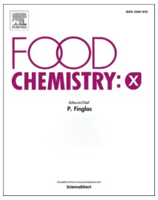
Food Chemistry: X 22 (2024) 101395

Contents lists available at ScienceDirect

Food Chemistry: X

journal homepage: www.sciencedirect.com/journal/food-chemistry-x



Identification of key sensory and chemical factors determining flavor quality of Xinyu mandarin during ripening and storage

Xin-Cheng Liu[a,1], Yu-Qing Tang[a,1], Yin-Chun Li[b], Shao-Jia Li[b], Hui-Dong Yang[a], Shui-Lin Wan[a], Yu-Ting Wang[a,c,d,*], Zhong-Dong Hu[a,c,d,*]

[a] Institute of Horticulture, Jiangxi Academy of Agricultural Sciences, Nanchang 330200, China
[b] College of Agriculture and Biotechnology, Zhejiang University, Hangzhou 310058, China
[c] Jiangxi Key Laboratory of Horticultural Crops (Fruit, Vegetable & Tea) Breeding, Nanchang 330200, China
[d] Nanchang Key Laboratory of Germplasm Innovation and Utilization of Fruit and Tea, Nanchang 330200, China

ARTICLE INFO

Keywords:
Citrus
Postharvest storage
Sensory evaluation
Flavor
Volatile organic compound

ABSTRACT

Xinyu mandarin is popular for its good flavor, but its flavor deteriorates during postharvest storage. To better understand the underlying basis of this change, the dynamics of the sensory profiles were investigated throughout fruit ripening and storage. Sweetness and sourness, determined especially by sucrose and citric acid content, were identified as the key sensory factors in flavor establishment during ripening, but not in flavor deterioration during storage. Postharvest flavor deterioration is mainly attributed to the reduction of retronasal aroma and the development of off-flavor. Furthermore, sugars, acids and volatile compounds were analyzed. Among the 101 detected volatile compounds, 10 changed significantly during the ripening process. The concentrations of 15 volatile components decreased during late postharvest storage, among which α-pinene and D-limonene were likely to play key roles in the reduction of aroma. Three volatile compounds were found to increase during storage, associated with off-flavor development.

1. Introduction

Xinyu mandarin (*Citrus reticulata* cv. Xinyu) is a citrus cultivar selected as a mutation of Bendizao mandarin (*Citrus reticulata* cv. Succosa), widely cultivated in Xinyu area of Jiangxi Province, China. This mandarin has pleasant sweetness and a typical mandarin-like flavor inherited from Bendizao, making it competitive in the market. However, flavor deterioration during postharvest storage is one of the major obstacles to the development of Xinyu mandarin industry.

Fruit loss and quality deterioration are two main limitations to the extension of citrus shelf life. Fruit loss can be due to various factors, such as fungal infection and mechanical damage, usually leading to direct economic losses. However, internal quality deteriorations in juice sacs, such as section drying (Cao et al., 2020; Kang, Cao, Wang, & Sun, 2022) and flavor deterioration (López-Gómez, Ros-Chumillas, Buendía-Moreno, Navarro-Segura, & Martínez-Hernández, 2020; Owoyemi et al., 2022), are often difficult to detect before sale because the fruit peel seems normal, and thus may result in damage to the reputation among consumers.

Up to now, flavors of fresh mandarins have been extensively studied (Goldenberg, Yaniv, Porat, & Carmi, 2018; Tietel, Plotto, Fallik, Lewinsohn, & Porat, 2011). By the comparison of 42 mandarin varieties, the most liked flavors are characterized by attributes of high sweetness, moderate to low acidity, low bitterness and gumminess, strong fruity and mandarin flavor, and high juiciness (Goldenberg et al., 2015). The sweetness and sourness of mandarins are mainly determined by sugars and organic acids, respectively. In most mandarin varieties, sucrose, glucose and fructose constitute the vast majority of sugar content, while citric acid is the dominant organic acid (Goldenberg et al., 2018). In comparison, volatile organic compounds, which determine aroma, mandarin-specific flavor or variety-specific flavor, can vary significantly among species or varieties. In previous studies, the volatile composition of mandarins were widely compared among varieties (Goldenberg, Yaniv, Doron-Faigenboim, Carmi, & Porat, 2016; Miyazaki, Plotto, Baldwin, Reyes-De-Corcuera, & Gmitter Jr, 2012; Xiao et al., 2017) or with other citrus species (Feng, Suh, Gmitter, & Wang, 2018; Ren et al.,

---

* Corresponding authors at: Institute of Horticulture, Jiangxi Academy of Agricultural Sciences, Nanchang 330200, China.

E-mail addresses: liuxch25@jxaas.cn (X.-C. Liu), YuQingT1020@163.com (Y.-Q. Tang), 22116151@zju.edu.cn (Y.-C. Li), shaojiali@zju.edu.cn (S.-J. Li), yanghd@jxaas.cn (H.-D. Yang), wanshuilin@163.com (S.-L. Wan), wyuting1220@163.com (Y.-T. Wang), hzd204028@163.com (Z.-D. Hu).

[1] These authors contributed equally to this work.

https://doi.org/10.1016/j.fochx.2024.101395
Received 2 January 2024; Received in revised form 15 April 2024; Accepted 16 April 2024

2015; Zhang et al., 2017). The key compounds in mandarin-like aroma have been identified. Based on the analysis of the juice of LB8–9 mandarin with volatile compound quantification, aroma extract dilution analysis (AEDA), odor activity value (OAV), aroma recombination and omission tests, α-pinene, limonene, octanal, linalool and (E,E)-2,4-decadienal were identified as the most important components of mandarin-like aroma. (Feng et al., 2018). In a comparative study of the juices of five mandarin varieties including Tankan, Miyagawa-wase, Mashui, Skiranui, and Ponkan, 36 odor-active compounds were identified using gas chromatography-olfactometry (GC-O) and OVA. The aroma of Ponkan mandarin juice was further successfully simulated by an aroma recombination model consisting of 22 odor-active compounds, among which nonanal, hexanal, linalool, (R)-(+)-limonene, β-ionone, decanal, γ-terpinene, and methyl butanoate were determined as the key odorants by omission tests (Xiao et al., 2017). A more inclusive study involving 20 mandarin varieties found 14 common odor-active compounds with OAV > 1. The aroma recombination could roughly simulate the aroma of Nanfeng tangerine, and the omission tests indicated that ᴅ-limonene, linalool, nonanal, hexanal, α-pinene and γ-terpinene were the key aroma compounds (Zhou et al., 2021). However, it should be noted that the above studies on the key aroma compounds in mandarin-like aroma were all based on smelling tests rather than tasting. Considering that the orthonasal and retronasal olfactory pathways can elicit different perceptions, these compounds may not be equally crucial to the retronasal aroma (mandarin-like flavor) than the smelling-based orthonasal aroma (Hannum, Fryer, & Simons, 2021). Despite this, these previous researches are still valuable for further studies on the flavor of mandarins or other citrus species.

Deterioration of citrus flavor during postharvest storage can involve a series of sensory factors. The key factors may vary among species or varieties. The study on ‘Mor’ mandarin showed that the decreased taste score during storage was attributed to the decreases in sourness and mandarin-like flavor and the accumulation of off-flavor (Tietel et al., 2010). In the 6-week storage of ‘Orri’ mandarin, decreases in sourness, bitterness and aroma, and an increase in off-flavor was observed (Otieno et al., 2022). While the sensory analysis on four different orange varieties showed that most flavor attributes did not significantly changed during fruit storage, except that the off-flavor increased in the four varieties and the bitterness increased in ‘Washington navel’ and ‘Moro’ oranges (Fabroni, Amenta, Timpanaro, Todaro, & Rapisarda, 2020). However, in terms of the chemistry underlying citrus postharvest flavor deterioration, only sugars and acids have been observed during this process, while the changes in volatile composition have not been well studied, and the key aroma compounds contributing to flavor deterioration have not been identified (Tietel et al., 2010).

To gain a detailed understanding of the postharvest flavor deterioration of Xinyu mandarin, a weekly sensory evaluation was conducted to present the changes in flavor-related attributes throughout its shelf life, so that the key sensory factors determining overall flavor quality could be identified. Total soluble solids (TSS) and acidity in the juice were also observed weekly. Furthermore, flavor-related chemicals, including sugars, acids and volatile compounds, were investigated at the selected crucial stages when sensory attributes changed greatly to identify the key chemicals contributing to the changes in sensory qualities.

2. Material and methods

2.1. Plant material, harvest and storage

Approximately 600 Xinyu mandarin trees were cultivated in a 10,000 m^2 field in an orchard of the Institute of Horticulture, Jiangxi Academy of Agricultural Sciences in Nanchang, China. From October 18th 2022, 60 on-tree fruits were randomly harvested each week from the entire orchard for quality analysis. On October 25th and November 8th, respectively, one thousand fruits were randomly harvested for postharvest storage.

The harvested fruits were naturally cooled to ambient temperature and stored in commercial cardboard boxes with air holes, placed in a ventilated storehouse near the orchard, so that the storage environment changed along with the ambient environment. The environmental temperature, humidity and precipitation were recorded every 30 min by a weather station near the orchard (Supplementary Fig. 1). In this paper, “X#Y” is used for denoting the fruits harvested at X weeks after blossom (WAB) and stored for Y week(s) (Fig. 1A).

2.2. Sensory evaluation

All procedures involving human participants were in accordance with the ethical standards of the academic ethics committee of Jiangxi Academy of Agricultural Sciences, and informed consent was obtained from the participants.

Evaluators were randomly recruited from the local community. Their sex and age compositions are shown in Supplementary Fig. 2. The evaluation was based on a questionnaire-directed interview and followed the “dual-random” principle (a random evaluator assessed a random fruit) to simulate a real consumption situation. Every week, at least 20 fruits for each harvest time were used for sensory evaluation. At 36 WAB, the sensory evaluation was canceled due to the COVID-19 epidemic as the virus significantly affected human senses.

The evaluators were served with an intact fruit, peeled and tasted as an ordinary consumer. For each harvest-storage group, one or two segments of each of 15 fruits were remained and labeled for quality analysis. The questionnaire consisted of two parts: the first part collected basic personal information (gender and age) of the evaluator, and the second was about the emotional reaction of the evaluator on the assessed fruit. The evaluation involved five aspects of fruit flavor, and the evaluators were asked to choose the most appropriate description based on their subjective feelings:

- Overall flavor (Delicious | Acceptable | Awful);
- Sweetness (Too sweet and uncomfortable | Appropriate | Perceptible but insufficient | Almost imperceptible);
- Sourness (Too sour to tolerate | Too sour but tolerable | Appropriate | Almost imperceptible);
- Mandarin-like flavor, or retronasal aroma (Aromatic | Modest | Flavorless);
- Off-flavor (Perceptible | Imperceptible).

2.3. Total soluble solids and acidity

In each group, 15 fruits used for sensory evaluation were partly remained for the measurement of TSS and acidity. The measurement was conducted with a hybrid refractometer (PAL-BX/ACID 1, ATAGO). The device measures TSS through refractometry method using light refraction, and measures acidity through electroconductivity method using electrical current.

2.4. Soluble sugars and organic acids

Following previously described method (Liu et al., 2021), fruit juice sacs were carefully separated from fruits, ground to powder in liquid nitrogen and stored at −80 °C. For extraction of sugars and acids, 0.1 g powder was added to 1.4 mL pre-chilled methanol and vibrated at 70 °C for 15 min. After centrifugation at 4 °C 11,000 g for 10 min, the supernatant was collected. For derivatization, 100 μL supernatant was added to 20 μL 0.2 mg/mL ribitol (internal standard) and dried in a vacuum. The residue was then dissolved in 60 μL 20 mg/mL pyridine methoxyamine hydrochloride, and vibrated at 37 °C for 1.5 h, added with 40 μL BSTFA +1% TMCS, and vibrated at 37 °C for 0.5 h.

For gas chromatography (GC) analysis, 1 μL of the derivatized sample was injected into a gas chromatograph (7890B, Agilent) equipped with an HP-5 column. The injector temperature was set to 250 °C, and

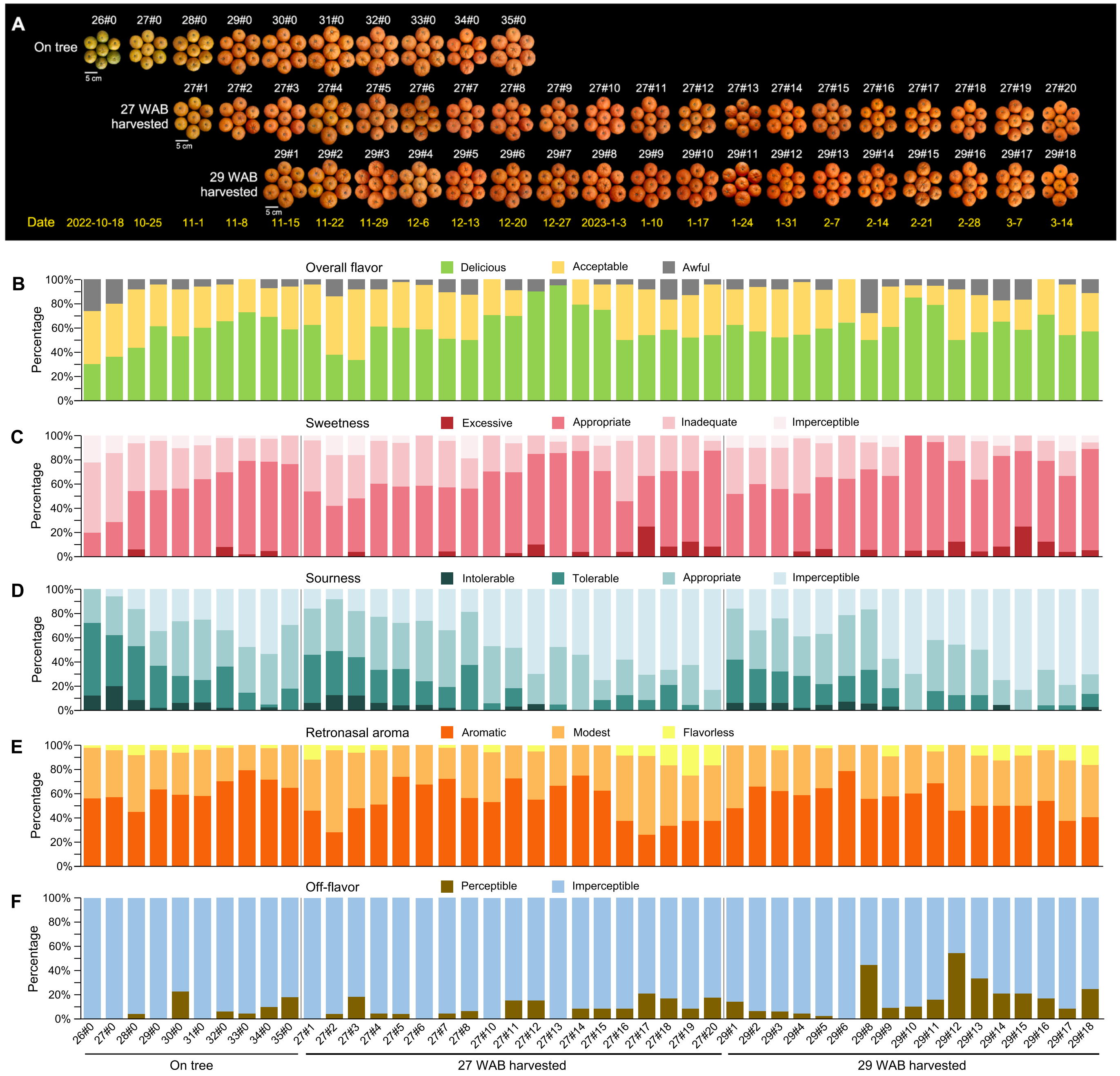


Fig. 1. Sensory evaluation of Xinyu mandarin during ripening and postharvest storage. (A) The harvest-storage strategies and their sampling dates. (B—F) Evaluators' comments on overall flavor, sweetness, sourness, retronasal aroma and off-flavor. "X#Y" denotes the fruits harvested at X weeks after blossom and stored for Y week(s).

the split ratio was 10:1. The oven temperature was held at 100 °C for 1 min, increased to 185 °C at 2.5 °C/min, to 190 °C at 0.35 °C/min, and to 250 °C at 8 °C/min, and then held for 5 min, increased to 280 °C at 5 °C/min, and held at 280 °C for 3 min. The peaks of sucrose, glucose, fructose, citric acid and malic acid were identified by comparing retention time of authentic standards, and quantification was carried out with standard curves after calibration with the peak of the internal standard.

2.5. Volatile compounds

Volatile compounds in the juice sacs were determined based on the widely targeted volatilomics method (Yuan et al., 2022). Five hundred milligrams of the powder prepared in liquid nitrogen was transferred to a 20 mL head-space vial, containing 2 mL of NaCl saturated solution and 10 μL of 50 μg/mL 3-hexanone (internal standard). The vials were sealed using crimp-top caps with TFE-silicone headspace septa. For headspace-solid phase microextraction (HS-SPME), each vial was placed at 60 °C for 5 min, then a 120 μm DVB/CWR/PDMS fiber was exposed to the headspace of the sample for 15 min at 60 °C.

Based on previously described method (Yue et al., 2023) with slight modification, desorption of the volatile compounds from the fiber coating was carried out in the injection port of the GC apparatus (Model 7890B; Agilent) at 250 °C for 5 min in the splitless mode. The identification and quantification of volatiles was carried out using 7890B–7000D GC–MS (Agilent), equipped with a 30 m × 0.25 mm × 0.25 μm DB-5MS (5% phenyl-polymethylsiloxane) capillary column. Helium was used as the carrier gas at a linear velocity of 1.2 mL/min. The injector temperature was kept at 250 °C and the detector at 280 °C.

The oven temperature was programmed from 40 °C (3.5 min), increasing at 10 °C/min to 100 °C, at 7 °C/min to 180 °C, at 25 °C/min to 280 °C, and held for 5 min. Mass spectra were recorded in electron impact (EI) ionization mode at 70 eV. The quadrupole mass detector, ion source and transfer line temperatures were set, respectively, at 150, 230 and 280 °C. The selected ion monitoring (SIM) mode was used for the identification and quantification of compounds. Volatile compounds were identified by comparing the linear retention index and selected qualitative ions with the data in the MWGC library (Metware Biotechnology Co., Ltd., Wuhan, China). Selected quantitative ion peaks were used for quantification. The concentration of each volatile compound was quantified by dividing the peak area of each compound by the peak area of the internal standard.

2.6. *Statistical analysis*

One-way analysis of variance (ANOVA) on sugars, acids and volatile compounds was performed using SPSS software. Differences between groups were analyzed by Tukey's HSD test with a 95% confidence level.

The volatile compound data were scaled with the unit variance method. Cluster analysis and principal component analysis (PCA) were performed on the scaled data using the R packages "prcomp" and "ComplexHeatmap".

For correlation analysis, a numerical index was calculated for each sensory attribute as the sum of the percentages of comments weighted by continuous integers: overall flavor index = 2 × delicious% + 1 × acceptable% + 0 × awful%; sweetness index = 3 × excessive% + 2 × appropriate% + 1 × inadequate% + 0 × imperceptible%; sourness index = 3 × intolerable% + 2 × tolerable% + 1 × appropriate% + 0 × imperceptible%; aroma index = 2 × aromatic% + 1 × modest% + 0 × flavorless%; off-flavor index = 1 × perceptible% + 0 × imperceptible%. Pearson correlation coefficients were calculated using Microsoft Excel.

3. Results

3.1. *Overall flavor and sensory attributes during ripening and storage*

For on-tree fruits, consumer acceptability and satisfaction with overall flavor both steadily increased until 33 WAB (December 6th), and then worsened. During postharvest storage of both 27- and 29-WAB-harvested fruits, the best overall flavor appeared at 39–40 WAB, when the satisfaction rate reached 90%–95%. Subsequently, an obvious flavor deterioration was observed, mainly characterized by a decrease in consumer satisfaction rather than acceptance (Fig. 1B).

Fruit sweetness showed a similar changing pattern with overall flavor during on-tree ripening and early storage stage. However, during late postharvest storage, sweetness did not decrease along with overall flavor (Fig. 1C). Sourness of both on-tree and postharvest fruits showed a decreasing trend over the entire period. The sourness of fruits on tree decreased faster than the postharvest. In the late stage of storage, sourness continually decreased to almost "imperceptible" (Fig. 1D). In comparison, changes in aroma were mostly associated with the pattern of overall flavor, as fruits of higher maturity were more aromatic during ripening, while fruits in the middle postharvest stage were more aromatic than those in the early and late postharvest stages. In the late postharvest stage, more fruits were even commented as "flavorless" (Fig. 1E). As expected, off-flavor gradually occurred during both on-tree and postharvest processes (Fig. 1F).

3.2. *Sensory contributors to the changes in overall flavor acceptance and satisfaction*

To identify the key sensory factors determining consumer experience, a correlation analysis was conducted (Fig. 2). However, when all the 46 harvest-storage groups were analyzed, no specific factor was found to be closely correlated with overall flavor. Sweetness, sourness, aroma and acidity all showed modest correlation ($0.41 < |r| < 0.59$) with overall flavor (Fig. 2A). We then realized that some factors may play roles in determining flavor quality at specific stages rather than throughout the whole period. Because when the evaluators gave negative comments on the fruit, their reasons were quite differently in the early and late stages of our investigation. Therefore, we divided the entire period into two overlapping stages, where the flavor establishment stage contained 26–33#0, 27#1–14 and 29#1–11, whose overall flavor was improving or maintaining at high level, and the flavor deterioration stage contained 33–35#0, 27#12–20 and 29#10–18, whose overall flavor was maintaining or deteriorating. The analysis showed that in the flavor establishment stage, overall flavor was closely correlated with sweetness positively ($r = 0.80$) and sourness negatively ($r = -0.81$) (Fig. 2B). While in the flavor deterioration stage, the correlation with aroma ($r = 0.75$) and off-flavor ($r = -0.54$) was much higher than that with other factors (Fig. 2C).

3.3. *Sugars and acids*

TSS, a characteristic often used as an indicator of sugar content, was monitored throughout the investigation period (Fig. 3A). The results showed that the change in TSS was mainly associated with fruit ripening but not postharvest process. The changes were greater in the early stages than in the late stages. During ripening, the average TSS increased from 12.7% at 26 WAB to 16.6% at 35 WAB. During storage, the average TSS of the 27-WAB-harvested groups ranged from 14.4% to 15.6%, and that of the 29-WAB-harvested groups ranged from 15.0% to 16.2%, without much changes.

Soluble sugars (fructose, glucose and sucrose) in 12 selected groups of different sweetness levels (Fig. 1C) were determined by GC (Table 1).

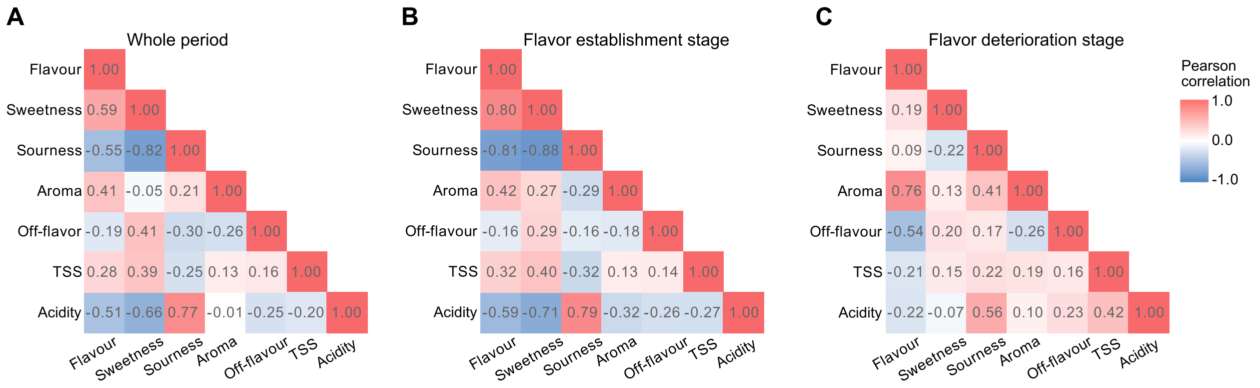


Fig. 2. Correlation analysis among overall flavor, sensory attributes, TSS and acidity. (A) Analysis on all the 46 harvest-storage groups during ripening and storage, (B) analysis on the 31 groups during flavor establishment stage, and (C) analysis on the 21 groups during flavor deterioration stage.

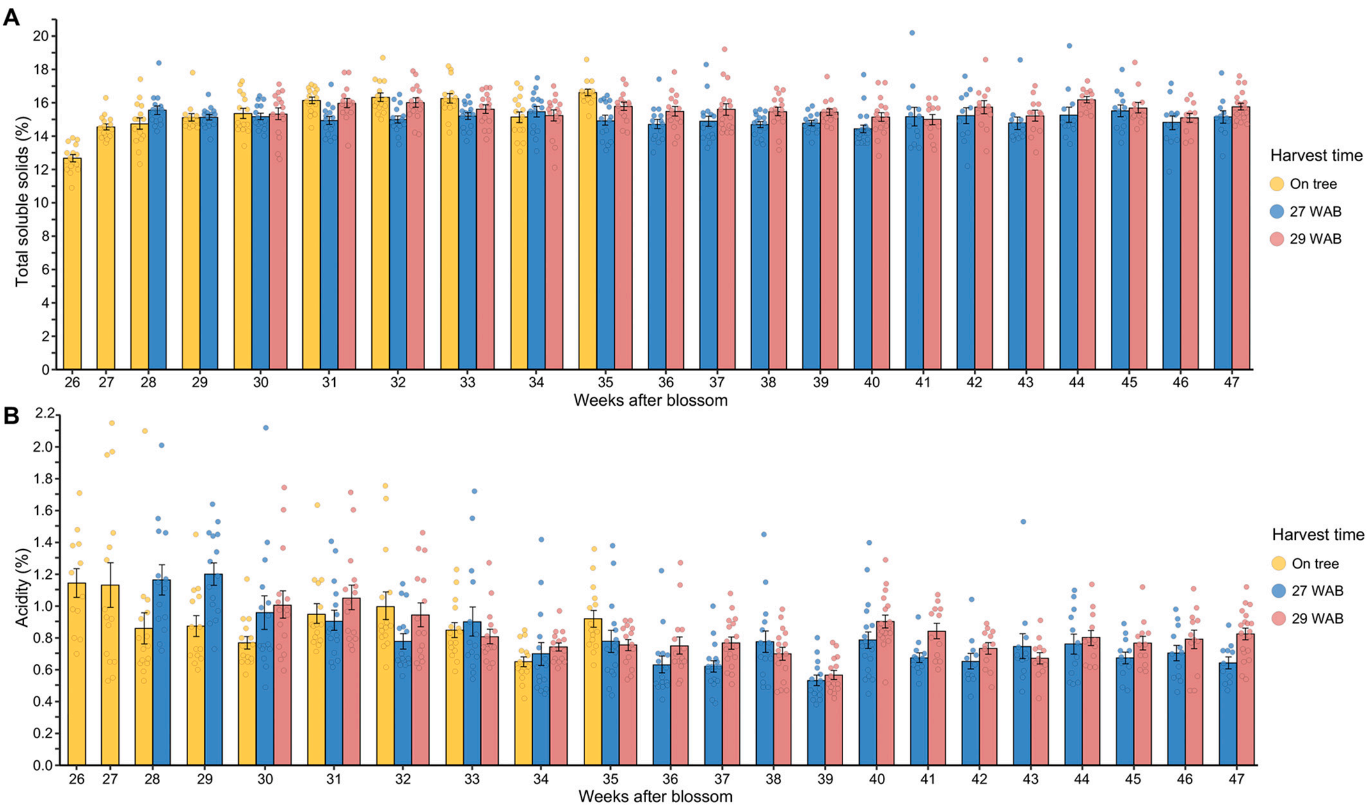


Fig. 3. TSS (A) and acidity (B) of Xinyu mandarin during ripening and postharvest storage. Error bars show means ± standard error.

Table 1
Sugars and acids content of Xinyu mandarin of different harvest and storage time (mg/g FW).

Harvest time	Group	Glucose	Fructose	Sucrose	Citric acid	Malic acid
On-tree	27#0	22.13 ± 0.22^{b}	17.19 ± 0.18^{c}	104.89 ± 4.61^{a}	18.52 ± 0.37^{b}	1.28 ± 0.14^{a}
	28#0	19.92 ± 0.07^{a}	14.74 ± 0.05^{ab}	144.76 ± 4.64^{b}	10.48 ± 1.67^{a}	1.57 ± 0.17^{a}
	29#0	18.85 ± 0.66^{a}	13.44 ± 0.78^{a}	140.93 ± 4.36^{b}	11.48 ± 1.27^{a}	1.62 ± 0.15^{a}
	33#0	20.17 ± 0.46^{a}	15.59 ± 0.52^{b}	162.85 ± 5.18^{c}	10.66 ± 1.02^{a}	1.52 ± 0.10^{a}
27 WAB	27#2	22.43 ± 0.71^{a}	18.20 ± 0.84^{a}	124.00 ± 4.46^{a}	15.64 ± 1.03^{c}	1.07 ± 0.09^{b}
	27#5	21.22 ± 0.64^{a}	16.89 ± 0.69^{a}	133.62 ± 7.11^{a}	11.65 ± 1.02^{b}	1.03 ± 0.13^{ab}
	27#14	20.92 ± 0.24^{a}	17.33 ± 0.40^{a}	123.19 ± 5.83^{a}	7.87 ± 0.44^{a}	0.72 ± 0.09^{a}
	27#19	20.55 ± 1.07^{a}	17.70 ± 1.40^{a}	124.34 ± 9.09^{a}	6.69 ± 0.46^{a}	0.85 ± 0.06^{ab}
29 WAB	29#1	18.58 ± 0.73^{a}	13.40 ± 0.91^{a}	139.72 ± 1.70^{a}	10.01 ± 0.69^{b}	1.56 ± 0.14^{b}
	29#6	22.26 ± 0.74^{b}	18.68 ± 1.11^{b}	159.12 ± 6.18^{b}	11.65 ± 0.61^{b}	1.00 ± 0.04^{a}
	29#8	20.50 ± 0.94^{ab}	16.35 ± 1.36^{ab}	139.41 ± 4.28^{a}	10.01 ± 0.84^{b}	0.94 ± 0.07^{a}
	29#17	19.29 ± 0.49^{a}	15.52 ± 0.66^{a}	140.12 ± 2.49^{a}	7.28 ± 0.32^{a}	1.05 ± 0.12^{a}

Data are expressed as means ± standard error of three replications.
The significance of differences between groups was analyzed for each harvest time respectively, and different letters indicate significant differences among storage periods ($P < 0.05$).

Sucrose could best explain the changes in TSS and sweetness, which significantly accumulated during on-tree fruit ripening, remained stable during postharvest storage, and had a higher level in 29-WAB-harvested fruits than in 27-WAB-harvested fruits.

Changes in acidity were generally in accordance with the comments on sourness, with a decreasing trend over the entire period of ripening and storage (Fig. 3B). At early stage of ripening, the acidity had a wide distribution of 0.5%–1.8%, while at late stage of storage it ranged from 0.5% to 1.2%. However, there were also exceptions in which acidity was not in accordance with sourness. For example, 29#15 fruits were almost sourless (Fig. 1D), but the acidity (0.85%) was not as low as expected (Fig. 3B). Another inconsistency was that, although the evaluated sourness decreased continually along time (Fig. 1D), the acidity no longer reduced after 34 WAB (Fig. 3B).

Citrate and malate, two major organic acids in mandarin fruit pulp, were measured using GC (Table 1). Citrate was the dominant organic acid in Xinyu mandarin, and the citrate/malate ratio ranged from 6 to 15 among different groups. Citrate content decreased continually during postharvest storage, which well explained the reduction in sourness. However, its dramatic decrease from 27#0 to 28#0 was not in accordance with the gradual decrease in sourness.

3.4. Volatile compounds

GC–MS analysis of volatile compounds was conducted on eight representative groups: 28#0, unripe fruits; 33#0, ripe fruits on tree; 27#2 and 29#1, early postharvest fruits; 27#5 and 29#6, middle postharvest fruits; 27#19 and 29#17, late postharvest fruits. A total of 101 volatiles were identified, including 11 monoterpenes, four sesquiterpenes, 23 monoterpene derivatives, two sesquiterpene derivatives, 10 alcohols, seven aldehydes, 19 esters, 13 aromatics, five fatty hydrocarbons, two acids and five others (Supplementary Table 1).

Cluster analysis showed that the samples of each ripening and postharvest stage had unique volatile profiles. The closest clustering was observed between early and middle postharvest fruits, indicating that the volatile components did not changed a lot during the first 5–6 weeks of storage (Fig. 4A). Principal component analysis also showed that

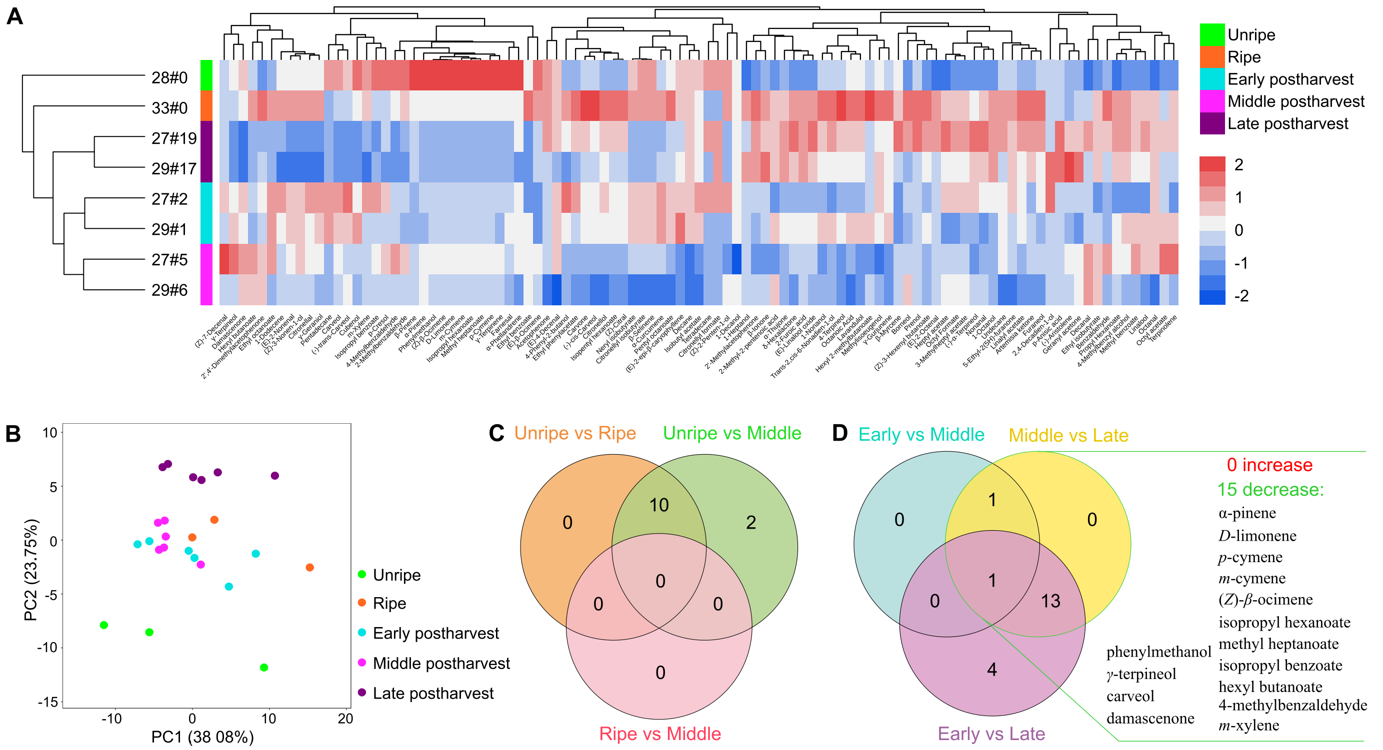


Fig. 4. Comparison of volatile profiles among different ripening and postharvest stages of Xinyu mandarin. (A) Heat map of volatile contents, and clustering of samples and compounds. Red color represents high levels and blue color represents low levels. (B) Principal component analysis. (C) Venn diagram of differential volatile compounds among unripe, ripe and middle postharvest fruits. (D) Venn diagram of differential volatile compounds among fruit at different postharvest stages. Differential volatile compounds between middle and late stages are listed. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

volatile constitutions of early and middle postharvest stages are similar to each other, and they were also similar to the volatile profile of fruits ripened on tree. While the unripe fruits and late postharvest fruits could be clearly distinguished, respectively (Fig. 4B).

The comparison between ripe and unripe fruits showed 10 differential volatile compounds (fold change >2) during ripening process: *α*-pinene, D-limonene, *p*-cymene, *m*-cymene, (*Z*)-*β*-ocimene, isopropyl hexanoate, methyl heptanoate, benzyl alcohol and isopropyl benzoate increased, whereas hexyl butanoate decreased (Table 2). These compounds were also the main differential metabolites between unripe and middle postharvest fruits. No differential metabolite was found between the on-tree ripe fruits and middle postharvest fruits (Fig. 4C). These comparisons indicated that the changes during early postharvest storage were similar to the changes during on-tree ripening.

During storage, only two differential metabolites were found between early and middle postharvest stages, while 15 were found between middle and late postharvest stages (Fig. 4D, Table 2). This was in accordance with the cluster analysis and PCA (Fig. 4A, B), indicating that volatiles of postharvest fruits maintained relatively stable from early to middle stage, but changed greatly from middle to late stage. Between middle and late stages, all the 15 differential metabolites (*α*-pinene, D-limonene, *p*-cymene, *m*-cymene, (*Z*)-*β*-ocimene, *γ*-terpineol, carveol, damascenone, isopropyl hexanoate, methyl heptanoate, isopropyl benzoate, hexyl butanoate, 4-methylbenzaldehyde, *m*-xylene and benzyl alcohol) decreased (Fig. 4D), corresponding to the aroma weakening during this period (Fig. 1E).

In association with the development of off-flavor, three differential metabolites were found to increase during postharvest storage: one sesquiterpene ((−)-aristolene), one aromatic compound (*p*-anisic acid) and one monoterpene derivative (geranylacetone) (Table 2). As described in the TGSC database (http://www.thegoodscentscompany.com), *p*-anisic acid has a "faint, putrid, sweet, cadaverous" odor, geranylacetone has a "green and fruity" flavor, while the odor of (−)-aristolene has not been recorded. Thus, it seemed that *p*-anisic acid was likely to generate an unpleasant flavor and contribute to the off-flavor of mandarin. For validation, we prepared a *p*-anisic solution in water at 0.2 mg/mL, which is a relatively high concentration compared to its solubility in water (0.53 mg/L at 37 °C). However, as far as we feel by smelling and tasting, its orthonasal and retronasal smells were both too weak to compare with the intensity of off-flavor in the fruits.

4. Discussion

Flavor is the most important quality of fresh fruit. Horticulture researchers have made much progress in the chemistry of fruit flavor (Etienne, Genard, Lobit, Mbeguie, & Bugaud, 2013; Lado, Gambetta, & Zacarias, 2018), but the linkage between chemical and sensory attributes is poorly established. Although scoring-based sensory evaluation has been applied to compare sensory qualities of fruit samples (Deterre et al., 2023; Goldenberg et al., 2015; Tietel et al., 2010), this approach is not suitable for commercial purposes. The numeric scores only reflect relative intensities of sensory attributes rather than evaluators' subjective attitudes towards the senses. Therefore, preference analysis is essential to achieving goals in market decision, such as shelf life determination.

In the previous studies on Xinyu mandarin storage and preservation, the assessments of fruit flavor were limited to the measurements of sugars and acids (Chen, Nie, Wan, & Chen, 2019; Chen, Zheng, Wan, Chen, & Chen, 2016). Our findings provide new insights into this issue and suggest that future work should pay more attention to the retronasal smell and volatile components. The results indicated that the limiting factors in consumer satisfaction of this mandarin changed during ripening and postharvest storage. The main cause of the flavor deterioration in late storage period was the decreasing retronasal aroma and

Table 2
Differential volatile compounds identified in Xinyu mandarin of different harvest and storage times (μg/g).

Compound	Unripe	Ripe	Early postharvest		Middle postharvest		Late postharvest	
	28#0	33#0	27#2	29#1	27#5	29#6	27#19	29#17
Monoterpene								
(*E*)-*β*-ocimene	0.15 ± 0.00^{c}	0.14 ± 0.01^{bc}	0.07 ± 0.01^{a}	0.07 ± 0.01^{a}	0.11 ± 0.02^{abc}	0.10 ± 0.00^{ab}	0.09 ± 0.01^{a}	0.09 ± 0.01^{a}
(*Z*)-*β*-ocimene	39.29 ± 10.15^{b}	10.15 ± 3.36^{a}	6.47 ± 1.37^{a}	3.55 ± 0.20^{a}	12.48 ± 4.19^{a}	6.26 ± 1.08^{a}	0.37 ± 0.04^{a}	0.46 ± 0.29^{a}
D-limonene	37.65 ± 9.66^{b}	9.73 ± 3.23^{a}	6.15 ± 1.29^{a}	3.37 ± 0.21^{a}	11.93 ± 4.08^{a}	5.98 ± 1.03^{a}	0.37 ± 0.03^{a}	0.47 ± 0.29^{a}
m-cymene	0.96 ± 0.05^{c}	0.33 ± 0.07^{b}	0.25 ± 0.03^{ab}	0.15 ± 0.02^{ab}	0.31 ± 0.09^{b}	0.20 ± 0.03^{ab}	0.04 ± 0.00^{a}	0.03 ± 0.01^{a}
p-cymene	6.36 ± 1.45^{b}	2.64 ± 0.76^{a}	1.68 ± 0.30^{a}	1.06 ± 0.07^{a}	2.80 ± 0.74^{a}	1.75 ± 0.23^{a}	0.13 ± 0.01^{a}	0.16 ± 0.09^{a}
α-pinene	0.08 ± 0.01^{e}	0.03 ± 0.00^{cd}	0.02 ± 0.00^{bc}	0.01 ± 0.00^{ab}	0.03 ± 0.00^{d}	0.03 ± 0.00^{cd}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}
β-pinene	0.40 ± 0.03^{b}	0.24 ± 0.02^{a}	0.17 ± 0.02^{a}	0.17 ± 0.01^{a}	0.21 ± 0.01^{a}	0.21 ± 0.01^{a}	0.20 ± 0.00^{a}	0.19 ± 0.02^{a}
γ-terpinene	0.09 ± 0.01^{b}	0.06 ± 0.01^{ab}	0.05 ± 0.01^{a}	0.04 ± 0.00^{a}	0.06 ± 0.01^{ab}	0.05 ± 0.00^{a}	0.04 ± 0.00^{a}	0.04 ± 0.00^{a}
Sesquiterpene								
(−)-aristolene	0.03 ± 0.00^{ab}	0.03 ± 0.00^{ab}	0.04 ± 0.00^{abc}	0.03 ± 0.00^{a}	0.05 ± 0.00^{cd}	0.04 ± 0.00^{bcd}	0.06 ± 0.01^{d}	0.08 ± 0.00^{e}
Monoterpene alcohol								
γ-terpineol	1.11 ± 0.03^{bc}	0.86 ± 0.10^{b}	0.95 ± 0.15^{bc}	1.38 ± 0.09^{cd}	1.88 ± 0.20^{d}	0.69 ± 0.04^{b}	0.16 ± 0.02^{a}	0.16 ± 0.01^{a}
carveol	0.27 ± 0.02^{b}	0.18 ± 0.01^{ab}	0.28 ± 0.05^{b}	0.26 ± 0.02^{b}	0.19 ± 0.03^{ab}	0.18 ± 0.01^{ab}	0.09 ± 0.01^{a}	0.08 ± 0.01^{a}
(−)-*trans*-carveol	0.12 ± 0.01^{bc}	0.09 ± 0.01^{ab}	0.15 ± 0.02^{c}	0.12 ± 0.01^{abc}	0.09 ± 0.01^{ab}	0.10 ± 0.01^{ab}	0.07 ± 0.00^{a}	0.07 ± 0.00^{a}
Monoterpene ketone								
geranylacetone	0.09 ± 0.01^{a}	0.11 ± 0.00^{abc}	0.10 ± 0.00^{ab}	0.07 ± 0.01^{a}	0.13 ± 0.01^{bc}	0.12 ± 0.01^{abc}	0.15 ± 0.02^{cd}	0.19 ± 0.00^{d}
damascenone	0.07 ± 0.01^{c}	0.06 ± 0.00^{bc}	0.07 ± 0.01^{c}	0.06 ± 0.00^{abc}	0.08 ± 0.01^{c}	0.07 ± 0.00^{c}	0.03 ± 0.00^{ab}	0.03 ± 0.00^{a}
Sesquiterpene alcohol								
cubenol	0.20 ± 0.00^{b}	0.12 ± 0.02^{a}	0.12 ± 0.01^{a}	0.18 ± 0.03^{b}	0.11 ± 0.00^{a}	0.09 ± 0.00^{a}	0.07 ± 0.00^{a}	0.07 ± 0.01^{a}
Sesquiterpene aldehyde								
farnesal	0.23 ± 0.01^{b}	0.14 ± 0.02^{a}	0.13 ± 0.00^{a}	0.14 ± 0.02^{a}	0.13 ± 0.01^{a}	0.11 ± 0.00^{a}	0.09 ± 0.00^{a}	0.10 ± 0.01^{a}
Alcohol								
benzyl alcohol	15.35 ± 3.93^{b}	4.18 ± 1.33^{a}	2.69 ± 0.55^{a}	1.53 ± 0.08^{a}	5.05 ± 1.65^{a}	2.63 ± 0.43^{a}	0.17 ± 0.02^{a}	0.21 ± 0.13^{a}
Aldehyde								
(*Z*)-7-decenal	0.02 ± 0.00^{a}	0.02 ± 0.00^{ab}	0.03 ± 0.00^{ab}	0.02 ± 0.00^{a}	0.04 ± 0.01^{b}	0.02 ± 0.00^{ab}	0.02 ± 0.00^{a}	0.02 ± 0.00^{a}
decanal	0.01 ± 0.00^{a}	0.02 ± 0.00^{ab}	0.02 ± 0.00^{ab}	0.02 ± 0.00^{ab}	0.03 ± 0.00^{ab}	0.03 ± 0.01^{ab}	0.03 ± 0.00^{b}	0.02 ± 0.00^{ab}
Ester								
ethyl benzoate	0.02 ± 0.00^{abc}	0.03 ± 0.00^{c}	0.01 ± 0.00^{a}	0.02 ± 0.00^{bc}	0.02 ± 0.00^{bc}	0.02 ± 0.00^{bc}	0.01 ± 0.00^{ab}	0.01 ± 0.00^{ab}
hexyl butanoate	0.02 ± 0.00^{a}	0.04 ± 0.01^{b}	0.02 ± 0.00^{a}	0.01 ± 0.00^{a}	0.04 ± 0.00^{b}	0.03 ± 0.00^{b}	0.01 ± 0.00^{a}	0.01 ± 0.00^{a}
isopropyl benzoate	0.46 ± 0.07^{c}	0.19 ± 0.01^{a}	0.37 ± 0.03^{bc}	0.19 ± 0.01^{a}	0.23 ± 0.02^{ab}	0.18 ± 0.05^{a}	0.08 ± 0.01^{a}	0.09 ± 0.01^{a}
isopropyl hexanoate	4.13 ± 0.28^{c}	1.40 ± 0.40^{b}	0.92 ± 0.16^{ab}	0.57 ± 0.02^{ab}	1.51 ± 0.40^{b}	0.93 ± 0.13^{ab}	0.07 ± 0.01^{a}	0.09 ± 0.05^{a}
methyl heptanoate	0.17 ± 0.01^{c}	0.05 ± 0.01^{b}	0.04 ± 0.01^{ab}	0.02 ± 0.00^{ab}	0.06 ± 0.02^{b}	0.04 ± 0.00^{ab}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}
octyl acetate	0.01 ± 0.00^{a}	0.02 ± 0.00^{ab}	0.02 ± 0.00^{ab}	0.03 ± 0.00^{ab}	0.04 ± 0.01^{b}	0.02 ± 0.01^{ab}	0.03 ± 0.01^{ab}	0.02 ± 0.00^{ab}
Aromatic								
4-methylbenzaldehyde	0.05 ± 0.01^{c}	0.03 ± 0.00^{ab}	0.02 ± 0.00^{ab}	0.02 ± 0.00^{ab}	0.04 ± 0.01^{bc}	0.02 ± 0.00^{ab}	0.01 ± 0.00^{a}	0.01 ± 0.00^{a}
m-xylene	0.05 ± 0.01^{b}	0.04 ± 0.00^{ab}	0.05 ± 0.01^{b}	0.03 ± 0.00^{ab}	0.03 ± 0.01^{ab}	0.03 ± 0.01^{ab}	0.02 ± 0.00^{ab}	0.01 ± 0.00^{a}
2-methylbenzaldehyde	0.07 ± 0.01^{c}	0.05 ± 0.00^{bc}	0.04 ± 0.01^{ab}	0.03 ± 0.00^{ab}	0.05 ± 0.01^{bc}	0.04 ± 0.00^{ab}	0.03 ± 0.00^{ab}	0.02 ± 0.00^{a}
p-anisic acid	0.01 ± 0.00^{a}	0.01 ± 0.00^{a}	0.01 ± 0.00^{ab}	0.01 ± 0.00^{ab}	0.01 ± 0.00^{ab}	0.01 ± 0.00^{ab}	0.02 ± 0.00^{ab}	0.02 ± 0.00^{b}
thymol	0.01 ± 0.00^{ab}	0.01 ± 0.00^{ab}	0.01 ± 0.00^{ab}	0.01 ± 0.00^{ab}	0.01 ± 0.00^{a}	0.01 ± 0.00^{ab}	0.01 ± 0.00^{ab}	0.01 ± 0.00^{b}

Data are expressed as means ± standard error of three replications.
Different letters indicate significant differences among groups according to Tukey's HSD test ($P < 0.05$).

emerging off-flavor, while the roles of sweetness and sourness were not important (Fig. 2C). This conclusion is similar to the findings in 'Orri' mandarin, whose postharvest flavor deterioration was mainly featured by the sensation of off-flavor resulting from changes in volatile compounds (Otieno et al., 2022). However, the main causes of flavor deterioration can vary among different citrus varieties. In the storage of 'Mor' mandarin, flavor deterioration was associated with a decreases in sweetness, sourness and mandarin-like flavor, and the accumulation of off-flavor (Tietel et al., 2010). In 'Moro', 'Tarocco Gallo', 'Tarocco TDV' and 'Washington' oranges, only off-flavor significantly accumulated during storage, while aroma, sweetness and acidity did not significantly change (Fabroni et al., 2020).

Sweetness is the main quality of fruit that brings human with pleasure. We observed that TSS or sugars during postharvest storage remained relatively stable at the level when fruits were harvested (Fig. 3A, Table 1), which could be validated by previously published data for other mandarin varieties (Fabroni et al., 2020; Matsumoto & Ikoma, 2012; Ornelas-Paz et al., 2017; Otieno et al., 2022), but unlike the increment in oranges (Habibi & Ramezanian, 2017). These data suggest that sugar contents of mandarins are determined at the time of harvest. Interestingly, although the contents of sucrose and TSS were associated, they were not well correlated with the level of sweetness (Fig. 2). This was not in agreement with the general intuition that fruit sweetness depends on sugar content. In this case, the changes in sweetness were likely to be attributed to the sensation interactions involving organic acids and volatile components (Barba, Beno, Guichard, & Thomas-Danguin, 2018; Fan et al., 2021; Junge et al., 2020).

Sourness plays a key role in determining the acceptability of citrus fruits (Liu, Heying, & Tanumihardjo, 2012). Apparently, excessive sourness creates an awful experience for all consumers. However, attitudes towards low-level sourness may vary among individuals. Some people expect citrus fruit to be as sourless as possible, while some others regard sourness as an important property of citrus flavor and prefer fruits with appropriate sourness. Our data showed that unlike sourness which continually decreased over the entire storage period (Fig. 1D), the overall flavor had an independent changing pattern, although it also decreased from middle to late storage stage (Fig. 1B). This means that fruits with either appropriate sourness or imperceptible sourness are satisfactory for most consumers. Correlation analysis further confirmed that overall flavor was poorly correlated with sourness at late postharvest stage when the sourness was low or imperceptible (Fig. 2C). Thus, we conclude that the reduction in sourness was not the main cause of flavor deterioration, although it was observed during postharvest storage in both this and previous studies (Marcilla, Martínez, Carot, Palou, & Del Río, 2009; Tietel et al., 2010).

In this study, off-flavor was identified as an important factor leading to postharvest flavor deterioration of mandarin. Mandarin off-flavor has previously been reported to be related to a series of volatile compounds.

Ethanol and acetaldehyde are the most reported that increase with off-flavor development in harvested mandarin, especially those with wax coatings (Cohen, Shalom, & Rosenberger, 1990; Hagenmaier & Shaw, 2002; Tietel et al., 2010). The development of ethanol, acetaldehyde and off-flavor can be even faster in mandarin than in other citrus varieties, because mandarins have higher alcohol dehydrogenase activity in the pulp and their peel is less permeable to gases (Shi, Goldschmidt, Goren, & Porat, 2007). It has also been proposed that products of fatty acid and amino acid catabolism (Tietel et al., 2010; Tietel, Feldmesser, Lewinsohn, Fallik, & Porat, 2011), ethyl esters (Otieno et al., 2022), and a non-volatile flavor compound putrescine (Fabroni et al., 2020) may be potential substances responsible for the off-flavor of mandarins during storage. In this study, *p*-anisic acid, geranylacetone and (−)-aristolene were found to be associated with off-flavor. Among these, *p*-anisic acid is described to have an unpleasant smell in the chemical database, but its retronasal smell was too weak according to our preliminary test, so its role in forming the off-flavor may be trivial. Geranylacetone is described to have fruity flavor in the database, but it may also cause "overripe" flavor when accumulated to a high concentration. To our knowledge, the smell and flavor of (−)-aristolene have not been described.

Given that retronasal aroma was a key sensory factor in maintaining postharvest flavor of Xinyu mandarin, we further analyzed volatile profiles of the mandarin at different stages. In this study, 15 volatile compounds were found to decrease significantly during late postharvest storage. Thus, they were all potential contributors to the decrease in aroma. Among these compounds, the roles of α-pinene and D-limonene are of particularly concern. On the one hand, they were previously identified key aroma compounds for mandarins, supported by evidence from GC-O, OAVs, aroma recombination and omission tests (Feng et al., 2018; Xiao et al., 2017; Zhou et al., 2021). On the other hand, the decrease in α-pinene and D-limonene was also associated with a decrease in mandarin-like flavor in other mandarins during storage. In both 'Orri' and 'Mor' mandarins whose "fruity aroma" or "mandarin flavor" decreased during storage, their α-pinene and D-limonene contents also decreased greatly (Otieno et al., 2022; Tietel et al., 2010). By contrast, in both 'Owari' and 'W. Murcott' mandarins whose mandarin-like flavor (richness) did not changed by storage, neither α-pinene nor D-limonene of them was found to significantly changed during storage (Obenland, Collin, Mackey, Sievert, & Arpaia, 2011). Taken together, α-pinene and D-limonene are likely to be the key chemicals associated with aroma reduction of mandarins caused by storage.

In summary, this study provides new insights into the sensory and chemical basis underlying flavor deterioration of postharvest mandarin fruit. The detailed dynamics of sensory attributes indicate that the fading of retronasal aroma and emergence of off-flavor, but not sweetness or sourness, contribute to mandarin flavor deterioration. The decrease in retronasal aroma mainly occurs in late postharvest period. During this period, 15 volatile compounds decrease, and two key aroma compounds, α-pinene and D-limonene, are included. These chemicals are likely determinants of mandarin flavor deterioration.

CRediT authorship contribution statement

Xin-Cheng Liu: Methodology, Investigation, Formal analysis, Writing - original draft. **Yu-Qing Tang:** Investigation, Validation, Writing - review & editing. **Yin-Chun Li:** Investigation, Data curation. **Shao-Jia Li:** Validation, Writing - review & editing. **Hui-Dong Yang:** Data curation. **Shui-Lin Wan:** Resources. **Yu-Ting Wang:** Writing - review & editing, Funding acquisition. **Zhong-Dong Hu:** Conceptualization, Funding acquisition, Supervision, Writing - review & editing.

Declaration of competing interest

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

Data availability

Data will be made available on request.

Acknowledgements

The authors acknowledge all the volunteers who participated in the fruit sensory evaluation. This research was supported by the Modern Agricultural Research Collaborative Innovation Program of Jiangxi Province (JXXTCXQN202106, JXXTCX202203), the earmarked fund for China Agriculture Research System (CARS-26), and the Earmarked Fund for Jiangxi Agriculture Research System (JXARS-07).

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fochx.2024.101395.

References

Barba, C., Beno, N., Guichard, E., & Thomas-Danguin, T. (2018). Selecting odorant compounds to enhance sweet flavor perception by gas chromatography/olfactometry-associated taste (GC/O-AT). *Food Chemistry, 257*, 172–181. https://doi.org/10.1016/j.foodchem.2018.02.152

Cao, J. P., Kang, C., Chen, Y. Y., Karim, N., Wang, Y., & Sun, C. D. (2020). Physiochemical changes in *Citrus reticulata* cv. Shatangju fruit during vesicle collapse. *Postharvest Biology and Technology, 165*, Article 111180. https://doi.org/10.1016/j.postharvbio.2020.111180

Chen, C., Nie, Z., Wan, C., & Chen, J. (2019). Preservation of Xinyu tangerines with an edible coating using *Ficus hirta* Vahl. Fruits extract-incorporated chitosan. *Biomolecules, 9*(2). https://doi.org/10.3390/biom9020046

Chen, C., Zheng, J. P., Wan, C. P., Chen, M., & Chen, J. Y. (2016). Effect of carboxymethyl cellulose coating enriched with clove oil on postharvest quality of 'Xinyu' mandarin oranges. *Fruits, 71*, 319–327. https://doi.org/10.1051/fruits/2016019

Cohen, E., Shalom, Y., & Rosenberger, I. (1990). Postharvest ethanol buildup and off-flavor in 'Murcott' tangerine fruits. *Journal of the American Society for Horticultural Science jashs, 115*(5), 775–778. https://doi.org/10.21273/jashs.115.5.775

Deterre, S. C., Jeffries, K. A., McCollum, G., Stover, E., Leclair, C., Manthey, J. A., … Plotto, A. (2023). Sensory quality of *Citrus* scion hybrids with *Poncirus trifoliata* in their pedigrees. *Journal of Food Science, 88*(4), 1684–1699. https://doi.org/10.1111/1750-3841.16499

Etienne, A., Genard, M., Lobit, P., Mbeguie, A. M. D., & Bugaud, C. (2013). What controls fleshy fruit acidity? A review of malate and citrate accumulation in fruit cells. *Journal of Experimental Botany, 64*(6), 1451–1469. https://doi.org/10.1093/jxb/ert035

Fabroni, S., Amenta, M., Timpanaro, N., Todaro, A., & Rapisarda, P. (2020). Change in taste-altering non-volatile components of blood and common orange fruit during cold storage. *Food Research International, 131*, Article 108916. https://doi.org/10.1016/j.foodres.2019.108916

Fan, Z., Hasing, T., Johnson, T. S., Garner, D. M., Schwieterman, M. L., Barbey, C. R., … Whitaker, V. M. (2021). Strawberry sweetness and consumer preference are enhanced by specific volatile compounds. *Horticulture Research, 8*(1), 66. https://doi.org/10.1038/s41438-021-00502-5

Feng, S., Suh, J. H., Gmitter, F. G., & Wang, Y. (2018). Differentiation between flavors of sweet orange (*Citrus sinensis*) and mandarin (*Citrus reticulata*). *Journal of Agricultural and Food Chemistry, 66*(1), 203–211. https://doi.org/10.1021/acs.jafc.7b04968

Goldenberg, L., Yaniv, Y., Doron-Faigenboim, A., Carmi, N., & Porat, R. (2016). Diversity among mandarin varieties and natural sub-groups in aroma volatiles compositions. *Journal of the Science of Food and Agriculture, 96*(1), 57–65. https://doi.org/10.1002/jsfa.7191

Goldenberg, L., Yaniv, Y., Kaplunov, T., Doron-Faigenboim, A., Carmi, N., & Porat, R. (2015). Diversity in sensory quality and determining factors influencing mandarin flavor liking. *Journal of Food Science, 80*(2), S418–S425. https://doi.org/10.1111/1750-3841.12742

Goldenberg, L., Yaniv, Y., Porat, R., & Carmi, N. (2018). Mandarin fruit quality: A review. *Journal of the Science of Food and Agriculture, 98*(1), 18–26. https://doi.org/10.1002/jsfa.8495

Habibi, F., & Ramezanian, A. (2017). Vacuum infiltration of putrescine enhances bioactive compounds and maintains quality of blood orange during cold storage. *Food Chemistry, 227*, 1–8. https://doi.org/10.1016/j.foodchem.2017.01.057

Hagenmaier, R. D., & Shaw, P. E. (2002). Changes in volatile components of stored tangerines and other specialty citrus fruits with different coatings. *Journal of Food Science, 67*(5), 1742–1745. https://doi.org/10.1111/j.1365-2621.2002.tb08716.x

Hannum, M. E., Fryer, J. A., & Simons, C. T. (2021). Non-food odors and the duality of smell: Impact of odorant delivery pathway and labeling convention on olfactory perception. *Physiology & Behavior, 238*, Article 113480. https://doi.org/10.1016/j.physbeh.2021.113480

Junge, J. Y., Bertelsen, A. S., Mielby, L. A., Zeng, Y., Sun, Y.-X., Byrne, D. V., & Kidmose, U. (2020). Taste interactions between sweetness of sucrose and sourness of citric and tartaric acid among Chinese and Danish consumers. *Foods, 9*(10), 1425. https://doi.org/10.3390/foods9101425

Kang, C., Cao, J., Wang, Y., & Sun, C. (2022). Advances of section drying in citrus fruit: The metabolic changes, mechanisms and prevention methods. *Food Chemistry, 395*, Article 133499. https://doi.org/10.1016/j.foodchem.2022.133499

Lado, J., Gambetta, G., & Zacarias, L. (2018). Key determinants of citrus fruit quality: Metabolites and main changes during maturation. *Scientia Horticulturae, 233*, 238–248. https://doi.org/10.1016/j.scienta.2018.01.055

Liu, X. C., Lin, X. H., Liu, S. C., Zhu, C. Q., Grierson, D., Li, S. J., & Chen, K. S. (2021). The effect of NH_4^+ on phosphoenolpyruvate carboxykinase gene expression, metabolic flux and citrate content of citrus juice sacs. *Plant Physiology and Biochemistry, 167*, 123–131. https://doi.org/10.1016/j.plaphy.2021.07.041

Liu, Y. Q., Heying, E., & Tanumihardjo, S. A. (2012). History, global distribution, and nutritional importance of citrus fruits. *Comprehensive Reviews in Food Science and Food Safety, 11*(6), 530–545. https://doi.org/10.1111/j.1541-4337.2012.00201.x

López-Gómez, A., Ros-Chumillas, M., Buendía-Moreno, L., Navarro-Segura, L., & Martínez-Hernández, G. B. (2020). Active cardboard box with smart internal lining sased on encapsulated essential oils for enhancing the shelf life of fresh mandarins. *Foods, 9*(5), 590. https://doi.org/10.3390/foods9050590

Marcilla, A., Martínez, M., Carot, J., Palou, L., & Del Río, M. (2009). Relationship between sensory and physico-chemical quality parameters of cold-stored "Clemenules" mandarins coated with two commercial waxes. *Spanish Journal of Agricultural Research, 7*(1), 181–189. https://doi.org/10.5424/sjar/2009071-410

Matsumoto, H., & Ikoma, Y. (2012). Effect of different postharvest temperatures on the accumulation of sugars, organic acids, and amino acids in the juice sacs of Satsuma mandarin (*Citrus unshiu* Marc.) fruit. *Journal of Agricultural and Food Chemistry, 60* (39), 9900–9909. https://doi.org/10.1021/jf303532s

Miyazaki, T., Plotto, A., Baldwin, E. A., Reyes-De-Corcuera, J. I., & Gmitter, F. G., Jr. (2012). Aroma characterization of tangerine hybrids by gas-chromatography–olfactometry and sensory evaluation. *Journal of the Science of Food and Agriculture, 92*(4), 727–735. https://doi.org/10.1002/jsfa.4663

Obenland, D., Collin, S., Mackey, B., Sievert, J., & Arpaia, M. L. (2011). Storage temperature and time influences sensory quality of mandarins by altering soluble solids, acidity and aroma volatile composition. *Postharvest Biology and Technology, 59* (2), 187–193. https://doi.org/10.1016/j.postharvbio.2010.09.011

Ornelas-Paz, J.d. J., Meza, M. B., Obenland, D., Rodríguez, K., Jain, A., Thornton, S., & Prakash, A. (2017). Effect of phytosanitary irradiation on the postharvest quality of seedless Kishu mandarins (*Citrus kinokuni mukakukishu*). *Food Chemistry, 230*, 712–720. https://doi.org/10.1016/j.foodchem.2017.02.125

Otieno, J., Owoyemi, A., Goldenberg, L., Yaniv, Y., Carmi, N., & Porat, R. (2022). Effects of packinghouse operations on the flavor of ‘Orri’ mandarins. *Food Science & Nutrition, 10*(4), 1320–1328. https://doi.org/10.1002/fsn3.2778

Owoyemi, A., Porat, R., Lichter, A., Doron-Faigenboim, A., Jovani, O., Koenigstein, N., & Salzer, Y. (2022). Large-scale, high-throughput phenotyping of the postharvest storage performance of ‘Rustenburg’ navel oranges and the development of shelf-life prediction models. *Foods, 11*(13), 1840. https://doi.org/10.3390/foods11131840

Ren, J. N., Tai, Y. N., Dong, M., Shao, J. H., Yang, S. Z., Pan, S. Y., & Fan, G. (2015). Characterisation of free and bound volatile compounds from six different varieties of citrus fruits. *Food Chemistry, 185*, 25–32. https://doi.org/10.1016/j.foodchem.2015.03.142

Shi, J. X., Goldschmidt, E. E., Goren, R., & Porat, R. (2007). Molecular, biochemical and anatomical factors governing ethanol fermentation metabolism and accumulation of off-flavors in mandarins and grapefruit. *Postharvest Biology and Technology, 46*(3), 242–251. https://doi.org/10.1016/j.postharvbio.2007.05.009

Tietel, Z., Bar, E., Lewinsohn, E., Feldmesser, E., Fallik, E., & Porat, R. (2010). Effects of wax coatings and postharvest storage on sensory quality and aroma volatile composition of ‘Mor’ mandarins. *Journal of the Science of Food and Agriculture, 90*(6), 995–1007. https://doi.org/10.1002/jsfa.3909

Tietel, Z., Feldmesser, E., Lewinsohn, E., Fallik, E., & Porat, R. (2011). Changes in the transcriptome of ‘Mor’ mandarin flesh during storage: Emphasis on molecular regulation of fruit flavor deterioration. *Journal of Agricultural and Food Chemistry, 59* (8), 3819–3827. https://doi.org/10.1021/jf104614s

Tietel, Z., Plotto, A., Fallik, E., Lewinsohn, E., & Porat, R. (2011). Taste and aroma of fresh and stored mandarins. *Journal of the Science of Food and Agriculture, 91*(1), 14–23. https://doi.org/10.1002/jsfa.4146

Xiao, Z. B., Wu, Q. Y., Niu, Y. W., Wu, M. L., Zhu, J. C., Zhou, X., … Kong, J. L. (2017). Characterization of the key aroma compounds in five varieties of mandarins by gas chromatography–olfactometry, odor activity values, aroma recombination, and omission analysis. *Journal of Agricultural and Food Chemistry, 65*(38), 8392–8401. https://doi.org/10.1021/acs.jafc.7b02703

Yuan, H., Cao, G., Hou, X., Huang, M., Du, P., Tan, T., … Luo, J. (2022). Development of a widely targeted volatilomics method for profiling volatilomes in plants. *Molecular Plant, 15*(1), 189–202. https://doi.org/10.1016/j.molp.2021.09.003

Yue, C., Cao, H., Zhang, S., Hao, Z., Wu, Z., Luo, L., & Zeng, L. (2023). Aroma characteristics of Wuyi rock tea prepared from 16 different tea plant varieties. *Food Chemistry: X, 17*, Article 100586. https://doi.org/10.1016/j.fochx.2023.100586

Zhang, H. P., Xie, Y. X., Liu, C. H., Chen, S. L., Hu, S. S., Xie, Z. Z., … Xu, J. (2017). Comprehensive comparative analysis of volatile compounds in citrus fruits of different species. *Food Chemistry, 230*, 316–326. https://doi.org/10.1016/j.foodchem.2017.03.040

Zhou, Q., Tan, A. Q., Ou Yang, Z., Liao, H. B., Li, G. J., Cheng, Y. J., … Wang, H. (2021). Comparison of key aromas between sweet orange and mandarin juices and their roles in the presentation of main flavors of the two juices (in Chinese). *Food and Fermentation Industries, 47*(21), 259–267. https://doi.org/10.13995/j.cnki.11-1802/ts.027023