Journal of Food Composition and Analysis 116 (2023) 105072

Contents lists available at ScienceDirect

Journal of Food Composition and Analysis

journal homepage: www.elsevier.com/locate/jfca

Key aroma-active compounds identification of *Ziziphus jujuba cv.* Huizao: Effect of pilot scale freeze-drying

Min Gou [a,c], Qinqin Chen [a,*], Yening Qiao [a,c], Xinwen Jin [d], Jingjian Zhang [b], Hui Yang [d], Marie-Laure Fauconnier [c], Jinfeng Bi [a,*]

[a] *Institute of Food Science and Technology, Chinese Academy of Agricultural Sciences (CAAS)/ Key Laboratory of Agro-Products Processing, Ministry of Agriculture and Rural Affairs, 100193 Beijing, China*

[b] *CangZhou Academy of Agriculture and Forestry Sciences, 061001 Cangzhou, China*

[c] *Laboratory of Chemistry of Natural Molecules, Gembloux Agro-Bio Tech, University of Liége, 5030 Gembloux, Belgium*

[d] *Institute of Agro-products Processing Science and Technology, XinJiang Academy of Agricultural and Reclamation Science, 832000 Shihezi, China*

ARTICLE INFO

Keywords:
Red jujube
GC-O-MS
Omission tests
Odor activity values
Detection frequency

ABSTRACT

Aroma profile and key aroma-active of red jujube subjected to pilot scale freeze drying (FD) were investigated based on molecular sensory science. After FD, 41 aroma compounds were identified through gas chromatography-olfactometry-mass spectrometry (GC-O-MS) and quantified through calibration curves. The total aroma compounds content was decreased 26.71% compared with raw red jujube, of which ketones and acids contents were decreased 63.33% and 62.88%, while, the esters, lactones and alcohols contents were increased 34.10%, 8.52% and 480.17%, respectively. Through the GC-O-MS, odor active values combined with recombination and omission tests, 14 key aroma-active compounds were identification in freeze-dried red jujube. In which, 2-ethyl-3,5-dimethyl-pyrazine had the highest OAV (2687.00) and dominated the roast note of aroma profile. In addition, ethyl heptanoate, hexyl acetate, 2,3-butanediol, ethyl dodecanoate and 5-heptyloxolan-2-one were newly identified as the key aroma-active compounds in freeze-dried red jujube.

1. Introduction

Jujube (*Zizyphus jujuba* Mill.) is a plant of the family Rhamnaceae and originated in China with a long history of more than 4000 years (Chen et al., 2018). As a characteristic resource of homology of medicine and food in China, it has high biological activities, such as sedation, tranquilization, blood enrichment, brain strengthening, and anti-cancer (Choi et al., 2012). In addition, it has a pleasant aroma, which is one of the important factors to attract consumers and directly affects its commodity value. *Ziziphus jujuba cv.* Huizao is one of the most popular cultivars, which is mainly used for drying. Freeze drying (FD) is a drying method with direct dehydration of frozen materials under reduced pressure. This process could better retain the color, shape, texture and nutritional of raw materials, and it is more and more widely applied in the food production, such as, fruit and vegetable crisps, coffee powder and yogurt cubs (An et al., 2016; Xu et al., 2021).

Aroma is an important characteristic of dried fruit products, however, most freeze-dried products showed different degrees of aroma loss. The changes of aroma compounds in freeze-dried process of different materials have attracted the attention of many researchers. To date, Song et al. (2020) studied the volatile compounds in red jujube subjected to different drying processes, the aldehydes compounds in red jujube were no longer detected after FD. Chin et al. (2008) studied the changes in aroma of durian after FD for 12 h, the amount of major aroma decreased dramatically, ranging from 71% to 97%, and most of them were esters. Rajkumar et al. (2017a, 2017b) compared the volatile components between fresh cabbage and the freeze-dried one, found aldehydes, esters, alcohols, and ketones had a certain degrees of loss. Similar aroma loss were also observed in freeze-dried tomato (Jeyaprakash et al., 2020), garlic (Feng et al., 2021), golden pompano (Zhang et al., 2019), banana (Mui et al., 2002) and bread crumbs (Dimelow et al., 2005), the average loss ratio of volatile compounds of freeze-dried banana slices and carvone in bread crumbs was 37.5% and 55%, respectively. The causes of aroma loss are complex in freeze-dried process, especially in the last stage of FD, the temperature of the material would be close to the heating plate, and the volatile compounds might be transformed or migrated. Else, some volatile compounds, which have the low vapor pressure, would be combined with the sublimated water

* Corresponding authors.
E-mail addresses: celerylc@163.com (Q. Chen), bjfcaas@126.com (J. Bi).

https://doi.org/10.1016/j.jfca.2022.105072
Received 12 October 2022; Received in revised form 27 November 2022; Accepted 28 November 2022
Available online 30 November 2022

vapor in the cold trap of the freeze dryer. Some volatile compounds condensed and discharged by the vacuum pump, so the change of the volatile compounds of the freeze-dried products is a very complicated physical and chemical process, which is closely related to the drying process condition (Yan et al., 2019).

However, lower constant freeze-drying temperature (less than 30 °C) and experimental FD machine were usually used in the published literature, which showed significant difference with the one used in the industry. To improve productivity and reduce energy consumption, multi-stage and variable-temperature procedure was used in industry with a higher temperature of heating plate (85–65 °C) to provide higher latent heat for the sublimation of water in the material, and accelerate the FD rate and shorten the FD time. In this process, the temperature of the material will gradually increase from the freezing temperature (−40 °C) to the heating plate temperature (65 °C). In the later desorption drying stage, chemical reactions will occur to enhance the aroma of products, which might offset the aroma loss in experimental FD. Fortunately, drying conditions used in the industry FD could be well simulated by pilot scale FD, which could be used for better understanding aroma change of jujube during multi-stage and variable-temperature procedure.

To understand how the jujube aroma was modified after FD, the effect of pilot scale FD on the aroma profile of red jujube was first analyzed, and key aroma-active compounds were further identified based on molecular sensory science and technology. It could provide theoretical basis for aroma quality improvement during FD process.

2. Materials and methods

2.1. Materials and chemicals

Red jujubes (*Zizyphus jujuba* cv. Huizao) were harvested from a local farm orchard in Akesu, Xinjiang, China, in November 2020. The red jujubes were selected and transported to Beijing within 2 days. The red jujubes without any damage and incubated at 4 °C controlled atmosphere storage room until used. The water content of "Huizao" was 25.57%, the pH was 5.5, and the solid soluble content was 69.0%.

Oct-1-en-3-ol, 2,3-butanediol, hexanal, (*E*)− 2-hexenal, nonanal, furfural, 2-octenal, decanal, benzaldehyde, butane-2,3-dione, 3-octanone, 3-hydroxybutan-2-one, oct-1-en-3-one, 6-methyl-5-hepten-2-one, 3-oxobutan-2-yl acetate, undecan-2-one, acetic acid, 3-methyl-butanoic acid, pentanoic acid, (*E*)-but-2-enoic acid, hexanoic acid, heptanoic acid, (*E*)− 2-hexenoic acid, octanoic acid, methyl hexanoate, hexyl acetate, ethyl heptanoate, methyl decanoate, ethyl decanoate, methyl benzoate, methyl dodecanoate, ethyl dodecanoate, hexamethyl decanoate, oxolan-2-one, 5-ethyloxolan-2-one, 5-propyloxolan-2-one, 5-butyloxolan-2-one, 5-heptyloxolan-2-one, limonene, γ-terpinene, styrene, α-farnesene, 2-pentyl-furan, *p*-cymene, naphthalene, 2-ethyl-3,5-dimethyl-pyrazine, 2,6-dimethyl-pyrazine, tetramethyl-pyrazine and 2-ethyl-6-methyl-pyrazine were purchased from Yuanye Bio-Technology (Shanghai Yuanye Bio-Technology Co., Ltd) or Macklin (Shanghai Macklin Biochemical Co., Ltd). All of the chemical standards used above with purity $\geq$ 99%.

2.2. Freeze drying (FD) treatment

Briefly, the kernel of jujubes was removed and the remaining part was cut into 5 mm slices; then 500 g jujube slices soaked in 80 °C water for 1 min to keep a relatively flat surface before freeze drying. Jujube slices were drained in a colander and put into − 40 °C refrigerator for 48 h. A pilot scale freeze dryer (Advantech Co., Ltd. China) was used with drying conditions was as follows: The cold trap temperature and vacuum pressure were − 30 °C and 60 Pa, respectively; the drying temperature of the heating plate was from room temperature to 85 °C within 45 min and kept for 3 h, then decreased to 70 °C within 30 min and maintained for 5 h, and finally decrease to 65 °C within 30 min and kept for 1 h. The sample tray was in the middle of two heating plates and not directly connected, the diagram was shown in Fig. S1. At the same time, the temperature of jujube slices was monitored online through the temperature probe which equipped in FD machine (Fig. S2).

2.3. Extraction volatile compounds from jujube by headspace solid-phase microextraction (HS-SPME)

HS-SPME was carried out according to Gou et al., (2022) and Qiao et al., (2021). Briefly, the kernel of jujubes was removed and the remaining part was cut into 5 mm slices; then crushed using a Joyoung pulverizer (JYL-CO20, Joyoung Co., Ltd., Shandong, China). The 2 g of the sample were placed in 20-mL headspace vials, and 2 μL of 2-cyclohexen-1-one (1 mg/mL) and 0.1 g NaCl in 0.5 mL of distilled water was added, followed by incubation at 50 °C for 40 min to reach equilibrium. A resolved SPME fiber (polydimethylsiloxane-divinylbenzene (PDMS/DVB), 2 cm, 65 μm; Supelco, Inc., Bellefonte, PA, USA) was placed in the sample headspace, and adsorption was carried out for 30 min at 50 °C. After extraction, the SPME fiber was withdrawn and directly insert into the GC injector for desorbing at 250 °C for 3 min. The experiment was repeated at least three times independently for each sample.

2.4. Gas chromatography-olfactometry-mass spectrometry (GC-O-MS) analysis

2.4.1. Detection of volatile compounds

The volatile compounds of the sample were identified by GC-MS (7890B GC System, 5977 A MSD) equipped with sniffing port (Sniffer 9000, Brechbühler, Schlieren, Switzerland) and a DB-Wax column (60 m × 0.25 mm, 0.25 μm). The temperature programs were designed as follows: the column temperature was held 40 °C for 3 min, heated to 90 °C at 7 °C /min, increased to 120 °C at 4 °C /min, then rose to 170 °C at 5 °C/min, thereafter, ramped to 200 °C at 4 °C/min, and held for 8 min. The helium carrier gas (purity = 99.99%) was input at a constant flow rate of 1.0 mL/min. The ionization method was Electron-impact (EI), and the fragments created by EI were scanned from 35 to 550 *m/z*.

A sniffing port (Sniffer 9000) coupled to the GC-MS instrument was used to discriminate the aroma-active compounds in the HS-SPME isolates. At the end of the capillary column, the effluent between the olfactometers (Sniffer 9000, Brechbühler, Schlieren, Switzerland) and the MS detector was split at ratios of 1:1, of which one was delivered to the sniffing port (280 °C), and the other was delivered to the MS.

2.4.2. Qualification of volatile compounds

The aroma compounds were identified by comparing the NIST17 library of the MS and were confirmed by the linear retention indices (LRI), odor qualities and authentic standards. A total of 3 trained sensory evaluators were selected to smell the aroma compounds in the samples by olfactometry. These professionals had outstanding sensory organs, no bad habits, and no allergies. The evaluators recorded the odor characteristics and detection frequency (DF) of the aroma compounds in the samples. DF analysis was repeated twice for every panelist. Odorant with DF $\geq$ 2 (reported by at least two assessors) could be considered as having aroma potential activity. Authentic flavor standards were also used to confirm the volatile compounds as external references under same GC-O-MS conditions.

2.4.3. Quantification of volatile compounds

All aroma compounds were quantified by calibration curves constructed with authentic flavor standards in the odorless matrix. To obtain an odorless matrix, the aroma compounds of samples were removed by a mixture of ether and pentane (v:v = 1:1). Subsequently, the samples were dried at 20 °C for 48 h using a freeze dryer (Alpha 1–4 LD plus, Marin Christ, Osterode, Germany) until nothing was detected by GC-O-MS (Liu et al., 2020).

The extraction method and GC-MS condition of standard volatiles for preparation of standard curves as same as described for the analysis above mentioned. The standard curves for different compounds ($R^2 >$ 0.99) were established. The calculation of each identified compound was according to the standard curve and results were shown in Table S1.

2.5. Odor activity value (OAV)

To obtain information of the importance of a single compound in the aroma profile of red jujube, the OAV was calculated, which is the ratio of the concentration of each compound to its orthonasal detection odor threshold. The threshold values of volatile compounds in water referred to the literature (Gemert, 2011).

2.6. Aroma recombination and omission experiments

To identify the key aroma-active compounds based on GC-O-MS and OAV results, recombination and omission were carried out. Each omission sample was evaluated against complete recombination model prepared by mixing the standard aroma compounds at the concentrations in freeze-dried jujube (recombination model 1). If there is a significant difference, it means that the missing aroma component is the key aroma-active compound of sample. On this basis, the key aroma-active compounds were selected for the recombination test (recombination model 2). Sensory evaluation analyzes the similarity of the odor between the recombinant model 2 and freeze-dried jujube, and determines the aroma characteristics of freeze-dried jujube. In the end, this study selected 10 sensory evaluators (23–27 years old, 4 males and 6 females) who were experienced and engaged in food flavor research for sensory evaluation. The analysis was according to Gou et al., (2022).

2.7. Quantitative descriptive analysis

The quantitative descriptive analysis (QDA) was often applied to describe the aroma profiles of samples. In this study, 3-hydroxybutan-2-one for "cream" note, 5-butyloxolan-2-one for "floral" note, hexanal for "green" note, methyl dodecanoate for "fruity" note, 2-ethyl-3,5-dimethyl-pyrazine for "roast" note, and (*E*)-but-2-enoic acid for "sweet" note, acetic acid for "sour" note, 3-methyl-butanoic acid for "rancid" note and 2-ethyl-6-methyl-pyrazine for "nut" note were the reference compounds of aroma descriptors which were dissolved in water at a concentration of 100 times of their respective odor threshold (Zhang et al., 2021). QDA was conducted in triplicate by same panelists above mentioned. Scores for each sample in 0.5 increments, from 0.0 to 3.0 on the basis of 7-point scales (0, none; 1.5, moderate; and 3, very strong) (Gou et al., 2022).

2.8. Statistical analysis

All statistical analyses were performed using SPSS version 20.0 software (SPSS Inc., Chicago, IL). Contents of different components were presented as the mean ± SD (standard deviation). The data were illustrated using Origin 2018 (OriginLab Corporation, Northampton, MA). The venn graph was performed by http://jvenn.toulouse.inra.fr/app/example.html.

3. Results and discussion

3.1. Effect of pilot scale FD treatment on aroma properties of red jujube

QDA was performed to evaluate the aroma profile of freeze-dried sample. As shown in Fig. 1**(a)**, there were only five major aroma attributes of freeze-dried red jujube, namely roast, sour/rancid, sweet, fruity and floral. Compared with the raw red jujube, significant change of the aroma characteristics was observed after FD. As for the overall aroma profile, the score of sweet (2.7), fruity (2.5) and cream (2.5) notes were higher than others, dominated aroma notes in raw red jujube. However, the roast note (2.7) exhibited the highest score compared with other aroma attributes in freeze-dried samples. That may be due to the accumulation of pyrazines in FD processing which present the roast and bake notes. Green and cream notes were not observed in the freeze-dried red jujube, which could be mainly interpreted by the loss of C6 aldehydes and ketones during FD processing (Fig. 1**(b)**). The sour/rancid note exhibited the weak intensity (1.2) in the freeze-dried sample because of the high loss ratio of acids (62.88%). Besides, the score of sweet, fruity and floral had less change after FD (Fig. 1**(b)**).

3.2. Effect of pilot scale FD treatment on volatile compounds of red jujube

A total of 41 volatile compounds were identified in pilot scale FD red jujube, including aldehydes (4), alcohols (2), acids (7), esters (9), ketones (3), lactones (5), pyrazines (4), olefins (4), *p*-cymene, naphthalene and 2-pentyl-furan (Table 1). The amount of volatile compounds after FD (41) was not changed obviously compared with raw jujube (42), and the content was only reduced by 26.71%, which represent a higher volatile compounds retention than previously reported literature (An

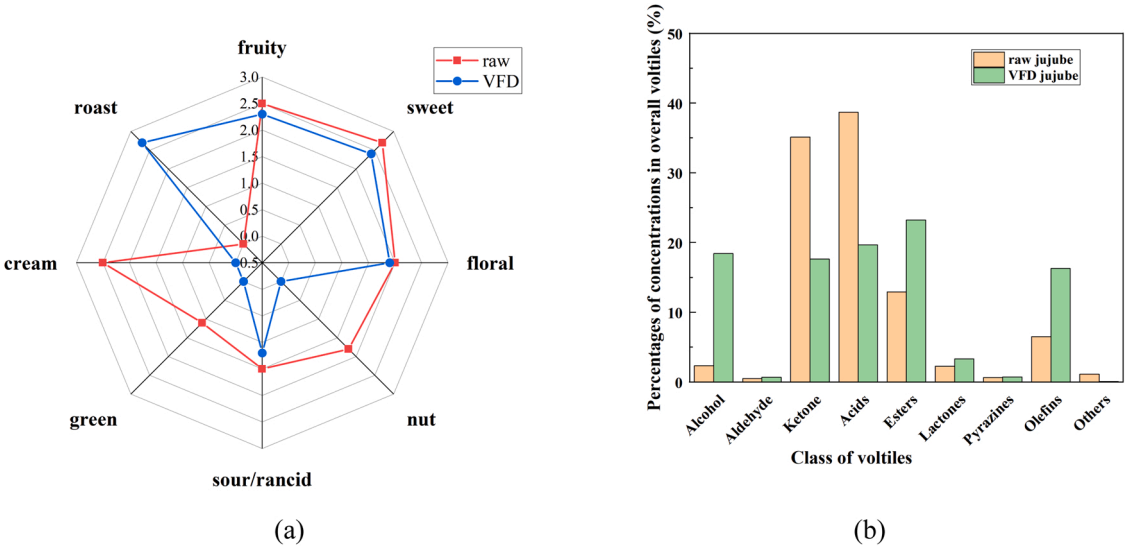

Fig. 1. Volatile profiles (a), content percentages of the major odor classes (b) in raw and freeze-dried jujube.

Table 1
Contents of volatile compounds in raw and freeze-dried jujube.

No	Compounds	Contents (μg/kg)	
		Raw jujube	VFD-jujube
Alcohol			
A1	Oct-1-en-3-ol	0.628 ± 0.05	20.45 ± 0.17
A2	2,3-Butanediol	799.97 ± 18.44	4624.43 ± 139.12
total		800.60	4644.88
Aldehyde			
B1	Hexanal	17.07 ± 0.00	24.02 ± 0.11
B2	(*E*)− 2-Hexenal	32.12 ± 1.26	nd
B3	Nonanal	nd	10.88 ± 1.07
B4	2-Octenal	0.34 ± 0.03	nd
B5	Furfural	37.23 ± 2.05	135.93 ± 2.17
B6	Decanal	0.47 ± 0.02	nd
B7	Benzaldehyde	80.05 ± 5.10	1.86 ± 0.05
total		167.29	172.68
Ketone			
C1	Butane-2,3-dione	2388.62 ± 80.32	nd
C2	3-Octanone	1.07 ± 0.04	1.41 ± 0.04
C3	3-Hydroxybutan-2-one	9242.39 ± 205.87	3266.01 ± 94.89
C4	Oct-1-en-3-one	0.12 ± 0.01	nd
C5	6-Methyl-5-hepten-2-one	0.88 ± 0.02	nd
C6	3-Oxobutan-2-yl acetate	497.90 ± 18.93	1178.03 ± 87.84
C7	Undecan-2-one	nd	2.66 ± 0.07
total		12130.99	4448.10
Acids			
D1	Acetic acid	3549.99 ± 93.54	1920.65 ± 84.26
D2	3-Methyl-butanoic acid	1952.98 ± 79.19	1792.30 ± 101.29
D3	Pentanoic acid	nd	505.98 ± 4.72
D4	(*E*)-But-2-enoic acid	1204.87 ± 35.81	505.52 ± 4.72
D5	Hexanoic acid	2180.26 ± 66.80	92.71 ± 3.72
D6	Heptanoic acid	158.67 ± 11.02	93.36 ± 9.03
D7	(*E*)− 2-Hexenoic acid	4040.63 ± 36.73	nd
D8	Octanoic acid	271.49 ± 27.02	47.88 ± 1.31
total		13358.89	4958.40
Esters			
E1	Methyl hexanoate	135.43 ± 10.94	203.61 ± 1.11
E2	Hexyl acetate	3900.47 ± 5.67	3875.27 ± 2.55
E3	Ethyl heptanoate	258.29 ± 18.64	911.18 ± 3.76
E4	Methyl decanoate	3.79 ± 0.33	27.48 ± 0.29
E5	Ethyl decanoate	31.61 ± 0.02	350.95 ± 0.06
E6	Methyl benzoate	79.12 ± 7.12	93.09 ± 0.81
E7	Methyl dodecanoate	5.38 ± 0.26	20.25 ± 0.64
E8	Ethyl dodecanoate	nd	349.02 ± 0.01
E9	Methyl hexadecanoate	41.05 ± 2.68	23.39 ± 2.06
total		4455.14	5854.24
Lactones			
F1	Oxolan-2-one	456.31 ± 17.30	519.52 ± 27.02
F2	5-Ethyloxolan-2-one	202.04 ± 0.99	91.72 ± 9.08
F3	5-Propyloxolan-2-one	86.21 ± 1.46	170.38 ± 2.79
F4	5-Butyloxolan-2-one	28.29 ± 0.31	9.19 ± 0.08
F5	5-Heptyloxolan-2-one	nd	47.85 ± 0.26
total		772.85	838.66
Pyrazines			
G1	2,6-Dimethyl-pyrazine	0.22 ± 0.00	1.33 ± 0.02
G2	2-Ethyl-6-methyl-pyrazine	16.80 ± 1.37	13.64 ± 0.99
G3	2-Ethyl-3,5-dimethyl-pyrazine	88.00 ± 7.84	107.48 ± 0.20
G4	Tetramethyl-pyrazine	116.01 ± 0.34	63.13 ± 0.95
total		168.15	238.45
Olefins			
H1	Limonene	1.07 ± 0.07	0.34 ± 0.04
H2	γ-Terpinene	nd	1519.69 ± 66.31
H3	Styrene	0.89 ± 0.03	9.67 ± 0.08
H4	α-Farnesene	2245.23 ± 91.23	2572.99 ± 166.27
total		2247.19	4102.69
Others			
I1	2-Pentyl-furan	nd	18.07 ± 0.02
I2	*p*-Cymene	389.50 ± 22.39	nd
I3	Naphthalene	0.11 ± 0.01	3.05 ± 0.02
total		389.62	21.12
Total		**34490.71**	**25279.23**

nd: not detected.

et al., 2016; Mui et al., 2002; Rajkumar et al., 2017a, 2017b). However, the volatile compounds composition changed significantly after FD. The distribution of these volatile compounds was presented in the Fig. 1 **(b)**, the proportions of acids and ketones of freeze-dried red jujube decreased (19.61% and 17.60% of the total volatiles) in comparison with those in the raw red jujube. Moreover, the proportions of esters (23.16%), alcohols (18.37%) and pyrazines (0.94%) increased.

Esters, related to fruity characteristics (Gou et al., 2021), were found to increase after FD. As shown in Table 1, methyl hexanoate and ethyl heptanoate, featured fruity odorant, ranged widely from 135 μg/kg (raw) to 203 μg/kg (FD), and 258 μg/kg (raw) to 911 μg/kg (FD), respectively. And ethyl dodecanoate was newly detected after FD. Besides, methyl decanoate, ethyl decanoate and methyl dodecanoate were increased obviously. These compounds have been reported to be important contributor to the aroma of "Huizao" jujube (Gou et al., 2022). However, no significant differences were found between raw and freeze-dried samples for the concentrations of hexyl acetate and methyl benzoate. The increased content of ester compounds are generally formed through non-enzymatic esterification of alcohols and organic acids, contributed the fruity note (Wang et al., 2020).

Alcohols were increased notably after FD (from 800 μg/kg to 4644 μg/kg), which might be caused by the glucose metabolism, amino acid decarboxylation and dehydrogenation with prolonged FD time or oxidation and degradation of polyunsaturated fatty acids (Ye et al., 2022). Among them, oct-1-en-3-ol with typical mushroom note, which might be generated from oxidation of fatty acids (Zhu et al., 2019) and 2, 3-butanediol, related with creamy, floral and fruity notes, which might be originated from metabolism of pyruvate (Zhang et al., 2020).

Acids content showed a decreasing tendency after FD treatments, which was decreased 62.88% compared with raw jujube. That might be due to the occurrence of chemical reaction, leading the acids transformed to esters through the multi-stage and higher temperature applied in this study. (*E*)− 2-Hexenoic acid is the most abundant volatile acid, while, it was not detected in freeze-dried red jujube. That might be discharged by vacuum pump, due to its volatility. Acetic acid also had an obvious loss rate (45.90%), which might be carried away by the water vapor because of its polarity, or involved in the Maillard reaction. Moreover, acids compounds having usually high thresholds, will contribute slightly to the overall aroma of freeze-dried red jujube.

The content of ketone compounds also obviously decreased after FD, with the loss ratio of 63.33%. The content of 3-hydroxybutan-2-one with cream note decreased 64.66%. Especially, butane-2,3-dione with cream note and 6-methyl-5-hepten-2-one with sweet note were key aroma-active compounds in raw red jujube (Gou et al., 2022), but they did not detected after FD, which caused the loss of "cream" and "sweet" aroma properties of freeze dried red jujube. Generally, most of ketones produced by reducing sugars and amino acid degradation or unsaturated fatty acid oxidation (Zhang et al., 2019). These ketones as α-dicarbonyl compounds could participate in the Maillard reaction and form pyrazines, such as butane-2,3-dione and 3-hydroxybutan-2-one (Mei et al., 2007; Xiao et al., 2018).

Pyrazines are generally results from the Maillard reaction, which are more favorable at high temperature. The percentage of pyrazines content (0.94%) in FD was almost two times higher than in the raw jujube (0.49%). Pyrazines contributed to important aroma characteristics due to their low thresholds, especially 2-ethyl-3,5-dimethyl-pyrazine, which was the major contributor of nut notes in red jujube (Gou et al., 2022). Meanwhile, the increase of pyrazine content may also lead to the enhancement of roast note of freeze-dried red jujube. The alkylpyrazine was produced by condensation of carbonyl compounds, which are degradation products from reducing sugars. The α-carbonyl compounds could react with amino acids to form α-aminocarbonyl compounds and Strecker aldehydes, furthermore, converted to corresponding alkylpyrazine (Deng et al., 2022; Guerra & Yaylayan, 2012; Mei et al., 2007; Scalone et al., 2015). Amino acids and reducing sugars were existed in red jujubes provided good resource for the formation of pyrazine

compounds (Song et al., 2019).

Six and four aldehydes were quantified in raw and freeze-dried samples, respectively. Though the number of aldehyde compounds was decreased, the content of aldehyde was slightly increased in freeze-dried jujube. That might be due to oxidative degradation of aroma precursors, such as fatty acids (Yu Zhao et al., 2021). The furfural was increased remarkably, because in the later stage of FD, the red jujube temperature gradually close to the heating plate temperature (65 ℃), resulted the degradation of carbohydrates or Maillard reaction (Yu et al., 2020). Five lactones compounds were detected in the freeze-dried samples which could be generated as a result of hydroxyl acid intramolecular esterification (Al-Dalali et al., 2020) or formed from the reaction between glycine and D-glucose in slightly acidic (Keyhani and Yaylayan, 1996). All of the detected lactone compounds were found in both raw and freeze-dried samples, except for 5-heptyloxolan-2-one, which was only detected in freeze-dried samples. Olefins contents were increased sharply in freeze-dried red jujube, and γ-terpinene was newly detected. In addition, *p*-cymene and naphthalene were quantified in raw samples, and 2-pentyl-furan and naphthalene were quantified in freeze-dried samples. However, the concentrations of these compounds were low in both samples.

3.3. Identification of aroma-active compounds in pilot scale FD red jujube by GC-O-MS analysis

DF was widely used as the recognition for aroma intensity of the aroma compound. The compound perceived more times by the evaluators, it could be considered to have a larger importance (Gou et al., 2021). There were alcohol (1), aldehydes (2), ketones (5), acids (5), esters (2), lactones (3), pyrazines (2) and olefin detected by DFA in raw red jujube. While, there were alcohols (2), aldehydes (1), ketones (2), acids (5), esters (4), lactones (4) and pyrazines (3) in FD red jujube (Table 2). Although the amount aroma-active compounds was 21 in both raw and freeze-dried samples (Table 2 and Fig. 2), the compositions of aroma-active compounds were quite different. In freeze-dried sample, 14 aroma-active compounds also recognized in raw jujube, and 7 compounds were newly detected as aroma-active compounds, such as 2, 3-butanediol, octanoic acid, ethyl dodecanoate, 5-heptyloxolan-2-one, 2,6-dimethyl-pyrazine and tetramethyl-pyrazine. Furthermore, roast-like 2-ethyl-3,5-dimethyl-pyrazine and fruit-like ethyl heptanoate were perceived by all assessors (DF=6), as well as oct-1-en-3-ol with mushroom and floral notes, 3-hydroxybutan-2-one with sweet and fat notes, 3-oxobutan-2-yl acetate with sweet, fat notes, methyl dodecanoate with coconut, fruit and sweet notes, and fruity-like ethyl decanoate, revealing that they had major contributions to the aroma profile of freeze-dried red jujube. In addition, 2,3-butanediol (fruity), 5-butyloxolan-2-one (fruity) and 5-heptyloxolan-2-one (sweet, coconut) were also recognized their moderate contributions to the aroma of freeze-dried red jujube, because of their relatively higher DF ($6 > DF \geq 4$). The other 9 compounds in freeze-dried red jujube, benzaldehyde (bitter almond), hexanoic acid (acid), 3-methyl-butanoic acid (rancid), (*E*)-but-2-enoic acid (sweet), ethyl dodecanoate (floral, fruit), 5-ethyloxolan-2-one (sweet), 5-propyloxolan-2-one (sweet), 2,6-dimethyl-pyrazine (baked), tetramethyl-pyrazine (roast)) were considered as the potent contributors in the aroma profile of FD red jujube.

In general, alcohols and aldehydes were perceived as floral and green odors, esters contributed fruity odor, acids mainly related to sweat and sour notes, ketones offered cream and sweet aroma, lactones described as sweet and fruity notes, while pyrazines contributed a nut or roast note. In raw red jujube, ketones identified as the dominant aroma-active compounds by DF, thus the cream note is the one of the characteristic aroma in raw jujube. While, roast and more intense fruity aroma characteristics performed in freeze-dried jujube, owing to the main aroma-active compounds were changed into esters and pyrazines after FD. These results are consistent with above mentioned sensory evaluation results.

3.4. Identification of aroma-active compounds in pilot scale FD red jujube by OAVs

OAV analysis is frequently applied to evaluate the aroma potency based on the equilibrium between the food matrix and the air. It is generally accepted that the greater the OAV of the compound, the more important the contribution to aroma (Zhang et al., 2019). According to the principle, compounds with OAVs greater than 1 were regarded the aroma-active compounds of the samples.

There were 16 aroma-active compounds in freeze-dried red jujube identified by OAV, including alcohols (2), aldehydes (2), ketones (1), acids (1), esters (6), lactones (2), pyrazines (1) and olefin (1) (Table 2). From Fig. 2, there were 12 aroma-active compounds identified by OAV joint by raw and freeze-dried red jujube, these compounds mainly contributed to the sweet and fruity notes of raw and freeze-dried red jujube. Nonanal, oct-1-en-3-ol, 5-heptyloxolan-2-one and γ-terpinene were newly identified as aroma-active compounds by OAV after FD, which would contribute the fruity, floral and sweet notes to freeze-dried red jujube. Meanwhile, butane-2,3-dione with the cream note was not detected after FD, while it had the highest OAV (OAV=2388.62) in raw jujube. These changes of OAV might explain the differences of aroma profile between freeze-dried and raw jujube.

In further comparison with raw red jujube, pyrazines and esters contributed higher OAVs to freeze-dried red jujube (Fig. 1**(c)**). On the other hand, 2-ethyl-3,5-dimethyl-pyrazine (OAV=2687.00) was present the highest OAV, followed by ethyl heptanoate (OAV=479.57), and 3-hydroxybutan-2-one (OAV=233.29). This could be explained by the low odor threshold of 2-ethyl-3,5-dimethyl-pyrazine (0.00004 mg/kg) and ethyl heptanoate (0.0019 mg/kg), and the high concentration of 3-hydroxybutan-2-one (3266 μg/kg). Otherwise, ethyl decanoate (OAV=70.19), 2,3-butanediol (OAV=48.63), hexyl acetate (OAV=33.99), and 5-heptyloxolan-2-one (OAV=22.79) also had the higher OAVs in freeze-dried red jujube. In contrast, the OAVs of volatile compounds were less than 1, demonstrating that, individually, these volatiles may make only subtle contributions to the overall aroma of freeze-dried red jujube. This might be due to the high odor threshold or the low concentrations of these volatiles, such as acetic acid and oxolan-2-one with high odor threshold (99 and 20 mg/kg, respectively). In addition, oct-1-en-3-ol, 2,3-butanediol, 3-hydroxybutan-2-one, 3-methyl-butanoic acid, ethyl heptanoate, ethyl decanoate, methyl dodecanoate, 5-butyloxolan-2-one, 5-heptyloxolan-2-one and 2-ethyl-3,5-dimethyl-pyrazine were identified as aroma-active compounds in freeze-dried red jujube by DF and OAV, demonstrating that they have the major contribution on aroma characteristics.

3.5. Identification of key aroma-active compounds in pilot scale FD red jujube by aroma recombination and omission experiments

As shown in Fig. 3, the aroma profile of the complete recombinant (**Model 1**) was similar to that of the original sample. This result indicated the success in identification and quantification of aroma compounds of freeze-dried red jujube, because the mixtures of these odorants are very similar to those of the original samples. To further investigate the contributions of aroma-active compounds to the overall aroma profiles of freeze-dried red jujube, all compounds with $OAVs \geq 1$ and $DF \geq 2$ were subjected to omission experiments. As shown in Table 3, the omission models of 3-hydroxybutan-2-one, 3-oxobutan-2-yl acetate, ethyl heptanoate, methyl decanoate, ethyl decanoate, methyl dodecanoate and 2-ethyl-3,5-dimethyl-pyrazine showed very highly significant differences ($p \leq 0.001$) when compared to the complete recombinant. Additionally, when hexyl acetate and 5-propyloxolan-2-one were omitted, highly significant differences ($p \leq 0.01$) between the omission models and complete recombinant were observed. Furthermore, the significant differences ($p \leq 0.05$) were observed when 2,3-butanediol, ethyl dodecanoate, (*E*)-but-2-enoic acid, 3-methyl-butanoic acid and 5-heptyloxolan-2-one were removed. However, no significant

Table 2
Aroma-active compounds in raw and freeze-dried jujube obtained from OAV and DFA.

No	Compounds	LRI[a]	Identification method[b]	DF[c]		OAV[d]		Odor description[e]
				Raw jujube	FD-jujube	Raw jujube	FD-jujube	
Alcohol								
A1	Oct-1-en-3-ol	1450	MS/O/RI/Std	6	6	0.42 ± 0.03	13.63 ± 0.11	earth, fat, floral, mold, mushroom
A2	2,3-Butanediol	1556	MS/O/RI/Std	< 2	4	8.41 ± 0.19	48.63 ± 1.46	fruit
Aldehyde								
B1	Hexanal	1083	MS/RI/Std	< 2	< 2	3.41 ± 0.00	4.80 ± 0.02	
B2	(*E*)− 2-Hexenal	1216	MS/RI/Std	< 2	nd	0.29 ± 0.01	nd	
B3	Nonanal	1391	MS/RI/Std	nd	< 2	nd	9.89 ± 0.97	
B4	2-Octenal	1429	MS/O/RI/Std	2	nd	0.11 ± 0.01	nd	green, nut, fat
B5	Furfural	1460	MS/RI/Std	< 2	< 2	0.04 ± 0.00	0.01 ± 0.00	
B6	Decanal	1480	MS/RI/Std	< 2	nd	0.16 ± 0.01	nd	
B7	Benzaldehyde	1520	MS/O/RI/Std	4	2	0.11 ± 0.01	0.00 ± 0.00	bitter almond, burnt sugar, cherry, malt
Ketone								
C1	Butane-2,3-dione	979	MS/O/RI/Std	6	nd	2388.62 ± 80.32	nd	butter, caramel, cheese, cream, fruit
C2	3-Octanone	1253	MS/RI/Std	< 2	nd	0.05 ± 0.00	0.07 ± 0.00	
C3	3-Hydroxybutan-2-one	1284	MS/O/RI/Std	4	6	660.17 ± 14.71	233.29 ± 6.78	butter, cream, green pepper, sweat
C4	Oct-1-en-3-one	1290	MS/RI/O/Std	4	nd	40.00 ± 3.33	nd	earth, green, metal, mushroom
C5	6-Methyl-5-hepten-2-one	1338	MS/O/RI/Std	4	nd	0.01 ± 0.00	nd	citrus, mushroom, rubber, strawberry
C6	3-oxobutan-2-yl acetate	1378	MS/O/RI/Std	6	6	–	–	cream, sweet, fat
C7	Undecan-2-one	1598	MS/RI/Std	nd	< 2	nd	0.48 ± 0.01	
Acids								
D1	Acetic acid	1449	MS/O/RI/Std	< 2	< 2	0.04 ± 0.00	0.02 ± 0.00	
D2	3-Methyl-butanoic acid	1666	MS/O/RI/Std	2	2	3.99 ± 0.16	3.66 ± 0.21	cheese, fecal, putrid fruit, rancid, sweat
D3	Pentanoic acid	1733	MS/RI	nd	< 2	nd	0.05 ± 0.00	
D4	(*E*)-But-2-enoic acid	1745	MS/O/RI/Std	2	4	–	–	sweet
D5	Hexanoic acid	1846	MS/O/RI/Std	3	3	2.45 ± 0.08	0.10 ± 0.00	acid, cheese, goat, pungent, rancid
D6	Heptanoic acid	1950	MS/O/RI/Std	2	5	0.25 ± 0.02	0.15 ± 0.01	apricot, floral, rancid, sour, sweat
D7	(*E*)− 2-Hexenoic acid	1967	MS/O/RI/Std	2	nd	–	nd	fat, must
D8	Octanoic acid	2060	MS/O/RI/Std	< 2	4	0.09 ± 0.01	0.16 ± 0.00	acid, cheese, fat, rancid, sweat
Esters								
E1	Methyl hexanoate	1184	MS/RI	< 2	< 2	1.93 ± 0.16	2.91 ± 0.02	
E2	Hexyl acetate	1272	MS/RI	< 2	< 2	34.21 ± 0.05	33.99 ± 0.02	
E3	Ethyl heptanoate	1326	MS/O/RI/Std	< 2	6	135.94 ± 9.81	479.57 ± 1.98	brandy, fruit, wine
E4	Methyl decanoate	1593	MS/RI/Std	< 2	< 2	1.08 ± 0.08	6.39 ± 0.07	
E5	Ethyl decanoate	1638	MS/O/RI/Std	4	6	6.32 ± 0.00	70.19 ± 0.01	bray, burnt, grape, nut, pear
E6	Methyl benzoate	1612	MS/RI	< 2	< 2	0.88 ± 0.10	0.27 ± 0.01	
E7	Methyl dodecanoate	1804	MS/O/RI/Std	6	6	3.59 ± 0.17	13.50 ± 0.43	coconut, fat
E8	Ethyl dodecanoate	1841	MS/O/RI/Std	nd	3	nd	0.87 ± 0.00	floral, fruit, green apple, leaf, nut
E9	Methyl hexadecanoate	2208	MS/RI	< 2	< 2	0.029 ± 0.00	0.01 ± 0.00	
Lactones								
F1	Oxolan-2-one	1632	MS/RI	< 2	< 2	0.02 ± 0.00	0.03 ± 0.00	
F2	5-Ethyloxolan-2-one	1694	MS/O/RI/Std	5	3	1.07 ± 0.00	0.35 ± 0.03	coconut, coumarin, onion, sweet, warm
F3	5-Propyloxolan-2-one	1787	MS/O/RI/Std	2	2	0.22 ± 0.00	0.43 ± 0.01	caramel, fat, nut, peach, sweet
F4	5-Butyloxolan-2-one	1910	MS/O/RI/Std	4	4	4.35 ± 0.05	1.41 ± 0.01	coconut, fruit
F5	5-Heptyloxolan-2-one	2024	MS/O/RI/Std	nd	5	nd	22.79 ± 0.12	apricot, cocoa, coconut, peach, sweet
Pyrazines								
G1	2,6-dimethyl-Pyrazine	1328	MS/O/RI/Std	< 2	2	0.001 ± 0.00	0.003 ± 0.00	baked, bell pepper, green, sweet
G2	2-ethyl-6-methyl-Pyrazine	1386	MS/O/RI/Std	2	< 2	0.42 ± 0.03	0.34 ± 0.02	grass, green, nut, roasted
G3	2-ethyl-3,5-dimethyl-Pyrazine	1455	MS/O/RI/Std	5	6	2200.00 ± 196.00	2687.00 ± 5.00	earth, must, nut, potato, roast
G4	Tetramethyl-pyrazine	1470	MS/O/RI/Std	< 2	2	0.05 ± 0.00	0.03 ± 0.00	cocoa, coffee, green, mocha, roast
Olefins								
H1	Limonene	1200	MS/O/RI/Std	4	< 2	0.01 ± 0.00	0.001 ± 0.00	citrus, orange
H2	γ-Terpinene	1246	MS/RI	nd	< 2	nd	1.52 ± 0.07	
H3	Styrene	1261	MS/RI/Std	< 2	< 2	0.01 ± 0.00	0.15 ± 0.00	
H4	α-Farnesene	1746	MS/RI	< 2	< 2	–	–	
Others								
I1	2-Pentyl-furan	1231	MS/RI/Std	nd	< 2	nd	0.30 ± 0.00	
I2	*p*-Cymene	1270	MS/RI/Std	< 2	nd	77.74 ± 4.47	nd	
I3	Naphthalene	1746	MS/RI/Std	< 2	< 2	0.02 ± 0.00	0.51 ± 0.00	

[a] Retention indices on DB-Wax columns were determined by *n*-alkanes.
[b] MS, identified by MS spectra; LRI, linear retention indices; O, identified by comparison of their odor description with the authentic compounds via GC–O; S, identified by comparison to standards.
[c] Sum of times detected by three assessors during DF, nd: not detected.

[d] OAV was equal to the odor concentration divided by the threshold in water. The threshold was obtained from information available in the website (https://www.vcf-online.nl) and L. J. van Gemert. "-": the threshold is not available. nd: not detected.

[e] Odor description perceived by the judges during DFA.

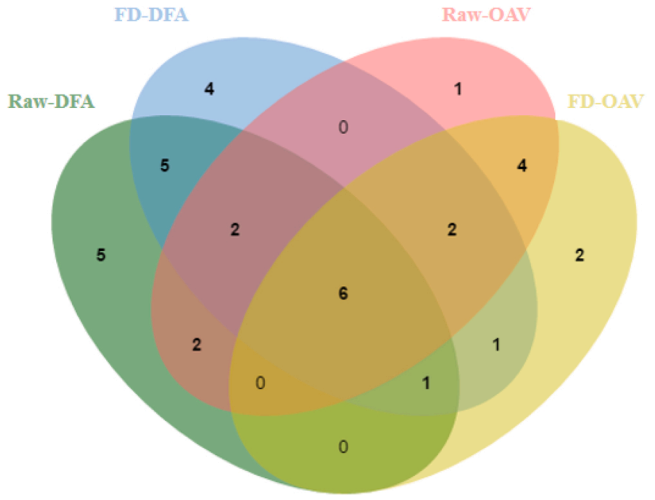

Fig. 2. The venn diagram of aroma-active compounds identified in raw and freeze-dried jujube by OAV and DFA (numbering refers to Table 1).

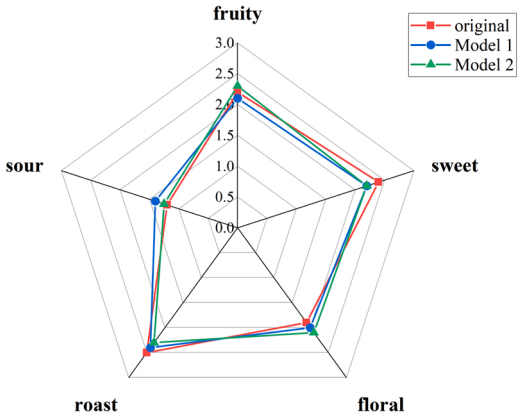

Fig. 3. Aroma profiles of freeze-dried jujube obtained from original sample, model 1, and model 2.

differences were observed by the omission of the other odorants ($p > 0.05$). These results demonstrated that these 14 odorants were the key odorants in complete recombinant. Based on the results of omission tests, the aroma recombination model 2 (Fig. 3) was estimated, which is consisted of above mentioned 14 odorants. Compared the model 2 with original sample, the aroma profile showed no significant difference to the original freeze-dried red jujube. This result verified these 14 compounds as the key aroma-active compounds of freeze-dried red jujube.

Compared to raw red jujube, ethyl heptanoate, hexyl acetate, 2,3-butanediol, ethyl dodecanoate and 5-heptyloxolan-2-one were new key aroma-active compounds in freeze-dried red jujube. While 3-hydroxybutan-2-one, 3-oxobutan-2-yl acetate, methyl decanoate, ethyl decanoate, methyl dodecanoate, 2-ethyl-3,5-dimethyl-pyrazine, 3-methyl-butanoic acid, (*E*)-but-2-enoic acid and 5-propyloxolan-2-one were common key aroma-active compounds of raw red jujube and freeze-dried red jujube. Overall, these composition differences of key aroma-active compounds in raw and freeze-dried red jujube caused the significant difference in their aroma profiles. For example, 2-ethyl-3,5-dimethyl-pyrazine was also key aroma-active compounds in raw jujube, but the content and OAV percentage increased after FD, the

Table 3
Results of omission experiments performed on aroma reconstitutes of freeze-dried jujube.

Number	Compound (s) omitted	N[a]	Significance[b]
1	**all alcohol**	7	*
1–1	Oct-1-en-3-ol	4	–
1–2	2,3-Butanediol	7	*
2	**all aldehyde**	6	–
2–1	Hexanal	5	–
2–2	Nonanal	3	–
2–3	Benzaldehyde	3	–
3	**all ketone**	10	* **
3–1	3-Hydroxybutan-2-one	10	* **
3–2	3-oxobutan-2-yl acetate	9	* **
4	**all esters**	10	* **
4–1	Methyl hexanoate	5	–
4–2	Hexyl acetate	8	* *
4–3	Ethyl heptanoate	9	* **
4–4	Methyl decanoate	9	* **
4–5	Ethyl decanoate	10	* **
4–6	Methyl benzoate	6	–
4–7	Methyl dodecanoate	10	* **
4–8	Ethyl dodecanoate	7	*
5	**all acids**	10	* **
5–1	3-Methyl-butanoic acid	7	*
5–2	(*E*)-But-2-enoic acid	8	* *
5–3	Hexanoic acid	3	–
5–4	Heptanoic acid	5	–
5–5	Octanoic acid	2	–
6	**all lactones**	10	* **
6–1	5-Ethyloxolan-2-one	3	–
6–2	5-Propyloxolan-2-one	8	* *
6–3	5-Butyloxolan-2-one	2	–
6–4	5-Heptyloxolan-2-one	7	*
7	**all pyrazines**	10	* **
7–1	2,6-Dimethyl-pyrazine	4	–
7–2	2-Ethyl-3,5-dimethyl-pyrazine	10	* **
7–3	Tetramethyl-pyrazine	2	–
8	γ-Terpinene	6	–
9	2-Pentyl-furan	1	–
10	Naphthalene	2	–

[a] Number of correct judgments from 10 panelist evaluating the aroma difference by means of a triangle test.

[b] Significance: * ** , very highly significant ($\alpha \leq 0.001$); * *, highly significant ($\alpha \leq 0.01$); and * , significant ($\alpha \leq 0.05$).

aroma profile is transformed from nut note to roast note. Moreover, hexanoic acid, hexanal and 6-methyl-5-hepten-2-one were no longer key aroma-active compounds in freeze-dried red jujube, that resulted the green and cream notes were no longer perceived and the sour note became the weakest aroma attribute in FD red jujube, these results were also consistent with the sensory evaluation results of aroma profiles in raw and freeze-dried jujube.

4. Conclusion

Forty-one volatile compounds were detected in freeze-dried red jujube, 26.71% of aroma loss was observed compared with raw red jujube. 3-Hydroxybutan-2-one, 3-oxobutan-2-yl acetate, ethyl heptanoate, methyl decanoate, ethyl decanoate, methyl dodecanoate, 2-ethyl-3,5-dimethyl-pyrazine, hexyl acetate, 3-methyl-butanoic acid, (*E*)-but-2-enoic acid, 5-propyloxolan-2-one, 2,3-butanediol, ethyl dodecanoate and 5-heptyloxolan-2-one were identified as key aroma-active compounds in freeze-dried jujube, in which ethyl heptanoate, hexyl acetate, 2,3-butanediol, ethyl dodecanoate and 5-heptyloxolan-2-one were newly identified. 2-Ethyl-3,5-dimethyl-pyrazine had the highest OAV (2687.00) and dominated the roast note of aroma profile in freeze-dried

jujube. This study will provide the technical basis for aroma retention during freeze drying in industry.

CRediT authorship contribution statement

Min Gou: Conceptualization, Data curation, Formal analysis, Methodology, Investigation, Writing - original draft & editing. **Qinqin Chen:** Conceptualization, Writing - review & editing, Methodology, Validation. **Yening Qiao:** Investigation. **Xinwen Jin:** Resources, Funding acquisition. **Jingjian Zhang:** Funding acquisition. **Hui Yang:** Resources, Funding acquisition. **Marie-Laure Fauconnier**: Supervision, editing. **Jinfeng Bi:** Conceptualization, Resources, Supervision.

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

The data that has been used is confidential.

Acknowledgements

The funding support of the Financial Fund of Agricultural Science and Technology Innovation Program, Institute of Food Science and Technology, Chinese Academy of Agricultural Sciences (CAAS-ASTIP-2022-IFST), Institute of Food Science, Technology, Nutrition and Health (Cangzhou), CAAS (CAAS-IFSTNH-CZ-2022–02) and Agricultural Science and Technology Innovation Program of Xinjiang Production and Construction Corps (NCG202226) are greatly appreciated by the authors.

Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.jfca.2022.105072.

References

Al-Dalali, S., Zheng, F., Sun, B., Chen, F., 2020. Characterization and comparison of aroma profiles and aroma-active compounds between traditional and modern sichuan vinegars by molecular sensory science. J. Agric. Food Chem. 68, 5154–5167. https://doi.org/10.1021/acs.jafc.0c00470.

An, K., Zhao, D., Wang, Z., Wu, J., Xu, Y., Xiao, G., 2016. Comparison of different drying methods on Chinese ginger (Zingiber officinale Roscoe): Changes in volatiles, chemical profile, antioxidant properties, and microstructure. Food Chem. 197, 1292–1300. https://doi.org/10.1016/j.foodchem.2015.11.033.

Chen, Q., Song, J., Bi, J., Meng, X., Wu, X., 2018. Characterization of volatile profile from ten different varieties of Chinese jujubes by HS-SPME/GC–MS coupled with E-nose. Food Res. Int. 105, 605–615. https://doi.org/10.1016/j.foodres.2017.11.054.

Chin, S.T., Nazimah, S.A.H., Quek, S.Y., Che Man, Y., Bin, Abdul Rahman, R., Mat Hashim, D., 2008. Changes of volatiles' attribute in durian pulp during freeze- and spray-drying process. LWT - Food Sci. Technol. 41, 1899–1905. https://doi.org/10.1016/j.lwt.2008.01.014.

Choi, S.-H., Ahn, J.-B., Kim, H.-J., Im, N.-K., Kozukue, N., Levin, C.E., Friedman, M., 2012. Changes in free amino acid, protein, and flavonoid content in jujube (Ziziphus jujube) fruit during eight stages of growth and antioxidative and cancer cell inhibitory effects by extracts. J. Agric. Food Chem. 60, 10245–10255. https://doi.org/10.1021/jf302848u.

Deng, S., Cui, H., Hayat, K., Zhai, Y., Zhang, Q., Zhang, X., Ho, C.T., 2022. Comparison of pyrazines formation in methionine/glucose and corresponding Amadori rearrangement product model. Food Chem. 382, 132500 https://doi.org/10.1016/j.foodchem.2022.132500.

Dimelow, C.P., Linforth, R.S.T., Taylor, A.J., 2005. Model studies on retention of added volatiles during breadcrumb production. J. Agric. Food Chem. 53, 3572–3576. https://doi.org/10.1021/jf048753i.

Feng, Y., Xu, B., ElGasim, A., Yagoub, A., Ma, H., Sun, Y., Xu, X., Yu, X., Zhou, C., 2021. Role of drying techniques on physical, rehydration, flavor, bioactive compounds and antioxidant characteristics of garlic. Food Chem. 343, 128404 https://doi.org/10.1016/j.foodchem.2020.128404.

Gemert, L.J. van, 2011. Compilations of odour threshold values in air, water and other media, 2011th ed. Oliemans Punter & Partners BV, Netherlands.

Gou, M., Bi, J., Chen, Q., Wu, X., Fauconnier, M.L., Qiao, Y., 2021. Advances and perspectives in fruits and vegetables flavor based on molecular sensory science. Food Rev. Int. 00, 1–14. https://doi.org/10.1080/87559129.2021.2005088.

Gou, M., Chen, Q., Qiao, Y., Li, J., Long, J., Wu, X., Zhang, J., Fauconnier, M., Jin, X., Lyu, J., Bi, J., 2022. Comprehensive investigation on free and glycosidically bound volatile compounds in Ziziphus jujube cv. Huizao. J. Food Compos. Anal. 112, 104665 https://doi.org/10.1016/j.jfca.2022.104665.

Guerra, P.V., Yaylayan, V.A., 2012. Double schiff base adducts of 2,3-butanedione with glycine: Formation of pyrazine rings with the participation of amino acid carbon atoms. J. Agric. Food Chem. 60, 11440–11445. https://doi.org/10.1021/jf303658m.

Jeyaprakash, S., Heffernan, J.E., Driscoll, R.H., Frank, D.C., 2020. Impact of drying technologies on tomato flavor composition and sensory quality. LWT - Food Sci. Technol. 120, 108888 https://doi.org/10.1016/j.lwt.2019.108888.

Keyhani, A., Yaylayan, V.A., 1996. Elucidation of the Mechanism of Pyrazinone Formation in Glycine Model Systems Using Labeled Sugars and Amino Acids. J. Agric. Food Chem. 44, 2511–2516. https://doi.org/10.1021/jf960067z.

Liu, H., Wang, Z., Zhang, D., Shen, Q., Hui, T., Ma, J., 2020. Generation of key aroma compounds in Beijing roasted duck induced via Maillard reaction and lipid pyrolysis reaction. Food Res. Int. 136, 109328 https://doi.org/10.1016/j.foodres.2020.109328.

Mei, Y.L., Parker, J.K., Mottram, D.S., 2007. Mechanisms of alkylpyrazine formation in a potato model system containing added glycine. J. Agric. Food Chem. 55, 4087–4094. https://doi.org/10.1021/jf070044s.

Mui, W.W.Y., Durance, T.D., Scaman, C.H., 2002. Flavor and texture of banana chips dried by combinations of hot air, vacuum, and microwave processing. J. Agric. Food Chem. 50, 1883–1889. https://doi.org/10.1021/jf011218n.

Qiao, Y., Bi, J., Chen, Q., Wu, X., Gou, M., Hou, H., Jin, X., Purcaro, G., 2021. Volatile profile characterization of winter jujube from different regions via HS-SPME-GC/MS and GC-IMS. J. Food Qual. 2021. https://doi.org/10.1155/2021/9958414.

Rajkumar, G., Shanmugam, S., Galvâo, M. de S., Dutra Sandes, R.D., Leite Neta, M.T.S., Narain, N., Mujumdar, A.S., 2017a. Comparative evaluation of physical properties and volatiles profile of cabbages subjected to hot air and freeze drying. LWT - Food Sci. Technol. 80, 501–509. https://doi.org/10.1016/j.lwt.2017.03.020.

Rajkumar, G., Shanmugam, S., Galvâo, M., de, S., Dutra Sandes, R.D., Leite Neta, M.T.S., Narain, N., Mujumdar, A.S., 2017b. Comparative evaluation of physical properties and volatiles profile of cabbages subjected to hot air and freeze drying. Lwt 80, 501–509. https://doi.org/10.1016/j.lwt.2017.03.020.

Scalone, G.L.L., Cucu, T., De Kimpe, N., De Meulenaer, B., 2015. Influence of free amino acids, oligopeptides, and polypeptides on the formation of pyrazines in maillard model systems. J. Agric. Food Chem. 63, 5364–5372. https://doi.org/10.1021/acs.jafc.5b01129.

Song, J., Bi, J., Chen, Q., Wu, X., Lyu, Y., Meng, X., 2019. Assessment of sugar content, fatty acids, free amino acids, and volatile profiles in jujube fruits at different ripening stages. Food Chem. 270, 344–352.

Song, J., Chen, Q., Bi, J., Meng, X., Wu, X., Qiao, Y., Lyu, Y., 2020. GC/MS coupled with MOS e-nose and flash GC e-nose for volatile characterization of Chinese jujubes as affected by different drying methods. Food Chem. 331, 127201 https://doi.org/10.1016/j.foodchem.2020.127201.

Wang, Y., Li, C., Zhao, Y., Li, L., Yang, X., Wu, Y., Chen, S., Cen, J., Yang, S., Yang, D., 2020. Novel insight into the formation mechanism of volatile flavor in Chinese fish sauce (Yu-lu) based on molecular sensory and metagenomics analyses. Food Chem. 323, 126839 https://doi.org/10.1016/j.foodchem.2020.126839.

Xiao, Z., Zhao, L., Tian, L., Wang, L., Zhao, J. yi, 2018. GC–FID determination of tetramethylpyrazine and acetoin in vinegars and quantifying the dependence of tetramethylpyrazine on acetoin and ammonium. Food Chem. 239, 726–732. https://doi.org/10.1016/j.foodchem.2017.07.015.

Xu, W., Pei, Y., Tian, J., Cao, X., Li, G., Jiang, Y., Zhu, G., 2021. Effects of different drying methods on sensory qualities and aroma compounds of finger citron (Citrus medica L. var. sarcodactylis Swingle) slices. J. Food Meas. Charact. 15, 4465–4474. https://doi.org/10.1007/s11694-021-01008-3.

Yan, Q., Wang, H., Zhu, H., Wu, Y., Zhou, F., Wang, Z., Wang.renlei, Hua, C., 2019. Changes and migration of volatile flavor components in freeze-dried peaches. Food Mach. 35, 20–25. https://doi.org/10.13652/j.issn.1003-5788.2019.07.004.

Ye, Y., Wang, L., Zhan, P., Tian, H., Liu, J., 2022. Characterization of the aroma compounds of Millet Huangjiu at different fermentation stages. Food Chem. 366, 130691 https://doi.org/10.1016/j.foodchem.2021.130691.

Yu, H.Y., Xie, T., Xie, J., Chen, C., Ai, L.Z., Tian, H.X., 2020. Aroma perceptual interactions of benzaldehyde, furfural, and vanillin and their effects on the descriptor intensities of Huangjiu. Food Res. Int. 129, 108808 https://doi.org/10.1016/j.foodres.2019.108808.

Zhang, S., Wakai, S., Sasakura, N., Tsutsumi, H., Hata, Y., Ogino, C., Kondo, A., 2020. Pyruvate metabolism redirection for biological production of commodity chemicals in aerobic fungus Aspergillus oryzae. Metab. Eng. 61, 225–237. https://doi.org/10.1016/j.ymben.2020.06.010.

Zhang, W., Dong, P., Lao, F., Liu, J., Liao, X., Wu, J., 2019. Characterization of the major aroma-active compounds in Keitt mango juice: Comparison among fresh, pasteurization and high hydrostatic pressure processing juices. Food Chem. 289, 215–222. https://doi.org/10.1016/j.foodchem.2019.03.064.

Zhang, W., Lao, F., Bi, S., Pan, X., Pang, X., Hu, X., Liao, X., Wu, J., 2021. Insights into the major aroma-active compounds in clear red raspberry juice (Rubus idaeus L. cv.

Heritage) by molecular sensory science approaches. Food Chem. 336, 127721 https://doi.org/10.1016/j.foodchem.2020.127721.
Zhao, Yue, Wang, Y., Li, C., Li, L., Yang, X., Wu, Y., Chen, S., Zhao, Yongqiang, 2021. Novel insight into physicochemical and flavor formation in naturally fermented tilapia sausage based on microbial metabolic network. Food Res. Int. 141, 110122 https://doi.org/10.1016/j.foodres.2021.110122.
Zhu, W., Luan, H., Bu, Y., Li, X., Li, J., Ji, G., 2019. Flavor characteristics of shrimp sauces with different fermentation and storage time. Lwt 110, 142–151. https://doi.org/10.1016/j.lwt.2019.04.091.