal	3.95	0	2.8	0	0	0
Other compounds							
caryophyllene oxide	woody	3.1	2.3	2.3	2.4	1.85	2.8
unknown							
unknown1	minty	3.0	3.05	3.45	3.5	2.85	2.8
unknown2	fruity	1.9	0	1.9	2	2.45	2.6
unknown3	garlic	2.5	0	0	2.6	3.75	0
unknown4	herbal	5.3	3.7	4.7	4.1	3.7	4.7
Unknown5	fresh	2.25	3.1	2.5	0	2.6	2.9

an unpleasant aroma and is composed of p-cymenene, 2-butenal, benzaldehyde, phenylacetaldehyde, 1-pentanol and an unidentified volatile. This odor was the third most intense aroma for the flower petals, stamen parts and pomelo flowers at stage-I. The unpleasant aroma in the pomelo flower stamen part was stronger than that in the petal part.

3.5. OAV of the odorants

The OAVs of the odorants were calculated to evaluate the contribution of the detected odorants to the overall aroma of pomelo flowers at the developmental stages and different parts of pomelo flowers. As shown in Table 4, a total of 18, 16, 16, 16, 13, and 16 types of odorants with OAVs >1 were separately characterized in pomelo flowers at

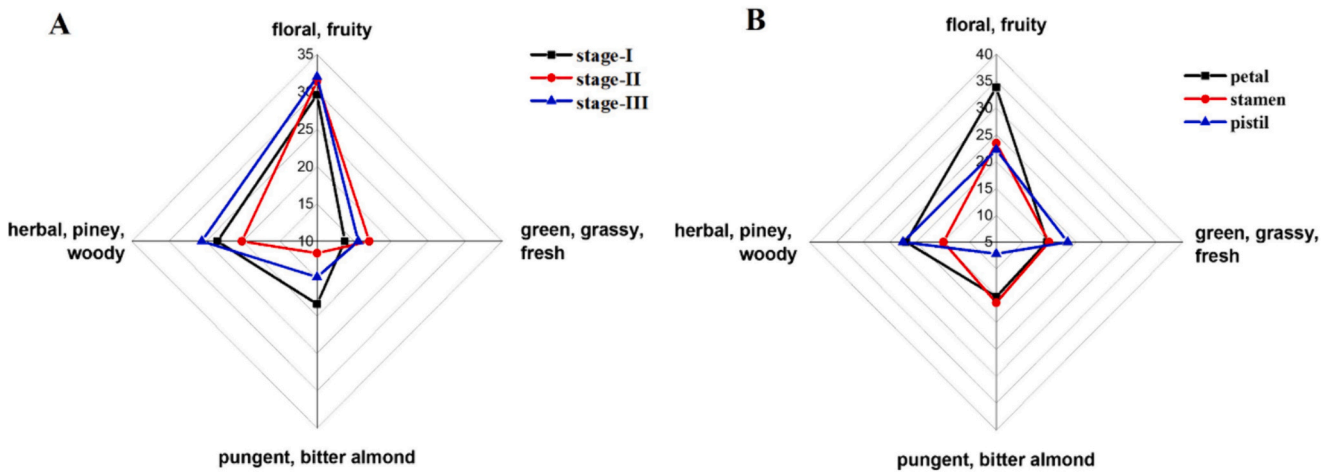

Fig. 3. Comparative descriptive odor profiles of Pomelo flowers at different developmental stages (A) and main organs of flowers (B).

Table 4
OAVs of odorants in Pomelo flower at different developmental stages and main organs.

compound	threshold(μg/L)	OAV					
		stage-I	stage-II	stage-II	petal	stamen	pistil
Terpenes							
a-pinene	9.5[1]	1312.97	2569.03	593.36	918.49	224.73	621.77
camphene	450[2]	<1	7.51	3.44	4.55	<1	3.20
β-myrcene	36[1]	511.62	672.32	6055.63	4621.03	358.52	5812.06
D-limonene	60[1]	7185.70	5429.03	1851.27	2520.21	225.80	1657.81
p-cymenene	85[3]	31.18	19.27	119.37	43.95	–	–
Aldehydes							
2-butenal	0.3[4]	9510.41	<1	<1	<1	<1	<1
hexanal	9.18[1]	1976.57	4501.49	5034.84	5867.37	4880.73	6366.30
(E)-2-hexenal	17[5]	1260.57	4852.59	4742.36	4593.46	3972.98	5074.11
(E,E)-2,4-heptadienal	15.4[4]	419.26	179.10	139.27	58.28	106.77	286.15
benzaldehyde	350[5]	10.22	6.35	10.93	9.83	8.32	7.06
neral	100[6]	38.27	68.14	80.80	158.26	38.88	31.65
citral	85.3[1]	49.66	144.83	295.41	432.17	68.68	41.74
Alcohols							
1-pentanol	4000[5]	<1	<1	<1	–	<1	<1
linalool	5.3[1]	11,215.08	28,014.14	53,084.81	57,095.25	36,718.97	10,958.91
citronellol	62[7]	35.09	214.40	98.41	111.29	56.07	20.48
Esters							
ethyl 2-methylbutanoate	0.013[8]	75,820.87	<1	<1	–	<1	–
ethyl hexanoate	1[9]	2938.79	2820.60	3783.24	3752.38	1651.05	2516.86
neryl acetate	42[10]	<1	–	–	–	–	63.12
geranyl acetate	9[5]	–	–	–	115.02	–	292.37
Ketones							
6-methyl-3,5-heptadien-2-one	380[5]	10.09	13.84	31.38	–	–	–
carvone	160[11]	125.00	29.94	62.22	30.64	17.39	31.64
Other compounds							
caryophyllene oxide	5500[7]	2.34	<1	<1	<1	0.00	<1

Reference:1 = (Ahmed et al., 1978), 2 = (Rigling et al., 2019), 3 = (Masanetz & Grosch, 1998), 4 = (Giri et al., 2010), 5 = (Buttery et al., 1988) , 6 = (Tamura, Fukuda and Padrayuttawat, 1996), 7 = (Tamura et al., 1993), 8 = (Czerny et al., 2008), 9 = (Pino & Mesa, 2006), 10 = (Pino et al., 1986), 11 = (Averbeck & Schieberle, 2011)

different developmental stages (stage-I, stage-II, stage-III), and three parts (petal, stamen, pistil) of pomelo flowers. The largest OAV (75821) was observed for ethyl 2-methylbutanoate, followed by linalool (11215), 2-butenal (9510), D-limonene (7186), ethyl hexanoate (2939), hexanal (1977), a-pinene (1313), and (E)-2-hexenal (1261) in pomelo flowers at stage-I. Six important odorants, including linalool (28014), D-limonene (5429), (E)-2-hexenal (4853), hexanal (4501), ethyl hexanoate (2821), and a-pinene (2569), exhibited OAVs >1000 in pomelo flowers at stage-II. In pomelo flower stage-III, the OAVs (>1000) of linalool (53085), β-myrcene (6056), hexanal (5035), (E)-2-hexenal (4742), ethyl hexanoate (3783) and D-limonene (1851) were greater than those of the other odorants detected. During the growth of citrus flowers, linalool, which had a floral odor, gradually becomes an important contributor to the overall profile of pomelo flowers, which was mainly related to promotion of linalool synthesis by terpene synthases (TPS) (Chen et al., 2011). The scent of linalool released by flowers is also an important odor signal that attracts bees to pollinate (Burdon et al., 2019). Fruity odor was the predominant odor in pomelo flowers at stage-I. Pomelo flowers at stage-II and stage-III presented the strongest floral odor. Fruity/floral were important odors for pomelo flowers, which coincided with the results obtained for the MDGC-MS/O aroma profile. In the petal and pistil parts of pomelo flowers, there were six odorants with OAVs>1000, including linalool, hexanal, β-myrcene, D-limonene, (E)-2-hexenal, and ethyl hexanoate. These odorants mainly included floral/fruity, herbal/camphor, and green/grassy notes. Four aroma active compounds with OAVs >1000 were identified in pomelo flower stamens. The two odor-active compounds with the highest OAVs in the three parts of pomelo flowers were linalool and hexanal.

3.6. Multivariate statistical analysis

To explore the distinguishing markers for the aroma profiles of pomelo flowers at different developmental stages and different parts of pomelo flowers, principal component analysis (PCA) and orthogonal partial least squares discriminant analysis (OPLS-DA) were carried out based on the OAVs of the obtained odorants. As shown in Fig. S2, the first and second principal components represented 62.1% and 34.4% of the total variance in pomelo flowers at different developmental stages, respectively. For the different organs of pomelo flowers, 95.4% of the variance was attributed to the two components. The pomelo flowers at stage-I, stage-II, and stage-III were completely separated according to first principal component (Fig. S2A), and the flower stamen, pistil, and petal samples were completely separated according to the first component (Fig. S2B). The results, revealed significant differences in the odorants of pomelo flowers at different developmental stages and in different parts. The credibility of all samples was within the 95% Hotelling T2 confidence level, and no significant extreme data or outliers were observed.

OPLS-DA, a supervised analytical method, was subsequently used to determine on the OAVs of the obtained odorants observed in pomelo flowers at the developmental stages and in different parts of pomelo

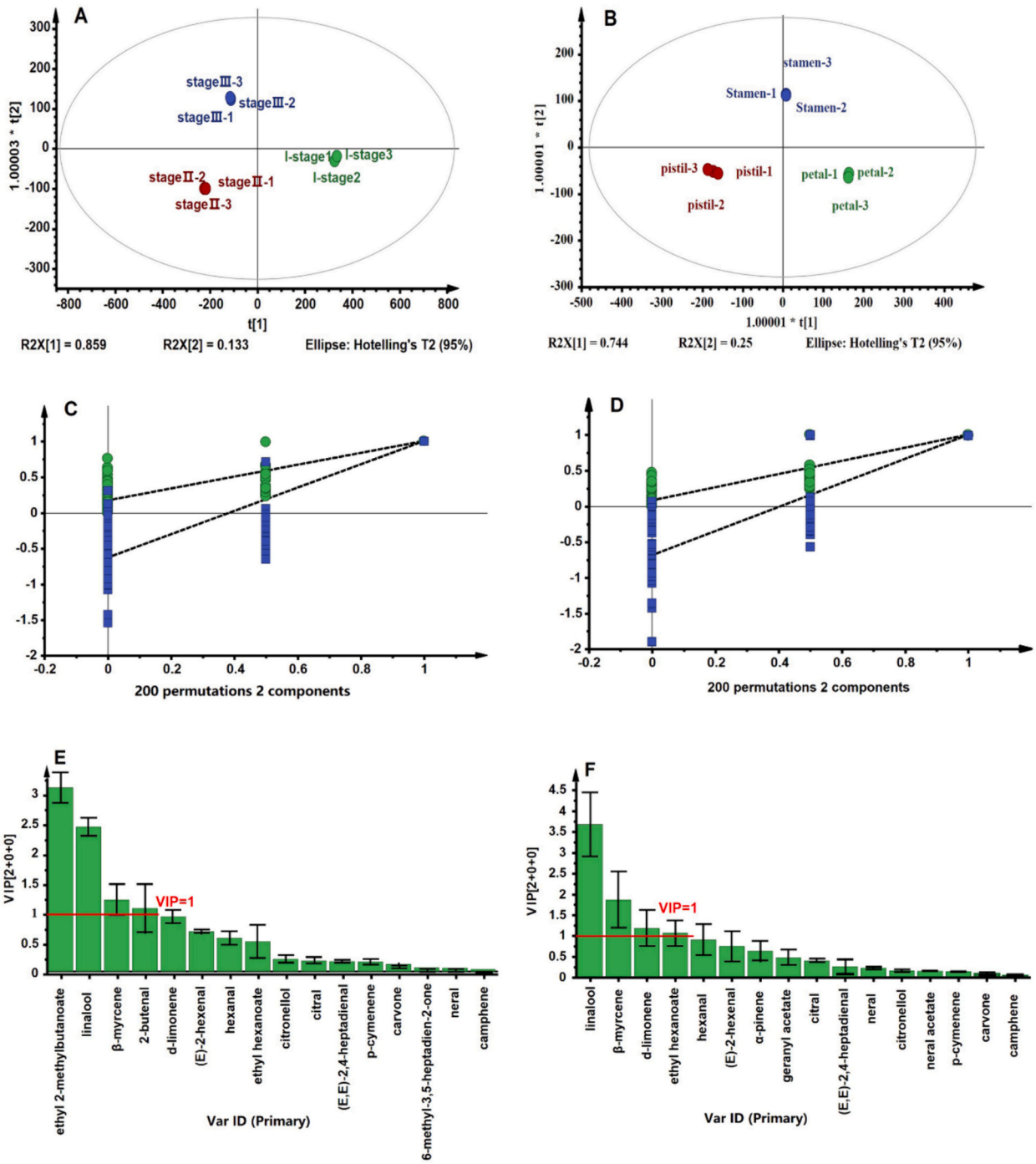

Fig. 4. The OPLS-DA plots of pomelo flowers at different developmental stages (A) and in the main organs (B), permutation plots of pomelo flowers at different developmental stages (C) and in the main organs of flowers (D).
Colors should be used both online and in print.

flowers. As shown in Fig. 4A, the score plot revealed that pomelo flowers at different developmental stages were divided into three camps (green, red and blue parts). An obvious separation was found on the first principal component axis between late camps (stage-II and stage-III) and the early camp (stage-I), indicating that the early and late camps exhibited major differences. Moreover, the pomelo flower stamen, petal and pistil completely separated on the first principal component, indicating that there were significant differences in these variables among the three camps (Fig. 4B). The overfitting of data was also investigated via permutation tests (Xue et al., 2022; Ye et al., 2022). Herein, R2(y) and Q2 values were 0.171 and − 0.633 (Fig. 4C), and 0.0766 and − 0.671 (Fig. 4D), respectively. The results showed that the OPLS-DA model was reliable, predictable and free of overfitting. In addition, the contribution of variables in projection can be weighted by variable importance in projection (VIP) values. Generally, when the VIP was >1, the variables with a VIP >1 were considered marker candidates and contributed to the pomelo flower samples at different stages or in different organs of pomelo flowers (Mais et al., 2018). Based on these criteria, four variables (ethyl 2-methylbutanoate, linalool, β-myrcene, and 2-butenal) and four variables (linalool, β-myrcene, D-limonene, and ethyl hexanoate) were screened as markers in the pomelo flower samples at different stages and different organs of pomelo flowers, respectively.

4. Conclusion

The flavor profiles of pomelo flowers at three developmental stages and three different organs of pomelo flowers were revealed via molecular sensory science approaches and multivariate statistical analysis. A total of 134 volatiles were analyzed qualitatively and quantitatively. The total volatile content increased gradually with the growth of pomelo flowers. The content of volatile compounds was distributed differently in the three main parts of the pomelo flower, among which petal > pistil > stamen. Except for pomelo flower stamens, terpenes were the most predominant volatile class, accounting for 36.98% ~ 65.69% of the total content in pomelo flowers at different developmental stages and in different organs. Twenty-five odorants were identified in pomelo flowers at different developmental stages and in different organs of pomelo flowers. Aroma profiles, including floral/fruity, green/grassy/fresh, pungent/bitter almond, and herbal/piney/woody, were described based on the relative intensities of odorants. Owing to the contributions of linalool and other odorants, floral/fruity was the strongest sensory characteristic for whole pomelo flowers and pomelo flower organs. OPLS-DA revealed four volatiles (ethyl 2-methylbutanoate, linalool, β-myrcene, and 2-butenal) and four variables (linalool, β-myrcene, D-limonene, and ethyl hexanoate) as potential markers for evaluating the differences in flavor of pomelo flowers at three different developmental stages and in different organs.

CRediT authorship contribution statement

Yujiao Cheng: Writing – review & editing, Writing – original draft, Methodology, Formal analysis, Data curation, Conceptualization. **Leng Han:** Writing – review & editing, Investigation, Data curation. **Linzi Shao:** Methodology, Formal analysis. **Hua Wang:** Supervision, Conceptualization. **Zheng Guo:** Writing – review & editing, Software, Methodology, Formal analysis, Data curation. **Guijie Li:** Writing – review & editing, Visualization, Validation.

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.2024.101568.

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