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The effects of controlled freezing-point storage on the postharvest quality of satsuma mandarin

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ABSTRACT

Present study introduced a controlled freezing-point storage (CFPS) technique for the postharvest storage of satsuma mandarin (*Citrus unshiu* Marc. cv. Miyagawa Wase), with the environment temperature of −2 °C during the whole storage period. The postharvest loss and quality changes of satsuma mandarin were investigated. According to the results, CFPS completely prevented fruit decay in the 90 days storage and the following shelf period without causing fruit chilling injury. In comparison, the decay rate of fruit stored at 10 °C increased with the elongation of storage time, and reach to 6.67% after 90 days storage, then further increased to 33.33% after transferring to the shelf. CFPS significantly reduced the incidences of calyx browning, completely prevented fruit puffing, alleviated the decreases of citric acid, fructose and glucose in the long-term storage and the following shelf life of satsuma mandarin, compared with the 10 °C storage. But CFPS alleviated the postharvest rind color development of satsuma mandarin, leading to paler rind color compared to the 10 °C storage group. Both 10 °C storage and CFPS group showed significant decrease of total aroma volatiles contents in the pulp after more than two months' storage. But most of the volatiles, including all of the monoterpenes and aliphatic alcohols, as well as part of the monoterpenes derivatives and aliphatic aldehydes, rebound after being transferred to the 20 °C shelf, with the extent much higher in 10 °C storage group than that in CFPS group. In the shelf-life, the total volatile content of the 10 °C storage group was about 2 fold that of the newly-harvested fruit, but the volatiles of the CFPS group were more similar to the newly-harvested fruit in content and composition. Correspondingly, the consumer's sensory evaluation results showed that the fruit of CFPS group was slightly sourer and with better typical mandarin flavor than those stored at 10 °C. Comprehensively, CFPS was applicable in reducing the postharvest loss and alleviating quality deterioration in the long-term storage of satsuma mandarin.

1. Introduction

Satsuma mandarin (*Citrus unshiu* Marc. cv. Miyagawa Wase) is one of the widely planted mandarin cultivars with delicious taste and attractive appearance. However, satsuma mandarins are relatively perishable and have shorter postharvest storage life than many other citrus fruit[1].

In practical production, the mandarin fruit were usually preserved at cold or room temperature after treated with preservative agents. The optimum storage temperature for satsuma mandarin is determined to be in a range of 5−10 °C[2-5]. Improper high storage temperatures lead to increase of decay rate and accelerated quality deterioration, while inappropriate low temperatures (0−2 °C) lead to chilling injury of mandarin fruit[5-8]. The combination of preservative agents and cold storage at optimal temperature effectively prolongs the storage period of satsuma fruit[9-10]. However, off flavor happens inevitably after long-term storage, due to the accumulation of volatile compounds,

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such as ethanol, acetaldehyde, and the ethyl esters derivatives[5,11]. Generally, the quality of mandarin fruit decreased with the elongation of storage time and the increase of temperature[12].

Freezing-point storage is a technique for storing foods in a temperature below 0 °C but higher than the freezing point of certain food. In the freezing-point storage, the cellular metabolism and the growth of microorganismare is largely inhibited, and thus the storage period is significantly prolonged[13-14]. The application of freezing-point storage technique on fruit is firstly reported in 1970s by Japanese scientists on the storage of pears[15]. So far, freezing-point storage technique has been applied widely in the fruit with high sugar content and strong respiratory intensity which are hard to store, such as litchi[16], pear[17], peach[18], blue berry[19], sweet cherry[20] and grape[21]. A number of advantages had been reported on freezing-point storage technology, especially for the long-term storage of fruit. Sub-zero temperature of −1.2 °C significantly prevents the decrease of sugars, acids, amino acids, juice rate and vitamin C contents of litchi fruit during the 35 days storage compared to 3 °C[16]. Sub-zero storage temperature of −1 °C significantly reduces the respiratory intensity and ethylene release rate, and delays the fruit softening by reducing the degradation of protopectin and fiber of litchi fruit during the 60 days storage, compared to 3 °C[22]. Controlled freezing-point storage (CFPS) at −1.2 °C largely prolong the shelf life of sweet cherry, prevented the decrease of fruit hardness, total soluble solid (TSS) and titratable acids in the 70 days storage[20]. However, the freezing-point storage is a technically demanding process, as the appropriate ranges of temperature is narrow. The freezing point varied among fruit species. For example, the freezing point of strawberry is detected to be −0.8 °C, while that of grape is detected to be −4.5 °C[23]. Thus, it is necessary to explore the optimal technical parameters before applying the freezing-point storage on specific fruit.

To the best of our knowledge, the storage characteristics of satsuma mandarin under above-zero temperatures (0−30 °C) have been actively studied, but the study on sub-zero temperature storage was rarely seen. The lowest storage temperature reported in citrus fruit is on sweet orange, which is set to be −0.6 °C during shipping for about 3−4 weeks[24]. Present study carried out CFPS on satsuma mandarin fruit, to investigate the storage and shelf life performance of satsuma mandarin under sub-zero non-freezing temperature for a long time.

2. Materials and methods

2.1 Materials

Disease and mechanical damage-free satsuma mandarin fruit of uniform size and color were picked in November from the orchard located in Linhai City, Zhejiang Province, China. The TSS contents of the newly-harvested fruit were detected with a refractometer PR101-a (Atago Co., Ltd., Tokyo, Japan), by using fifteen randomly selected single fruit, with two measurements per fruit.

2.2 Freezing curve construction and freezing point determination

The fruit were laid in the −10 °C freezer, and the temperature of the fruit tissue was recorded every 10 min for 14 h, by plugging the temperature probe into the center of the fruit from the blossom-end. Three biological repetitions were done, with 3 fruit for each repetition. The freezing curve was constructed based on the average value of repetitions, and the freezing point was determined according to the method of Wang et al.[23]. The first turning point existed in the sub-zero temperature range of the freezing curve was determined as the supercooling point. Then the liquid in the fruit began to freeze, and thus the fruit temperature increased due to the heat release during phase-transition. When the temperature reached to a maximum value, the second turning point come and was determined as the freezing point of fruit. After that, the fruit temperature decreased continuously as most of the liquid was frozen. The temperature above the freezing point was determined to be the non-freezing temperature of satsuma fruit.

2.3 Storage and shelf-life simulation

The fruit were put into sterilized plastic baskets and stored in the refrigerated warehouses. All the refrigerated warehouses were equipped with temperature and humidity monitoring and controlling systems, in which the temperature and humidity would be real-time detected and automatically controlled. The temperature for the CFPS group was set as −2 °C, with the actual temperatures were detected to be fluctuating between −1 and −2 °C during the whole storage period. The temperature for the cold storage group was set as 10 °C (relative humidity (RH) of 85%−90%), as commonly done in the citrus industry, with the actual temperatures were detected to be fluctuating between 9.5 and 10.5 °C during the whole storage period. After 90 days of storage, the fruit were moved out and transferred to the 20 °C warehouse (RH 85%−90%) and stored for 7 days to simulate the shelf-life environment.

2.4 Decay rate calculation and appearance evaluation

The decay rate was calculated as the percentage of decayed fruit in the total number of fruit. A total of 150 fruit were used for the decay rate determination for each group. The fruit with the pulp detached from the peel was determined as puffy fruit. The calyx browning was determined by observation according to a reported method[10].

2.5 Weight loss determination

Twenty-four fruit were randomly selected from each treatment and marked. The fruit weight was recorded at different time points. The weight loss rate was calculated according to the following formula:

$$\text{Weight loss rate (\%)} = \frac{m_0 - m_i}{m_0} \times 100 \quad (1)$$

Where m_0 represents the initial fruit weight before storage and m_i represents the fruit weight of the certain sampling day.

2.6 Rind color analysis

Rind color was measured according to the method of Tang et al.[25]. The data was collected at 4 evenly distributed equatorial sites of each fruit using Hunter Lab Mini Scan XEPlus colorimeter (Hunter Associates Laboratory, Inc., Reston, VA, USA). Nine fruit were

measured for each treatment. The CIE 1976 $L^*a^*b^*$ color scale was adopted. The rind color was evaluated by applying citrus color index (CCI) which was calculated according to the following formula:

$$CCI = 1\,000 \times \frac{a^*}{L^* \times b^*} \quad (2)$$

Where L^*, a^*, and b^* values were lightness, redness, and greenness, respectively.

2.7 Sugars composition analysis

The contents of sugars were detected according to the methods of Chen et al.[26] with slight modification. Briefly, 1 g of grounded fresh pulp was extracted with 3 mL of 80% methanol for 20 min. The supernatant was collected by centrifugation at 8 000 × *g* for 15 min. Then repeated the extraction for additional two times. Combined the supernatant of 3 extractions and added with 80% ethanol to a final volume of 10 mL. The extract of 1 mL was obtained for vacuum centrifugation evaporation at 30 °C to remove the solvent. The residue was dissolved in 0.5 mL of ddH_2O, and filtrated through 0.45 μm membrane for high performance liquid chromatography (HPLC) detection. Three repetitions were done for each treatment, with 3 fruit for each repetition. The HPLC detection was carried out in Waters 2695 HPLC system (Waters Corp., MA, USA) coupled with a RID-10A refractiveindex detector (Shimadzu Corp., Kyoto, Japan) and Intersil-NH_2 column (4.6 mm × 250 mm, 5 μm, GL Sciences, Tokyo, Japan). The elution was carried out using 80% acetonitrile as mobile phase, with a flow rate of 1 mL/min and column temperature of 25 °C. The sugars contents were calculated according to the standard curves of authentic compounds. The contents were expressed as g/kg on a fresh weight basis.

2.8 Organic acids composition analysis

The contents of organic acids were evaluated using gas chromatography (GC) method according to Lin et al.[27] with slight modification. For the sample extraction, 0.1 g of the grounded fresh sample was extracted with 1.4 mL of methanol by vortexing at 70 °C for 15 min. The supernatant was collected by centrifugation at 8 000 × *g* for 10 min. Then, 1.5 mL of H_2O and 750 μL of chloroform were added into the supernatant and vortexed. The mixture was centrifuged at 8 000 × *g* for 10 min and 1 mL of upper layer liquid was collected and stored in −40 °C for later use. Three repetitions were done for each treatment, with 3 fruit for each repetition. For the derivation, aliquots of 100 μL upper phase were obtained, added with 10 μL ribitol (0.2 g/L) as internal standard, and dried in centrifugal vacuum evaporator at 30 °C. The residue was dissolved in 40 μL of 20 g/L pyridine methoxyamine hydrochloride, and incubated for 1.5 h at 37 °C. The sample was then mixed with 40 μL of BSTFA reagent (containing 1% trimethylchlorosilane (TCMS)) and incubated at 37 °C for 30 min.

The GC analysis was carried out in an Agilent 7980 gas chromatograph instrument (Agilent J&W, Folsom, CA, USA) coupled with a flame ionization detector (FID) detector and a HP-5ms capillary-column (30 m × 0.25 mm, 0.25 μm). One microliter of each sample was absorbed and injected into GC with a split ratio of 25:1. The injector temperature was 250 °C and the nitrogen carrier gas had a flow rate of 1 mL/min. The column temperature was held at 100 °C for 1 min, increased to 184 °C with a temperature gradient of 3 °C/min, increased to 190 °C at 0.5 °C/min, heldfor 1 min, increased to 280 °C at 5 °C/min and then held for 3 min. The organic acids contents were calculated according to the standard curves of authentic compounds. The contents were expressed as g/kg on a fresh weight basis.

2.9 Detection of ethanol and acetaldehyde

The ethanol and acetaldehyde were detected using static headspace extraction-GC according to the method described by Min et al.[28]. The grounded fresh pulp of 3 g was mixed with 4 mL of saturated NaCl solution. The mixture of 3 mL was put into the 10 mL headspace extraction bottles, added with 10 μL of 1% sec-butyl alcohol as internal control, and heated at 60 °C water bath for 1 h. The gas of 1 mL was extracted from the head space of the bottle for GC analysis. Ethanol and acetaldehyde were detected with Agilent 7980A gas chromatograph instrument (Agilent J&W, Folsom, CA, USA) coupled with FID and HP-INNOWAX column (30 m × 0.25 mm, 0.25 μm, Agilent J&W, Folsom, CA, USA). The injector, detector, and oven temperatures were 150, 160, and 100 °C, respectively. The results were calculated using standard curves for acetaldehyde and ethanol, respectively. The internal standards were used for compensating for differences between samples. Three repetitions were done for each treatment, with 3 fruit for each repetition. Results were expressed as mg/kg on a fresh weight basis.

2.10 Sensory evaluation

Descriptive sensory analysis were conducted with the aid of a sensory panel comprised of 15 members. The sensory attributes included sweetness, sourness, mandarin odor, off-flavor, gumminess, and juiciness. The intensity of each attribute was evaluated in a 0−5 scale. The general sensory quality was graphically presented as spider chart.

2.11 GC-MS analysis of volatile compounds

The quantitation of volatile compounds was carried out according to the methods described by Shen et al.[29], combining headspace solid-phase microextraction (SPME) and gas chromatograph-mass spectrum (GC-MS) analysis. Briefly, 1 g of the grounded fresh pulp was mixed with 5 mL saturated NaCl solution in a 15 mL headspace vial, adding with 50 μL of 1-hexanol (0.1%, *V/V*) as internal standard. After vigorous vortexing, the samples were incubated at 40 °C for 30 min with continuous agitation. A SPME fiber coated with 50/30 μm divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS) (Supelco Co., Bellefonte, PA, USA) was used to extract the volatiles under the same conditions (40 °C with continuous agitation). Volatile analysis was carried out with an Agilent 7890A gas chromatograph coupled to an Agilent 5975C Network Mass Selective Detector (MS, inert XL MSD with triple-axis detector) (Agilent J&W, Folsom, CA, USA). After extraction, the fiber was exposed to the GC injection port at 250 °C for 5 min in splitless mode. Compounds were separated using a HP-5ms column (5% phenyl methyl siloxane, 60 m × 0.25 mm, 0.25 μm, Agilent J&W, Folsom,

CA, USA), with helium as carrier gas at a flow rate of 1 mL/min. The oven temperature was programmed to start at 40 °C for 3 min, and then ramped to 70 °C at a rate of 3 °C/min, followed by a second ramp to 130 °C at a rate of 1 °C/min, and a third ramp to 230 °C at a rate of 15 °C/min. MS conditions were as follows: ion source, 230 °C; electron energy, 70 eV; GC-MS interface zone, 250 °C, and a scan range of 35−350 mass units. Volatiles were identified based on the database of the NIST/EPA/NIH Mass Spectral Library (http://chemdata.nist.gov/). The identities of most of the volatiles were then confirmed by comparison with authentic standards. The internal standards were used for compensating for differences between samples, and the abundance of each volatile was calculated as its peak area. Three repetitions were done for each treatment, with 3 fruit for each repetition. The results were expressed as mg/kg on a fresh weight basis.

2.12 Statistics

Results were shown with standard deviations. All data was statistically assessed using SPSS version 20.0. Statistical significance of differences was calculated using one-way ANOVA. Comparisons between groups were performed using student's *t*-test or Tukey method. Principal component analysis (PCA) was carried out using SIMCA software (V14.1, Umetrics, Umea, Sweden).

3. Results and discussion

3.1 The freezing curve of satsuma mandarin

The satsumas mandarin fruit used for freezing curve construction had TSS contents in a range of 8.7%−13.6%, with the average value of (11.80 ± 1.07)%. The freezing curve of the satsuma mandarin was obtained (Fig. 1). At first, the fruit temperature decreased rapidly, and then slightly rebounded at around 20 min, which might be due to the release of field heat. After 1 hour, the fruit temperature steadily declined, and reached to the lowest point of −6.6 °C at 400 min, which was determined as the supercooling point. Then the liquid in the fruit began to freeze, and thus the fruit temperature increased due to the heat release during phase-transition, reaching a maximum value of about −3.0 °C. After that, the fruit temperature decreased continuously as most of the liquid was frozen. Thus, the temperature above −3.0 °C was determined to be the non-freezing temperature of satsuma fruit.

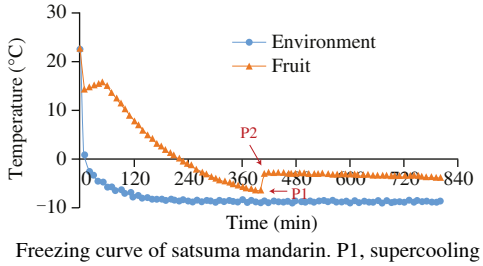

Fig. 1 Freezing curve of satsuma mandarin. P1, supercooling point; P2, freezing point.

Generally, the freezing curve of fruit included three stages. The first stage was characterized by a dramatic and continuous decrease of temperature, and the lowest point was determined as the supercooling point. In the second stage, a slight increase of temperature happened due to the heat release during phase-transition of the cell sap. In the third stage, most of the liquid was frozen, and the temperature declined slowly[16,18]. The freezing curve of satsuma mandarin in present study was basically similar to that of other fruit such as grape, winter jujube, tomato and litchi[16,23].

According to the freezing curve of satsuma mandarin fruit, we chose −2 °C as the CFPS temperature, taking temperature volatility risks into consideration. The actual temperature was detected to be fluctuating between −1 and −2 °C during the whole storage period.

3.2 The effects of CFPS on fruit decay rate and appearance

During the 90 days storage period, no decay happened in the CFPS group, comparing to 6.67% decay rate in the 10 °C storage group (Fig. 2B). This was in agree with previous studies, that lower temperatures were beneficial for reducing the decay rate of citrus fruit[30].

It is worth noting that, the decay rate of the 10 °C storage group dramatically increased to 33% in the 7 days shelf-life after 90 days storage, while no fruit decay happened in the CFPS group (Fig. 2B). As shelf-life is important for judging the applicability of a storage technique, this result indicated that the CFPS was applicable in the long-term storage of satsuma mandarin.

Chilling injury is the fruit physiological disorder commonly happened during cold storage. For citrus fruit, chilling injury developed in the form of pitting and scald on the surfaceofthe fruit, which was visible. In the study of Yuen et al.[6], chilling injury occurred in the 'Emperor' mandarin after 3 weeks of storage at 0 °C, and the incidence increased with the elongation of storage time. In the 'Fortune' mandarin, 2 °C was considered as the chilling injury temperature[7]. In the study of Bajwa et al.[2], *Citrus reticulata* Blanco mandarins developed chilling injury with watery lesions on the rind after storing at 2 °C for more than 45 days. According to the study of Ghasernnezhad et al.[8], chilling symptoms appeared after stored at 2 °C for more than 4 weeks and the symptoms increased with storage time. These previous studies indicated that, 0−2 °C was the chilling temperature of many mandarin cultivars. However, in our study, during the 90 days storage period and the following 7 days shelf life, no chilling injury was observed in both 10 °C group and CFPS group (Fig. 2A), indicating that a lower temperature of −2 °C was applicable for the long-term storage of satsuma mandarin. The shelf life performance further supported that the CFPS did not lead to chilling injury of satsuma mandarin fruit in the 90 days storage. Thus, the correlation between storage temperature and fruit chilling injury might not be linear. Similar phenomena happened in other fruits such as nectarine and apricot, that the near-freezing temperature was more beneficial in reducing chilling injury than medium-temperature[31-32].

The weight loss of the satsuma mandarin was increased with the storage time. The weight loss of the CFPS group was slightly higher than that of the 10 °C storage group (Fig. 2B), which might be due to a relative lower humidity in the −2 °C storage room, as the humidity become uncontrollable in sub-zero temperature environment. This result indicated that some necessary measures should be taken to prevent water loss of the fruit in the practical application of CFPS technology.

The CCI value of the fruit rind increased with storage time, with the rind color gradually transformed from light-yellow to red-orange. Compared to 10 °C storage group, the rind color change of CFPS group was slower, with the CCI value being lower and the

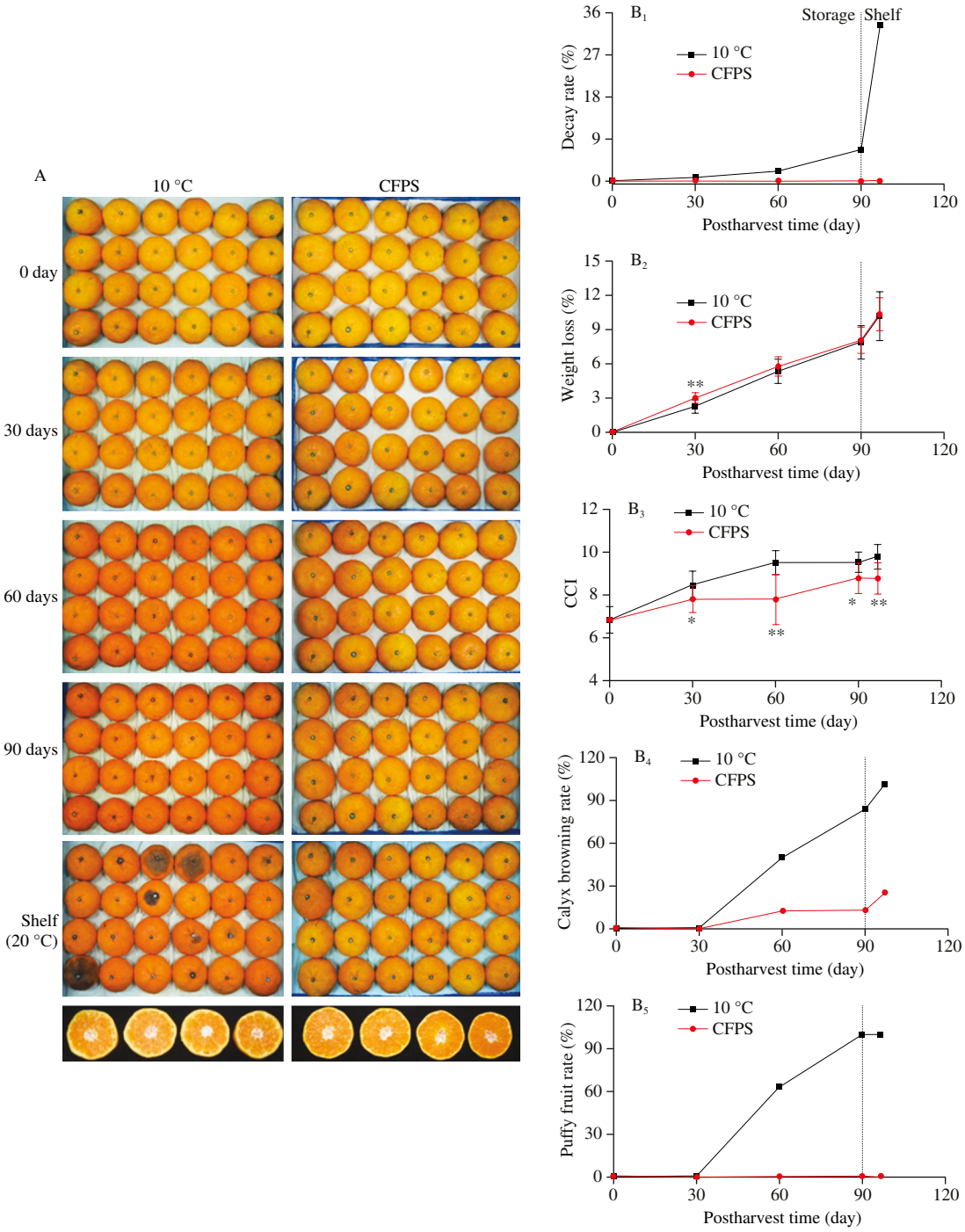

Fig. 2 Dynamic changes of appearance and general quality indexes of satsuma mandarin during storage and shelf period. (A) Appearance of the fruit; (B) General quality indexes. The weight loss (n = 24) and CCI value (n = 9) were shown as mean ± standard deviation. The decay rate, calyx browning rate and puffy fruit rate were shown as the proportion of fruits with relevant syndromes in a total of 150 fruits, Values marked with asterisks represented significant difference at $P < 0.05$ level (*) or $P < 0.01$ level (**) at each time point, the same below.

rind color being paler than that of the 10 °C storage group during the whole storage process and shelf life (Fig. 2). This is in according with the previous studies that the rind color of citrus fruit was highly sensitive to temperature[30,33-34], with the optimum temperature range for carotenoids biosynthesis and accumulation was between 15 and 25 °C[35]. Inappropriate high temperature 30 °C[35] or low temperature (−0.6 °C)[24] inhibited citrusrind color development.

Calyx senescence with the symptom of browning harmed the marketing of citrus fruit. In current citrus industry, auxin treatments were required to prevent the calyx senescence of mandarin fruit[10]. In present study, we found that the percentage of fruit with brown calyx increased with the elongation of storage time under 10 °C storage, with a rate of 83% in 90 days, and finally increased to 100% after 7 days of shelf life. CFPS effectively prevented calyx browning, with a rate of 25% in the 7 days of shelf life after 90 days' storage (Fig.2).

Fruit puffy was also one of the common quality deterioration phenomenon of satsuma mandarin, with the peel obviously detached from the pulp. After 60 days of storage, 63% of the fruit in the 10 °C

storage group was puffy, and the incidence further increased to 100% after 90 days of storage. While in the CFPS group, no obviously puffy fruit was found (Fig. 2).

Integrating the above characters into account, CFPS was beneficial in extending the storage period of satsuma fruit, by decreasing the decay rate, preventing the calyx browning and puffing of fruit. But it slightly increased the water loss and affected the rind color development.

3.3 The effects of CFPS on sensory quality

Sensory evaluation was carried out on the fruit with 90 days storage plus 7 days shelf life. The sensory attributes included sweetness, sourness, mandarin odor, off-flavor, gumminess, and juiciness. As shown in Fig. 3, the satsuma mandarin under CFPS and 10 °C storage shared similar sensory characteristics on sweetness, juiciness, gumminess and off-flavor degree. The fruit of CFPS group were slightly sourer than that of the 10 °C storage group. It is worth to mention that, CFPS technology was advantageous in maintaining the typical mandarin flavor than 10 °C storage (Fig. 3).

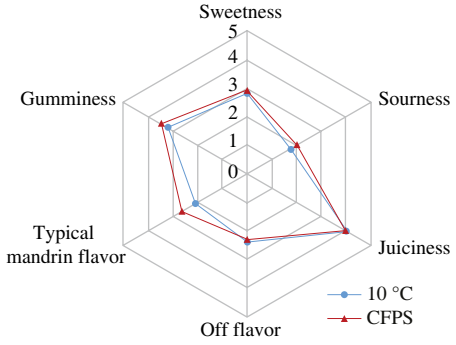

Fig. 3 Sensory evaluation of satsuma mandarin. Data was the mean value of 15 replicates.

3.4 The effects of CFPS on sugars and acids contents

Sucrose, fructose and glucose were the chief sugars components of satsuma mandarin. The sucrose content showed a trend of continuous increase with the elongation of storage time, and there was no significant difference between 10 °C storage and CFPS groups (Fig. 4A). In contrary, the contents of fructose and glucose slightly increased in the first month, and then decreased continuously in the next 2 months of storage and the following shelf life. CFPS significantly alleviated the decrease of fructose and glucose content, maintaining these two sugars in a relatively high level after 90 days storage (Figs. 4B and C).

Citric acid and malic acid were the chief acids components of satsuma mandarin. Citric acid, the major acids component, decreased during the 90 days storage at 10 °C (from 6.65 g/kg FW in the initial time to the 4.76 g/kg FW after 90 days of storage), and remained stable in the 7 days shelf life. CFPS significantly alleviated the citric acid decrease (Fig. 4D). The malic acid content was relatively stable during the storage and shelf life, and was less affected by the CFPS storage (Fig. 4E). Thus, the postharvest decrease of total acids contents was much slower in the CFPS group than that in the 10 °C group, which was in agree with the result of sensory evaluation shown in Fig. 3 that the fruit of CFPS group were slightly sourer than the 10 °C storage group. The higher acid content might also affect sweetness of fruit[36], this might explain that the CFPS group was not sweeter than the 10 °C storage group in spite of its higher sugar content.

The effects of above-zero temperature of 5−30 °C on the sugars and acids contents in citrus fruit during postharvest storage had been reported in plenty of previous studies. The higher temperature during storage, transportation and shelf life accelerated the decrease of acids[30,37]. The citric acid decreased more slowly under lower temperature[38], while malic acid was less sensitive to temperature than citric acid in the temperature range of 5−10 °C[4]. Our results showed that, under the sub-zero temperature, the decrease of citric acid content was prevented, while the malic acid contents were

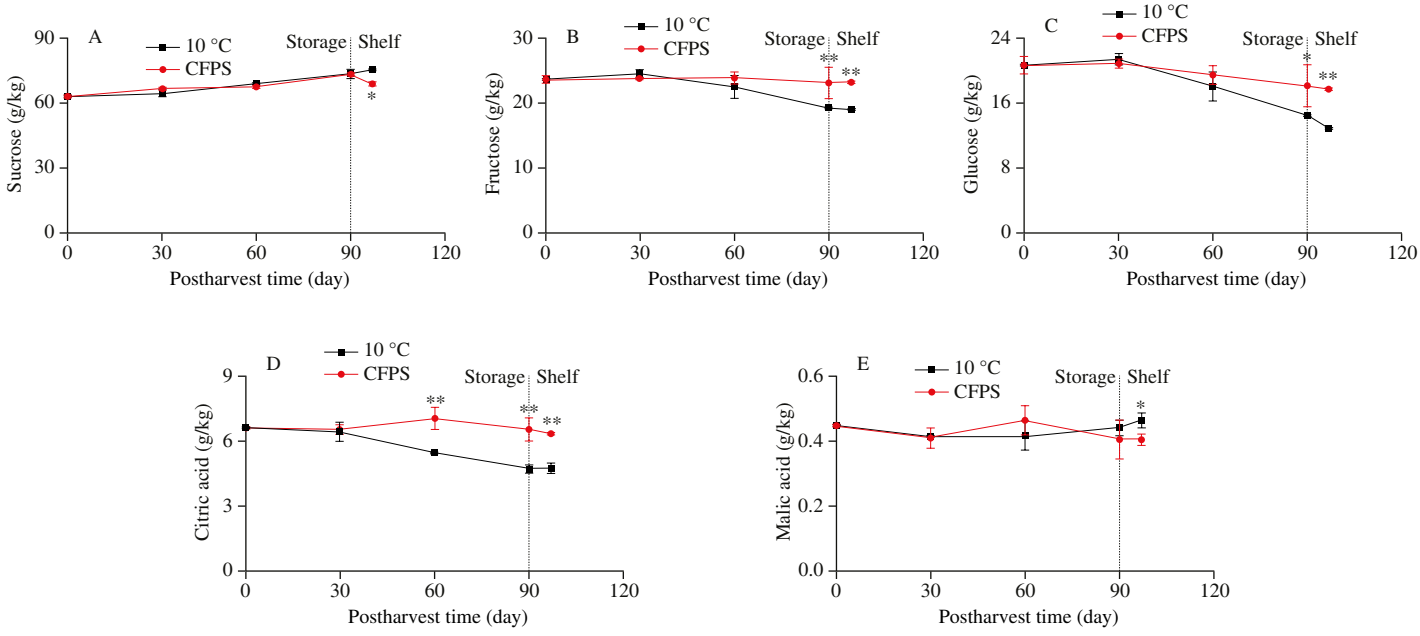

Fig. 4 Dynamic changes of sugars and acids content in the pulp. (A) Sucrose; (B) Fructose; (C) Glucose; (D) Citric acid; (E) Malic acid. The contents were expressed as g/kg on a fresh weight basis. Data were represented as mean ± standard deviation (n = 3).

hardly affected, which was well agreed with the previous results. In previous study[4], the differences of fructose, glucose and sucrose contents among satsuma mandarin stored in different temperatures were negligible in the temperature range of 5−30 °C during the 14 days' storage. In orange fruit, the glucose and fructose increased slightly while the sucrose fluctuated irregularly during the 60 days' storage at 6 °C [39]. Our results showed that, in the long-term storage of 90 days, the fructose and glucose decreased significantly after more than 30 days storage at 10 °C. While in the sub-zero temperature, the metabolism was slowed down, and thus the consumption of fructose and glucose was reduced due to the reduced energy requirement. The sucrose seemed to be insusceptible to the storage time and temperature, which was partially agreed with the previous study on citrus[4,39]. These results indicated that the sub-zero low temperature was beneficial in maintained the sugars and acids content of satsuma mandarin during long-term storage.

3.5 *The effects of CFPS on volatile profiles of the pulp*

A total of 51 volatiles were identified in the pulp, including 11 monoterpenes, 10 monoterpene derivates, 8 sesquiterpenes, 3 aldehydes, 2 aliphaticalcohols, 6 aromatics, 4 ketones, 3 esters, 2 hydrocarbons, as well as ethanol and acetaldehyde. *D*-Limonin was the chief volatile compound, accounting for 84.96% of the total volatiles content in the pulp of newly harvested fruit. *γ*-Terpinene was the second largest compound, accounting for 3.91% of the total volatile compounds in the pulp of newly harvested fruit (Fig. 5 and Table 1). These results were basically agreed with most of the citrus fruit reported in previous studies[12,40-41].

The total volatiles content in the pulp of the CFPS group increased in the first month of storage, then decreased during the next 2 months, and significantly increased again after transferring to the shelf at 20 °C, the general trend of which was similar to that of the 10 °C group. The difference is that, the increase of the total volatile contents of the 10 °C group in the shelf life was far higher than that of the CFPS group, with the total volatile content of the 10 °C storage group was about 2-fold that of the newly-harvested fruit (Fig. 5A). However, the total volatiles contents and composition of the CFPS group in the shelf life was seemed to be more close to that of the newly-harvested fruit (Fig. 5B). The monoterpenes were the chief volatile compounds, and showed a similar trend of dynamic change with that of the total volatiles, which rebounded after transferring to the shelf. The total contents of monoterpene derivatives significantly decreased during the storage, while becoming unrecoverable after transferring to the shelf (Fig. 5).

Basing on their dynamic change patterns, the volatile compounds can be generally clustered into 5 groups (Fig. 6). The compounds in pattern I increased during the first month of storage, and then decreased in the second and third month, and increased again after transferring to the shelf. In 10 °C storage group, all of the monoterpenes, 3 of the monoterpene derivatives, 1 aliphatic alcohol, 2 aliphatic aldehydes, and 1 aromatic compound showed such a dynamic change pattern. In the CFPS group, all of the monoterpenes, 3 of the monoterpene derivatives, 1 sesquiterpene, 2 aliphatic alcohols and 1 aromatic compound showed such a dynamic change pattern, while the compounds did not rise as sharply as those in the 10 °C storage group during shelf life.

The compounds in pattern II continuously increased during the storage and shelf life. Ethanol and acetaldehyde, which had been considered as the important compounds causing the off-flavor of citrus fruit[11,42], showed dynamic changes in such a pattern. It is worth to mention that, CFPS did not significantly alleviate the increase of ethanol and acetaldehyde. In the previous studies, low temperature storage (5 °C) had been found to be more effective on reducing the accumulation of off-flavor compounds such as ethanol and acetaldehyde than high temperature (20 °C)[5], while there was no significant difference among low storage temperatures (0, 4 and 8 °C)[43]. Our results were in agree with that of Obenland et al.[43]. It indicated that, in a storage temperature lower than 10 °C, storage time became the chief factor affecting the ethanol and acetaldehyde accumulation rather than storage temperature.

The compounds in pattern III increased during the first month of storage, and then decreased in the second and third month, and will not increase again after transferring to the shelf. The *α*-terpineol, perillaldehyde, citronellal and ethyl 4-etoxybenzoate showed such a change pattern. These 4 compounds were in trace amount or undetectable in the shelf life.

The compounds in pattern IV were stable during the 10 °C or CFPS storage, while increased after transferring to the shelf. (+)-Valencene and hexanal showed such a change pattern.

Two sesquiterpenes, *β*-elemene and *α*-caryophyllene, showed opposite trends of dynamic change, which were clustered as pattern V. In the 10 °C group, the compounds increase continuously during the whole storage and shelf period, while in the CFPS group, the compounds increased in the first month, then decreased in the next 2 months. After transferring to the shelf, the *α*-caryophyllene increased, while the *β*-elemene decreased.

Except for the compounds that were concluded in the referred patterns, there are some compounds specifically occurred at certain stages. Hexadecane appeared in the shelf life of both group, limonene

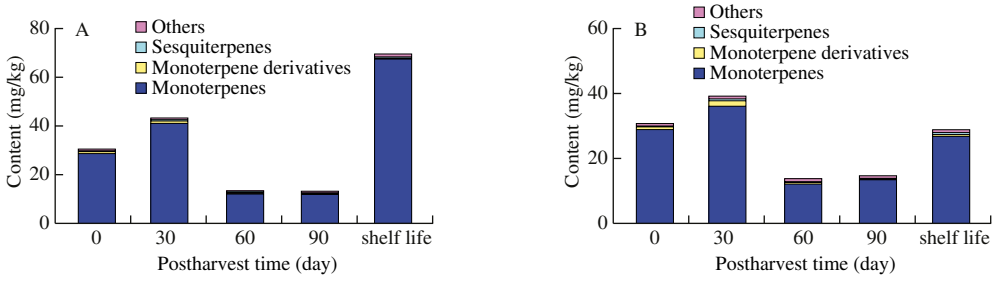

Fig. 5 Dynamic changes of different aroma volatiles classes during storage and shelf period. (A) 10 °C storage group; (B) CFPS group. The contents were expressed as mg/kg on a fresh weight basis.

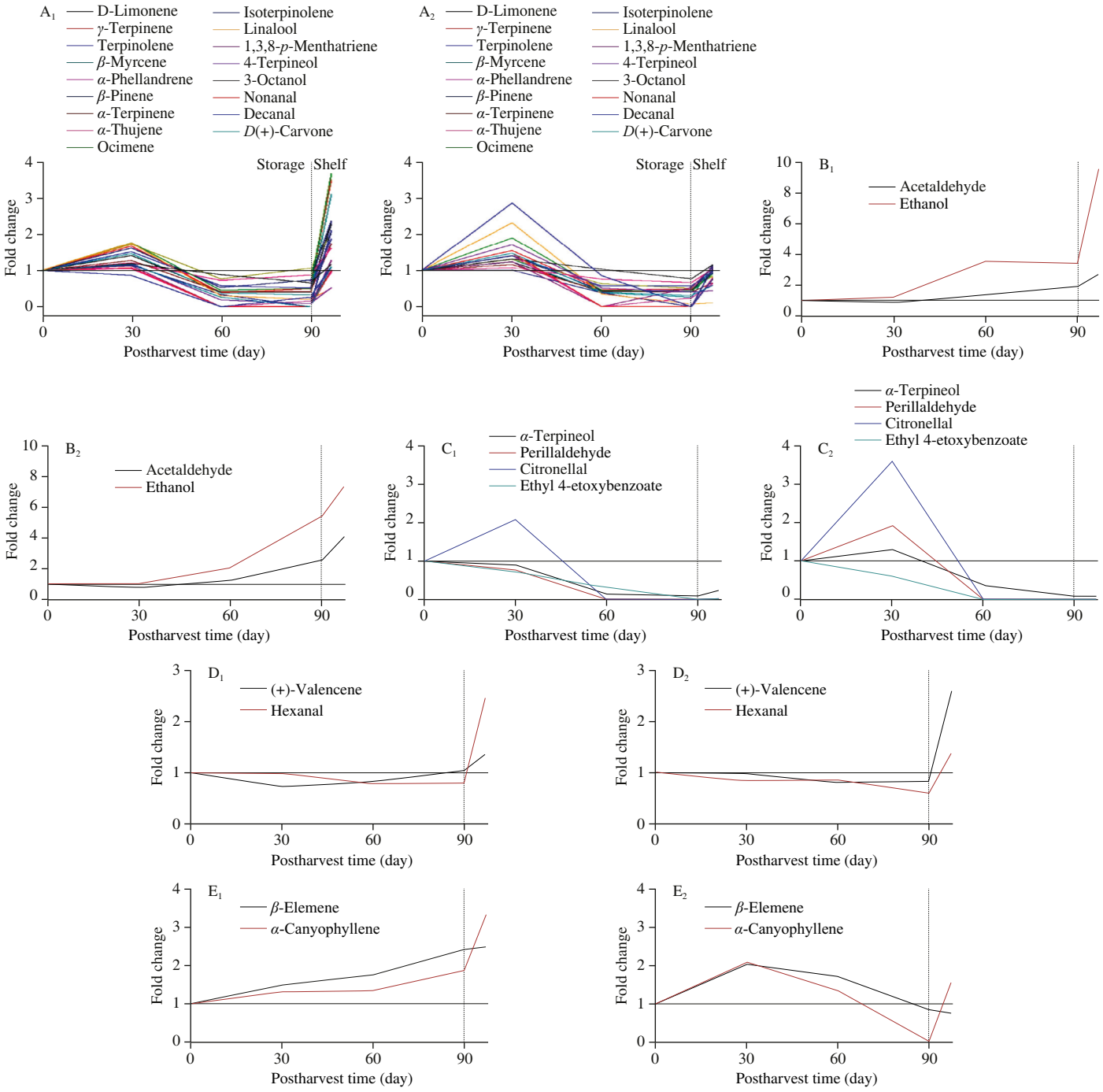

Fig. 6 Dynamic change patterns of aroma volatile compounds during storage and shelf period. A_1-E_1. the 10 °C storage group; A_2-E_2. the CFPS group. Data were represented as the fold change of contents to the newly-harvested fruit.

oxide appeared in the shelf life of 10 °C storage group, *trans*-*p*-mentha-2,8-dienol and *cis*-isolimonenol can be detected only in the shelf life of CFPS group. *p*-Cymene existed only in the storage period of 60 and 90 days. In addition, there were compounds existed in the harvested fruit, then disappeared in the storage, and existed again in the shelf life, such as benzaldehyde (Table 1).

There had been several studies on the changes of characteristic aroma volatiles of citrus fruit under different temperatures. In the mandarin with 4 weeks' cold storage at 2−8 °C following by 3 days at 20 °C, the valencene was increased, and the higher storage temperature led to the higher increase. In contrary, several other compounds, including α-cubebene, δ-cadinene, linalool and perillaldehyde, were decreased[44]. Obenland et al.[43] tracked the dynamic changes of volatile compounds in mandarin fruit under 5−8 °C for 7 weeks following by 1 week at 20 °C, and found that the volatile compounds including β-myrcene, octanal, α-terpinene, *p*-cymene, β-ocimene, γ-terpinene, terpinolene, 1,3,8-para-menthatriene, 4-terpineol, carvone and perillaldehyde showed a slight increase or kept stable in the first week of storage, and then decreased during the following storage period. In another study, Obenland et al.[5] also found that α-terpinene, limonene, (*E*)-β-ocimene, γ-terpinene and terpinolene had a trend of increase during the initial 4 weeks of

Table 1
Contents of volatile compoundsfrom the pulp of satsuma fruit.

Compounds	0 day	10 °C				CFPS			
		30 days	60 days	90 days	Shelf life	30 days	60 days	90 days	Shelf life
Ethanol fermentation									
Acetaldehyde	0.027±0.012^{c}	0.024 ± 0.006^{c}	0.035 ± 0.007^{c}	0.052 ± 0.019^{b}	0.073 ± 0.005^{b}	0.021 ± 0.007^{c}	0.034 ± 0.017^{c}	0.070 ± 0.020^{b}	0.111 ± 0.006^{a}
Ethanol	0.228 ± 0.104^{b}	0.282 ± 0.106^{b}	0.817 ± 0.399^{b}	0.773 ± 0.100^{b}	2.185 ± 0.502^{a}	0.233 ± 0.115^{b}	0.469 ± 0.325^{b}	1.248 ± 0.323^{ab}	1.686 ± 0.527^{a}
Monoterpenes									
D-Limonene	25.936 ± 1.963^{c}	36.682 ± 4.039^{b}	10.844 ± 2.612^{d}	10.443 ± 1.030^{d}	59.794 ± 18.210^{a}	31.944 ± 2.376^{bc}	10.825 ± 0.961^{d}	12.023 ± 3.401^{d}	24.090 ± 4.683^{c}
γ-Terpinene	1.195 ± 0.032^{c}	2.002 ± 0.329^{ab}	0.465 ± 0.121^{d}	0.588 ± 0.076^{d}	4.192 ± 1.601^{a}	1.847 ± 0.172^{b}	0.563 ± 0.074^{d}	0.580 ± 0.190^{d}	1.180 ± 0.286^{c}
Terpinolene	0.360 ± 0.032^{b}	0.545 ± 0.053^{a}	0.209 ± 0.027^{c}	0.186 ± 0.018^{c}	0.676 ± 0.178^{a}	0.506 ± 0.026^{a}	0.207 ± 0.020^{c}	0.208 ± 0.030^{c}	0.338 ± 0.041^{b}
β-Myrcene	0.448 ± 0.012^{c}	0.654 ± 0.071^{b}	0.173 ± 0.044^{d}	0.148 ± 0.016^{de}	1.397 ± 0.570^{a}	0.583 ± 0.059^{bc}	0.170 ± 0.018^{d}	0.119 ± 0.036^{e}	0.505 ± 0.121^{bc}
α-Phellandrene	0.284 ± 0.013^{b}	0.301 ± 0.006^{b}	nd	0.045 ± 0.002^{d}	0.269 ± 0.058^{b}	0.400 ± 0.002^{a}	nd	0.070 ± 0.028^{d}	0.181 ± 0.011^{c}
α-Pinene	0.153 ± 0.025^{cd}	0.265 ± 0.014^{a}	0.119 ± 0.009^{d}	0.163 ± 0.010^{c}	0.312 ± 0.070^{a}	0.201 ± 0.012^{b}	0.098 ± 0.007^{e}	0.078 ± 0.030^{e}	0.130 ± 0.026^{d}
β-Pinene	0.175 ± 0.028^{bc}	0.284 ± 0.020^{b}	0.092 ± 0.020^{cd}	0.128 ± 0.011^{c}	0.416 ± 0.110^{a}	0.247 ± 0.021^{b}	0.078 ± 0.003^{d}	0.072 ± 0.026^{d}	0.201 ± 0.023^{b}
α-Terpinene	0.119 ± 0.016^{bc}	0.152 ± 0.013^{ab}	0.049 ± 0.008^{d}	0.050 ± 0.004^{cd}	0.208 ± 0.054^{a}	0.138 ± 0.012^{b}	0.051 ± 0.006^{c}	0.048 ± 0.009^{c}	0.079 ± 0.013^{c}
α-Thujene	0.090 ± 0.006^{bc}	0.109 ± 0.006^{ab}	0.066 ± 0.003^{cd}	0.079 ± 0.005^{c}	0.146 ± 0.031^{a}	0.097 ± 0.010^{b}	0.069 ± 0.004^{cd}	0.060 ± 0.008^{d}	0.094 ± 0.022^{bc}
Ocimene	0.019 ± 0.000^{b}	0.033 ± 0.006^{ab}	0.008 ± 0.002^{c}	0.009 ± 0.001^{c}	0.068 ± 0.030^{a}	0.036 ± 0.004^{ab}	0.008 ± 0.001^{c}	0.008 ± 0.000^{c}	0.017 ± 0.004^{b}
Isoterpinolene	0.020 ± 0.006^{ab}	0.023 ± 0.002^{ab}	0.007 ± 0.001^{c}	nd	0.043 ± 0.023^{a}	0.020 ± 0.001^{ab}	0.008 ± 0.000^{c}	nd	0.022 ± 0.012^{ab}
Monoterpene derivatives									
Linalool	0.595 ± 0.017^{b}	1.049 ± 0.126^{a}	0.184 ± 0.053^{c}	0.121 ± 0.026^{cd}	0.304 ± 0.108^{bc}	1.386 ± 0.193^{a}	0.206 ± 0.029^{c}	0.046 ± 0.016^{e}	0.065 ± 0.009^{de}
1,3,8-*p*-Menthatriene	0.016 ± 0.002^{a}	0.018 ± 0.001^{a}	nd	nd	0.021 ± 0.007^{a}	0.020 ± 0.000^{a}	nd	0.008 ± 0.001^{b}	0.018 ± 0.002^{a}
trans-*p*-Mentha-2,8-dienol	nd	nd	nd	nd	nd	nd	nd	nd	0.031 ± 0.004^{a}
cis-Isolimonenol	nd	nd	nd	nd	nd	nd	nd	nd	0.026 ± 0.003^{a}
Limonene oxide	nd	nd	nd	nd	0.024 ± 0.0143^{a}	nd	nd	nd	nd
4-Terpineol	0.076 ± 0.001^{b}	0.091 ± 0.009^{ab}	0.015 ± 0.006^{d}	0.007 ± 0.000^{d}	0.040 ± 0.014^{cd}	0.132 ± 0.025^{a}	0.040 ± 0.007^{cd}	0.031 ± 0.003^{d}	0.034 ± 0.002^{d}
α-Terpineol	0.087 ± 0.004^{b}	0.078 ± 0.004^{b}	0.013 ± 0.004^{d}	0.007 ± 0.000^{e}	0.021 ± 0.004^{c}	0.113 ± 0.033^{a}	0.032 ± 0.012^{c}	0.008 ± 0.002^{e}	0.007 ± 0.001^{e}
cis-Carveol	nd	0.013 ± 0.002^{b}	nd	nd	0.027 ± 0.012^{a}	0.027 ± 0.004^{a}	nd	nd	0.021 ± 0.003^{a}
Perillaldehyde	0.028 ± 0.002^{b}	0.021 ± 0.002^{b}	nd	nd	nd	0.053 ± 0.009^{a}	nd	nd	nd
Citronellal	0.010 ± 0.001^{b}	0.021 ± 0.004^{ab}	nd	nd	nd	0.037 ± 0.006^{a}	nd	nd	nd
Sesquiterpenes									
(+)-Valencene	0.199 ± 0.006^{b}	0.145 ± 0.019^{d}	0.164 ± 0.016^{cd}	0.206 ± 0.025^{b}	0.271 ± 0.087^{b}	0.197 ± 0.034^{bc}	0.161 ± 0.013^{cd}	0.167 ± 0.014^{c}	0.519 ± 0.078^{a}
δ-Elemene	nd	0.008 ± 0.000^{a}	0.010 ± 0.003^{a}	0.013 ± 0.003^{a}	nd	0.011 ± 0.000^{a}	0.008 ± 0.001^{a}	nd	nd
α-Cubebene	nd	nd	nd	nd	nd	0.021 ± 0.003^{a}	nd	nd	nd
β-Elemene	0.049 ± 0.006^{b}	0.073 ± 0.014^{ab}	0.087 ± 0.019^{ab}	0.119 ± 0.010^{a}	0.123 ± 0.077^{a}	0.100 ± 0.028^{ab}	0.083 ± 0.016^{ab}	0.042 ± 0.021^{b}	0.036 ± 0.009^{b}
α-Caryophyllene	0.007 ± 0.000^{c}	0.009 ± 0.001^{bc}	0.010 ± 0.002^{bc}	0.014 ± 0.001^{b}	0.024 ± 0.016^{a}	0.015 ± 0.001^{ab}	0.010 ± 0.002^{bc}	nd	0.011 ± 0.002^{b}
Alloaromadendren	nd	nd	0.010 ± 0.004^{bc}	0.012 ± 0.006^{b}	nd	0.012 ± 0.002^{b}	0.007 ± 0.001^{c}	nd	0.025 ± 0.006^{a}
(−)-*α*-Panasinsen	nd	nd	0.007 ± 0.001^{b}	0.010 ± 0.002^{b}	0.019 ± 0.008^{ab}	nd	nd	nd	0.022 ± 0.004^{a}
δ-Cadinene	nd	nd	0.012 ± 0.002^{b}	0.014 ± 0.001^{b}	nd	0.019 ± 0.001^{a}	0.011 ± 0.001^{b}	nd	nd
Aliphatic alcohol									
3-Octanol	0.179 ± 0.016^{b}	0.212 ± 0.008^{a}	0.159 ± 0.019^{b}	0.120 ± 0.005^{c}	0.210 ± 0.0606^{a}	0.235 ± 0.018^{a}	0.184 ± 0.009^{ab}	0.138 ± 0.009^{bc}	0.208 ± 0.008^{a}
1-Octanol	0.070 ± 0.007^{ab}	0.068 ± 0.002^{ab}	0.051 ± 0.003^{c}	0.038 ± 0.002^{d}	0.065 ± 0.008^{bc}	0.083 ± 0.004^{a}	0.061 ± 0.004^{a}	0.049 ± 0.002^{c}	0.065 ± 0.003^{b}
Aliphatic aldehyde									
Nonanal	0.060 ± 0.011^{a}	0.064 ± 0.004^{a}	nd	nd	0.060 ± 0.010^{a}	0.088 ± 0.002^{a}	nd	nd	0.048 ± 0.007^{a}
Decanal	0.022 ± 0.002^{bc}	0.019 ± 0.001^{c}	nd	0.006 ± 0.000^{d}	0.025 ± 0.007^{bc}	0.064 ± 0.009^{a}	0.019 ± 0.001^{c}	nd	0.017 ± 0.002^{c}
Hexanal	0.146 ± 0.015^{c}	0.143 ± 0.007^{c}	0.114 ± 0.009^{cd}	0.115 ± 0.005^{cd}	0.359 ± 0.038^{a}	0.124 ± 0.006^{cd}	0.126 ± 0.006^{cd}	0.087 ± 0.003^{d}	0.203 ± 0.014^{b}
Aromatic									
p-Cymene	nd	nd	0.394 ± 0.002^{b}	0.424 ± 0.020^{b}	nd	nd	0.545 ± 0.071^{a}	0.523 ± 0.034^{a}	nd
Acetophenone	nd	0.010 ± 0.000^{a}	nd	nd	nd	0.012 ± 0.001^{a}	0.008 ± 0.000^{b}	nd	0.012 ± 0.002^{a}
D(+)-Carvone	0.046 ± 0.003^{b}	0.054 ± 0.002^{b}	0.012 ± 0.003^{d}	nd	0.052 ± 0.029^{b}	0.069 ± 0.003^{a}	0.019 ± 0.002^{d}	0.014 ± 0.001^{d}	0.028 ± 0.008^{c}
3-Methyl-4-isopropylphenol	nd	nd	nd	nd	nd	0.017 ± 0.002^{a}	nd	nd	nd
Benzaldehyde	0.024 ± 0.002^{b}	nd	nd	nd	0.350 ± 0.131^{a}	nd	nd	nd	0.351 ± 0.016^{a}
4-Acetyl-1-methylcyclohexene	0.013 ± 0.001^{b}	nd	nd	nd	nd	0.017 ± 0.001^{a}	nd	nd	nd
Ketones									
2-Undecanone	0.044 ± 0.009^{a}	nd	nd	nd	nd	nd	nd	nd	nd
Geranylacetone	0.007 ± 0.002^{b}	0.011 ± 0.001^{a}	nd	nd	nd	0.011 ± 0.001^{a}	nd	nd	nd
2-Methyl-5-(1-methylethenyl)-cyclohexanone	nd	nd	nd	nd	nd	0.012 ± 0.001	nd	nd	nd
Methylheptenone	nd	nd	0.010 ± 0.001^{c}	0.007 ± 0.000^{d}	nd	0.019 ± 0.001^{a}	0.011 ± 0.000^{c}	nd	0.015 ± 0.001^{b}
Esters									
3,7-Dimethyl-6-octen-1-o butyrate	nd	nd	nd	nd	nd	0.008 ± 0.000^{a}	nd	nd	nd
Ethyl 4-etoxybenzoate	0.047 ± 0.009^{a}	0.034 ± 0.004^{ab}	0.014 ± 0.002^{c}	nd	nd	0.029 ± 0.002^{b}	nd	nd	nd
Phthalic acid, isobutyl nonyl ester	nd	0.005 ± 0.000^{a}	nd	nd	nd	0.005 ± 0.000^{a}	nd	nd	nd
Hydrocarbon									
Hexadecane	nd	nd	nd	nd	0.018 ± 0.001^{a}	nd	nd	nd	0.010 ± 0.000^{b}
Cyclohexane, 1-Methyl-4-(1-methylethenyl)-,*cis*-	nd	0.023 ± 0.002^{a}	nd	nd	nd	nd	nd	nd	nd

Note: nd, not detected. The contents were expressed as mg/kg on a fresh weight basis; Data was represented as mean ± standard deviation (n = 3). The different letter in same line indicated significant difference (P < 0.05).

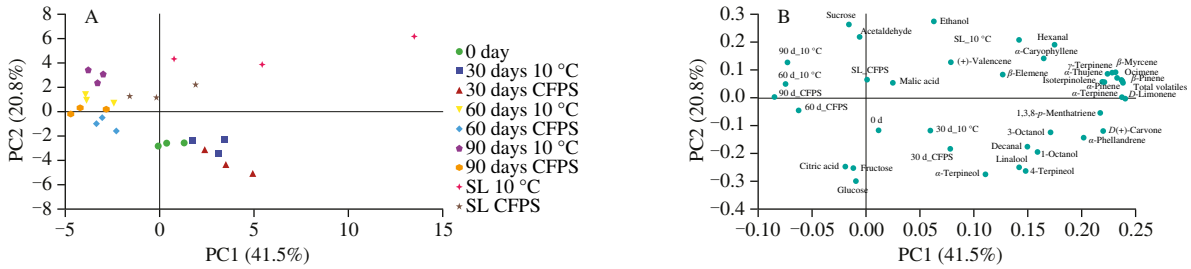

Fig. 7 PCA analyses of volatiles. (A) Scores plot; (B) Biplot. SL, shelf-life.

storage at 5 °C, and then decreased. The content of these compounds slightly rose after transferring to 20 °C. These referred studies indicated that long-term storage under low temperature might lead to the loss of aroma compounds in mandarin, some of which could be recovered in different degrees after transferring to higher temperature, while some of which were unrecoverable. Moreover, the situation varied depending on cultivars and storage temperatures, with some cultivars being more sensitive to low temperature on the aroma changes[44-45]. In the present study on the long-term storage (90 days) of satsuma mandarin, we found that most of the volatile compounds (especially the terpenes) in the pulp showed dynamic change rules as pattern I, which were decreased after long-term storage at low temperature but recovered after transferring to 20 °C shelf. The total volatiles contents of CFPS group in the shelf life wasmore close to that of the newly-harvested fruit compared with 10 °C storage group. This result was in consistent with the results of sensory evaluation, that the CFPS group showed better typical mandarin flavor than the 10 °C storage group (Fig. 3). Thus, it was indicating that the CFPS technology was beneficial in maintaining the flavor quality of satsuma mandarin in the long-term storage.

3.6 General effects of CFPS on flavor characteristics by PCA

PCA was applied to evaluate the general effects of CFPS technology on the flavor characteristics of satsuma mandarin during long-term storage. The sugars, organic acids, and volatiles compounds that was detectable in more than 50% of the samples during the whole storage period were applied for the PCA. Fig. 7 showed the score biplot and loading of the first two principle components. The results confirmed that the flavor quality indicators were separated between the CFPS and 10 °C storage groups in the long-term storage of more than 60 days and shelf-life (Fig. 7A). The 10 °C storage group in the shelf-life after 90 days storage was separated from other groups and located in the positive side of the PC1 and PC2, characterizing with the higher amounts of total volatiles and monoterpenes, which had very high positive loading of PC1, and also characterizing with lower amount of fructose, glucose and citric acid, which had negative loading of PC1. CFPS group in the shelf-life after 90 days storage was close to the newly harvested samples (0 day) in PC1 but was separated in PC2, characterizing with the reduced *D*(+)-carvone, *α*-phellandrene, linalool, 4-terpineol, *α*-terpineol and glucosecontents, as well as increased ethanol and acetaldehyde contents compared to the newly harvested fruit (Fig. 7B).

4. Conclusion

The freezing point and the supercooling point of satsuma mandarin (TSS content of (11.80 ± 1.07)%) were determined to be −3 and −6.6 °C respectively. A CFPS technology was developed for the long-term storage of satsuma mandarin, with the storage temperature being controlled at −2 °C. CFPS technology almost completely prevented fruit decay of satsuma mandarin in the 90 days storage and the following 7 days shelf life, without causing chilling injury of the fruit. In addition, CFPS technology was superior to 10 °C storage (RH 85%−90%) in maintaining the appearance and flavor of satsuma mandarin, during the tested period. Comprehensively, CFPS was applicable in reducing the postharvest loss and quality deterioration of satsuma mandarin in the long-term storage.

Declaration of interest

Authors declare no conflict of interest.

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Authors contribution

Chongde Sun conceived and designed the study; Qing Jiang and Jinping Cao performed mostexperiments, with help from Yezhi Chen, Shuting Xu, and Jue Wu; Jinping Cao and Qing Jiang analysed the data; Jinping Cao drafted the manuscript; Yue Wang and Chongde Sun revised the manuscript.

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