Ultrasonics Sonochemistry 90 (2022) 106219

Contents lists available at ScienceDirect

Ultrasonics Sonochemistry

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

Effects of cold plasma, high hydrostatic pressure, ultrasound, and high-pressure carbon dioxide pretreatments on the quality characteristics of vacuum freeze-dried jujube slices

Lin Yuan [a,b,c,d], Fei Lao [a,b,c,d], Xun Shi [e], Donghao Zhang [a,b,c,d], Jihong Wu [a,b,c,d,*]

[a] College of Food Science and Nutritional Engineering, China Agricultural University, Beijing 100083, China
[b] National Engineering Research Center for Fruit and Vegetable Processing, Beijing 100083, China
[c] Key Laboratory of Fruit and Vegetable Processing, Ministry of Agriculture and Rural Affairs, Beijing 100083, China
[d] Beijing Key Laboratory for Food Non-thermal Processing, Beijing 100083, China
[e] Haoxiangni Health Food Co., Ltd., Xinzheng 451100, China

ARTICLE INFO

Keywords:
Pretreatment
Vacuum freeze-drying
Physicochemical properties
Functional qualities
PLS-DA

ABSTRACT

Pretreatment combined with vacuum freeze-drying is an effective technique to extend the storage period of jujube fruits and reduce energy consumption and cost; however, the effects of pretreatment on the quality characteristics of jujube during vacuum freeze-drying remain unknown. In this study, the effects of cold plasma (CP), high hydrostatic pressure (HHP), ultrasound (US), high-pressure carbon dioxide (HPCD), and conventional blanching (BC) as pretreatments on the performance of vacuum freeze-dried jujube slices were investigated. The results indicated that the application of different pretreatments decreased the water activity and increased the rehydration capacity, owing to the pretreatment etching larger and more porous holes in the microstructure. Freeze-dried jujube slices pretreated with HPCD retained most of their quality characteristics (color, hardness, and volatile compounds), followed by the HHP- and US-pretreated samples, whereas samples pretreated with BC showed the greatest deterioration in quality characteristics, and hence, BC is not recommended as a pretreatment for freeze-dried jujube slices. Sensory evaluation based on hedonic analysis showed that jujube slices pretreated with HPCD and US were close to the control sample and scored highest. Compared to other pretreated samples and the control, freeze-dried jujube slices pretreated with HPCD showed the least degradation (4.93%) of cyclic adenosine monophosphate (cAMP), the highest contents of total phenol, total flavonoid, and L-ascorbic acid, and the highest antioxidant capacity. Partial least squares-discriminant analysis (PLS-DA) was performed to screen all the quality characteristic data of different pretreated samples, and 12 volatile compounds, including ethyl hexanoate and (*E*)-2-hexenal, along with color, L-ascorbic acid content, and cAMP content were found suitable to be used as discriminators for pretreated freeze-dried jujube slices. Therefore, non-thermal pretreatments, including HPCD, US, and HHP pretreatments, are promising techniques for the vacuum freeze-drying of jujube products.

1. Introduction

Jujube (*Ziziphus jujuba* Mill.) belongs to the *Rhamnaceae* family and is mainly cultivated in Europe, southern and eastern Asia, and Australia, particularly in the inland region of northern China [1]. Jujube fruits have been used as ingredients in various food items and dietary supplements because they possess abundant biologically active components, such as vitamins, polyphenols, amino acids, triterpenic acids, and polysaccharides [2]. For people with nutritional and health care needs, jujube is a highly nutritious fruit that improves the sleep quality, eliminates toxins, and beautifies the skin; it also acts as a functional herb exerting antioxidant, anti-inflammatory, anticancer, anti-hyperglycemic, anti-hyperlipidemic, and immunomodulatory effects [2]. However, the shelf-life of fresh jujube fruits is extremely short because of their high sensitivity to shrinkage, browning, softening, and decay, which limits their industrial use [3].

Drying is the most common processing method used to extend the shelf-life of fruits, and most jujube fruits are consumed in this form.

* Corresponding author at: College of Food Science and Nutritional Engineering, China Agricultural University, Beijing 100083, China.
E-mail address: wjhcau@hotmail.com (J. Wu).

https://doi.org/10.1016/j.ultsonch.2022.106219
Received 5 June 2022; Received in revised form 16 August 2022; Accepted 2 November 2022
Available online 8 November 2022

Currently, the drying methods for jujube fruits include sun drying, hot-air drying, microwave drying, vacuum freeze-drying, explosion puffing drying, infrared radiation drying, and instant controlled pressure drop drying [4]. Each drying method has its advantages and disadvantages; for example, microwave drying can significantly reduce the drying time and increase the drying rate, but will lead to the accumulation of moisture on the surface and loss of nutrients in the jujube fruits [5]. Non-thermal processing vacuum freeze-drying is a suitable drying method for jujube fruits because it maximizes the retention of color, shape, flavor, and nutrients in the fruits [6,7]. However, vacuum freeze-drying is costly because of its extremely high energy consumption. To overcome this problem, various pretreatments, including chemical and physical treatments, have been used to reduce the time and energy consumption involved in the vacuum freeze-drying of various fruits and vegetables [8].

Conventional hot-water blanching (BC) is the most common and commercially available pretreatment method before drying because of its simple equipment and ease of operation. BC deactivates enzymes and destroys microorganisms at high temperatures, expelling intercellular air from tissues and softening the texture [9]. Krzykowski et al. observed that hot-water BC at 90 °C for 1 min reduced the freeze-drying time of pepper by 30%, but resulted in a significant decrease in the contents of L-ascorbic acid (AA) and total phenols compared to those of untreated samples [10]. In addition, the large amount of wastewater generated by hot-water BC makes it not a sustainable and green pretreatment method. Ultrasound (US), a non-thermal physical pretreatment method, is receiving increasing interest as it enhances mass transfer via direct (inertial flow and sponge effect) and indirect effects (microchannel formation) through unique mechanical fluctuations and cavitation effects. Xu et al. reported that the US of okra before freeze-drying resulted in the displacement of pectin and hemifiber molecules in the samples, which promoted the diffusion of free water between the cell walls [11]. As a result, the total drying time was saved by 25% and the total energy consumption was reduced by 24.28%, while preserving the quality characteristics of freeze-dried okra [11]. Cold plasma (CP) and high hydrostatic pressure (HHP) are two novel non-thermal, environmentally friendly pretreatment methods that use ionized gases and high-pressure shockwaves (100–1000 MPa) to disrupt cell membranes and cell walls, respectively, while creating more intracellular spaces and cavities [12,13]. CP and HHP pretreatments have been reported to modify the microstructure and promote water transfer during the drying of freeze-dried sheep milk powder [14] and freeze-dried strawberry [15], respectively. Compared to HHP, the high-pressure carbon dioxide (HPCD) technique is milder in pressure (< 20 MPa), and pressurized CO_2 lowers the intracellular and extracellular pH and alters the cell membrane structure [16]. In the current industry, the application of HPCD has been innovatively expanded to areas, such as food pretreatment, microbial inactivation, extraction facilitation, and enzyme activity control [16–18], which implies that HPCD may have the potential to be combined with vacuum freeze-drying. Although the initial investment could be costly, these novel nonthermal technologies have proven to be energy-efficient, cost-effective, and environmentally friendly [8]. However, to the best of our knowledge, the comprehensive effects of the aforementioned novel non-thermal physical pretreatments, especially CP and HPCD, on vacuum freeze-dried jujubes have not yet been studied. In addition, little is known about the effects of HPCD pretreatment on the physicochemical properties of dried products.

Therefore, this study investigated the effects of CP, HHP, BC, US, and HPCD pretreatment on the properties of vacuum freeze-dried jujube slices, including water activity, rehydration ratio, color, texture, volatile compounds, bioactive compounds (AA content, total phenol content [TPC], total flavonoid content [TFC], and cyclic adenosine monophosphate [cAMP] content), antioxidant capacity, microstructure, and sensory evaluation. The aim of this study was to provide a useful pretreatment technique to produce vacuum freeze-dried jujube slices with improved product quality.

2. Materials and methods

2.1. Raw materials

Fresh jujube (*Ziziphus jujuba* Mill. cv. Dongzao) fruits from Akesu (Xinjiang Province, China) were obtained in October 2021. The manually collected jujube fruits were similar in size, uniform in color, and mature (50% of the fruit surface had turned red), with no mechanical damage. The fruits were transported to Beijing within 2 days and stored at 4 °C for use within a week. The average soluble solid content and pH of fresh jujube fruits were 22.7 ± 0.30°Brix and 4.87 ± 0.01, respectively. Before pretreatment and vacuum freeze-drying, they were washed, kernels were removed, and fruits were sliced with a tumbling-box slicer (Mad Shark Technology Co., Ltd., Shenzhen, China) into 3-mm slices.

2.2. Sample pretreatments

Jujube slices were divided into six groups (300 g per group) and processed in triplicates. For the HHP and US pretreatments, jujube slices were first packed at atmospheric pressure using a vacuum-packing machine (Deli Group Co., Ltd., Ningbo, China) in a clear polyvinyl chloride retort pouch (15 cm × 22 cm). The following pretreatment conditions were shown to have similar inactivation capacities for the activity of polyphenol oxidase (Fig. S1).

1) A CP device (CTP—2000S; Nanjing Suman Plasma Technology Co., Ltd., Nanjing, China) was used for CP pretreatment of the jujube fruits. The power and frequency were set to 350 W and 10 kHz, respectively, according to [19] with some modifications. Jujube slices (kept cold) were placed in a customized quartz Petri dish (12 cm diameter and 3 mm thickness) 5 cm from the end of the plasma nozzle and pretreated in the dielectric hindrance mode for 30 s.
2) Packed jujube slices for HHP pretreatment were placed into a CQC30L-600 HHP pressurization unit (Suyuanzhongtian Scientific Co., Ltd., Beijing, China) as per Denoya et al. [20]. Pressurization was performed at a rate of approximately 200 MPa/min using distilled water as the transfer fluid. The pouches were treated at 500 MPa for 5 min at 25 °C and immediately depressurized to minimize adiabatic heating.
3) The jujube slices used for BC pretreatment were treated in boiling water at four times the weight of the samples for 1 min.
4) For US pretreatment, packed jujube slices were placed in an ultrasound bath (KQ-500DE NC Ultrasound Cleaner; Kunshan Ultrasound Instrument Co., Ltd., Kunshan, China) with water as the medium and treated at 40 kHz for 20 min [21].
5) The components and diagram of the HPCD system used were described by Liao et al. [17], and the pretreatment method was performed according to Bi et al. [18], with minor modifications. Briefly, freshly cut jujube slices were laid flat on a Petri dish, placed in the stainless-steel pressure vessel of the HPCD system, and vacuumed at room temperature. The CO_2 inlet valve was opened and the vessel was pressurized to 2 MPa using a plunger pump and held for 10 min. Subsequently, depressurization was performed immediately by opening the pressure relief valve at the CO_2 outlet. The pressurization time was 10–40 s and the depressurization time was 30–90 s.
6) Jujube slices without any pretreatment were set as controls.

2.3. Vacuum freeze-drying

Six groups of samples were preliminarily frozen at −20 °C for 12 h and vacuum freeze-dried for 24 h using a vacuum freeze dryer (LGJ-25C; Foring Technology Development Co., Ltd., Beijing, China) at −40 °C (cold trap) and 10 Pa (absolute pressure). The samples were immediately placed in polypropylene boxes, nitrogen-filled, and heat-sealed using the DT-6D-modified atmosphere fresh-keeping packaging

machine (Dajiang Machinery Equipment Co., Ltd., Nanjing, China), followed by storage in a light-proof desiccator until further analysis.

2.4. Water activity (a_w), moisture content (MC), and rehydration ratio (RR)

The a_w of six groups of samples after vacuum freeze-drying was obtained using an HD-4 intelligent water activity meter (Wuxi Huake Instrument and Meter Co., Ltd., Wuxi, China) at 25 °C.

The MC of the dried samples was determined using a halogen rapid moisture tester (ST-105A, Xiamen Yishite Instruments Co., Ltd., Xiamen, China) with an accuracy of 0.005%.

Rajkumar et al. [22] briefly described the methods for the determination of RR as soaking the dried jujube slices (weight R_1) in boiling distilled water for 10 min at 100 °C, removing the excess water from the surface with filter paper, and weighing it as R_2. The percentage ratio of R_2 to R_1 was the RR.

2.5. Texture analysis

The hardness and brittleness of freeze-dried jujube slices were determined with a TAXT Plus texture analyzer (Stable Micro Systems, Surrey, UK) equipped with a cylindrical flat probe (50 mm diameter, P35, aluminum) as described by Wee et al. [23], with some modifications. The sample (1.0 cm × 1.0 cm × 0.3 cm) was excised longitudinally from the freeze-dried jujube slices and placed on the platform as an upright cube. The test standards were set as follows: 3.00 mm/s pre-speed, 1.0 mm/s test speed, 3.00 mm/s post-speed, and 5.0 g of trigger force. The hardness (g) and brittleness (g) were determined using the textural profile analysis test.

2.6. Color measurement

The color of the jujube slices was measured at an ambient temperature using a color difference meter (ColorQuest XE, Hunter Associated Laboratory Inc., USA) in the reflectance mode immediately after opening the package. The light source was set to D65 with a 0.375-in. observation aperture and 10° observation angle. The chromometer was calibrated using a white standard before the samples were measured. The color was recorded in units of L*, a*, and b* uniform color spaces. L* indicates lightness, a* ranges from negative values for green to positive values for red, and b* ranges from negative values for blue to positive values for yellow. The total color difference (ΔE) was calculated using the following equation:

$$\Delta E = \sqrt[2]{\left(L^* - L_0^*\right)^2 + \left(a^* - a_0^*\right)^2 + \left(b^* - b_0^*\right)^2} \quad (1)$$

where L^*_0, a^*_0, and b^*_0 are the control values for fresh jujube slices after vacuum freeze-drying.

2.7. Microstructure analysis

The microstructure of the freeze-dried jujube slices was observed using a field-emission scanning electron microscope (SEM) (SU-8020, Hitachi, Tokyo, Japan) at 50× magnification.

2.8. Volatile compounds

2.8.1. Isolation of volatile compounds

The volatile compounds were extracted following the method described in [7] using solid-phase microextraction (SPME), with minor modifications. Crushed samples (0.5 g) were transferred to a headspace bottle (20 mL; ANPEL Laboratory Technologies Inc., Shanghai, China) containing 10 μL of 10^5 times diluted cyclohexanone as an internal standard. The bottles were sealed using a PTFE-silicone septum and equilibrated at 50 °C for 40 min. Next, a 50/30 μm divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS) SPME fiber (Supelco, Bellefonte, PA, USA) was exposed to the headspace of the samples for 30 min at the same temperature with agitation. Finally, the fiber was withdrawn and introduced into the GC injector at 250 °C for 3 min.

2.8.2. Gas chromatography-mass spectrometry (GC–MS) analysis

GC–MS measurements were conducted following the method reported in previous studies [24], with minor modifications, using an Agilent 7890 gas chromatography system (Agilent Technologies, Santa Clara, CA, USA) equipped with an Agilent 5975C series mass spectrometer. The volatile compounds were isolated with a DB-WAX fused silica capillary column (30 m × 320 μm i.d. × 0.25 μm; Agilent Technologies). Helium (purity ≥99.999%) was used as the carrier gas at a rate of 1.0 mL/min constant flow. The oven temperature was held at 40 °C for 3 min, ramped at a rate of 5 °C/min to 120 °C, followed by ramping to 200 °C at a rate of 10 °C/min, and held for 5 min. MS was carried out in an electron impact mode of 70 eV with a scan range of 45–550 *m/z*.

2.8.3. Identification and quantification analysis

The volatile compounds in freeze-dried jujube slices under different pretreatments were identified by comparing sample mass spectra with those of the standard NIST12 database and by comparing the calculated linear retention indices (LRIs) with the open-access data of the NIST WebBook. The LRIs of volatile compounds were calculated using the retention time (RT) of *n*-alkanes obtained with the same GC–MS temperature program. A difference between the calculated LRI values and those from the NIST Chemistry WebBook (https://webbook.nist.gov/chemistry/) below 20 was acceptable. LRI was calculated using the following equation:

$$\mathrm{LRI} = 100\mathrm{N} + 100\mathrm{n}\left(t_{Ra} - t_{RN}\right) / \left(t_{R(N+n)} - t_{RN}\right) \quad (2)$$

where N is the number of carbon atoms in *n*-alkanes immediately before the RT of the compound, n is the difference in the number of carbon atoms in *n*-alkanes immediately before and after the RT of the compound, t_{Ra} is the RT of the compound, t_{RN} is the RT of *n*-alkanes immediately before the compound, and $t_{R(N+n)}$ is the RT of *n*-alkanes immediately after the compound.

Quantification of volatile compounds in freeze-dried jujube slices was performed using cyclohexanone as an internal standard. The peak areas were normalized to the cyclohexanone added to each sample. The concentrations of the identified compounds were calculated from the ratio of the peak area to that of the internal standard.

2.9. Bioactive compounds

2.9.1. AA

AA content was determined using a 1260 Infinity II (Agilent Technologies) high-performance liquid chromatography with diode-array detection (HPLC-DAD) system equipped with a quaternary pump, an oven for controlling column temperature set at 25 °C, an Athena C18 column (4.6 × 250 mm, 5 μm, ANPEL Laboratory Technologies Inc.). According to the guidelines of the China National Standards, the crushed sample (0.5 g) was transferred to a 50 mL volumetric flask and diluted with 20 g/L metaphosphoric acid solution to volume. It was shaken well and transferred to a 50 mL centrifuge tube. After ultrasonic extraction at 25 °C for 5 min, the sample was centrifuged at 4000 rpm for 5 min and the supernatant was passed through a 0.45 μm aqueous phase filter membrane and injected into HPLC with a 20 μL injection volume. AA was detected at 245 nm and quantified using a standard curve. The mobile phase consisted of methanol (A) and 50 mmol/L KH_2PO_4 and 2.5 mmol/L $C_{19}H_{42}BrN$ (B) in a ratio of 2:98 (*v*/v) at a flow rate of 0.7 mL/min. The column was maintained at 25 °C during the elution program.

2.9.2. cAMP

The extraction and detection methods for cAMP were described by Wang et al. [25]. Crushed jujube powder (3 g) was extracted using 10 mL deionized water in an ultrasonic bath at 25 °C for 20 min. The supernatant was transferred after centrifugation at 9000 *g* for 10 min at 4 °C, and the above steps were repeated to extract the precipitate twice more. The combined supernatants were filtered through a 0.45 μm aqueous phase filter membrane and then detected using HPLC-DAD. The mobile phases were methanol (A) and 50 mmol/L KH_2PO_4 (B) at a ratio of 10:90 (*v*/v) at a flow rate of 1.0 mL/min and the detection temperature and wavelength were 30 °C and 254 nm, respectively. cAMP content was quantified using a standard curve, and the results were expressed as μg/g dry weight (DW).

2.9.3. Preparation of jujube extracts

Extract preparation of TPC, TFC, and antioxidant activities followed the method of Wang et al. [25], with minor modifications. Extract 0.5 g of crushed dried jujube powder in an ultrasonic bath using 10 mL of 80% methanol at 25 °C for 30 min. The supernatant was separated by centrifugation at 10,000 *g* at 4 °C for 10 min, and the residue was re-extracted 3 times in the same manner. Supernatants were combined and stored at 4 °C until further analysis within 24 h.

2.9.4. Determination of TPC and TFC

The Folin–Ciocalteu assay was used to determine TPC. Data were expressed as mg gallic acid equivalents (GAE)/g DW. A colorimetric method was used to determine the TFC. The results were expressed as mg rutin equivalents (RE)/g DW [26].

2.9.5. Antioxidant activities

For the ferric reducing antioxidant power (FRAP) assay, 20 μL of jujube extract was incubated with 4 mL working FRAP solution at 37 °C for 10 min, followed by absorbance detection at 593 nm. AA was used as the antioxidant standard. The results were expressed as mg of AA equivalent antioxidant capacity/g dry weight (mg AAE/g DW) [27].

The DPPH (2, 2-diphenylpicrylhydrazyl) radical scavenging ability of the jujube extract was determined and modified using the previously described method by Wang et al. [28]. Jujube extract (0.1 mL) was mixed with DPPH (0.15 mM) (5.0 mL) at room temperature (approximately 25 °C) for 30 min, and the light absorbance was measured at 517 nm. The AA equivalent was calculated using a standard curve and the results were expressed as mg AAE/g DW.

For the 2, 2′ azino-bis-(3-ethylbenzothiazoline-6-sulphonic acid (ABTS+) diammonium salt radical cation decolorization assay, the absorbance of the blue-green ABTS radical solution was adjusted to 0.70 ± 0.02 at 734 nm. A 100-fold dilution of the extracts (1 mL) was added to the ABTS+ working solution (1 mL) and incubated in the dark for 10 min. Absorbance was measured at 734 nm. The mg AAE/g DW was also used to express the antioxidant capacity of ABTS [29].

2.10. Sensory evaluation

Sensory evaluation of the samples was conducted by eight trained and experienced panelists from China Agricultural University (Beijing, China). A five-point hedonic sensory evaluation was used, with scores ranging from −2 to 2 (extreme dislike to extreme like). The sensory attributes evaluated included appearance, aroma, texture, taste, and overall quality, according to Mohammadi et al. [30], with minor modifications. All six samples tested for sensory attributes were randomly numbered to minimize bias.

2.11. Statistics analysis

All results are presented as the mean ± standard deviation. One-way analysis of variance (ANOVA) and Duncan's multiple range test were used to determine the significant differences between samples using SPSS (version 25.0; Chicago, IL, USA), where the significance level was set at $p < 0.05$. GraphPad Prism 8.0 (San Diego, CA, USA) was used for plotting figures. Tests for correlation between the content of antioxidant compounds and antioxidant activities were performed using the standard Pearson correlation test. To analyze the differences among the samples, principal component analysis (PCA) and partial least squares discriminant analysis (PLS-DA) were performed using SIMCA software (version 11.5; Umetrics, Umeå, Sweden).

3. Results and discussion

3.1. Aw, MC, and RR

A_w and MC are closely related to product spoilage and are therefore considered to be the main reasons for the long shelf-life of dried foods [31]. Table 1 shows the a_w and MC of dried jujube slices after different pretreatments. Except for the BC pretreatment, the a_w values and MC of the other pretreated freeze-dried jujube samples were significantly ($p < 0.05$) lower than those of the samples without pretreatment (0.22 and 7.65%, respectively). In addition, no significant differences ($p > 0.05$) in a_w values were observed between the US sample and other pretreated samples, except for BC. These results were consistent with the changes in a_w and MC of freeze-dried quince slices pretreated with US [32], freeze-dried strawberry chips pretreated with HHP or US [15], and freeze-dried red beets pretreated with US or BC [33]. Although low-moisture foods with $a_w < 0.85$ have not traditionally been considered capable of causing foodborne illness and microbial spoilage, foods with $a_w < 0.3$ are largely protected against lipid oxidation, non-enzymatic browning, and enzymatic activity [34,35]. In the present work, all the samples had $a_w < 0.22$ and MC < 9.08%, indicating that vacuum freeze-drying is a good approach to maintain quality and extend shelf-life. The RR reflects the degree of drying damage within a material, which can partially explain the effectiveness of drying [36]. High RR values are generally considered to correspond to the porous microstructure of freeze-dried products. As shown in Table 1, the samples pretreated with HHP, US, and HPCD showed significantly ($p < 0.05$) higher RR than the unpretreated samples. Changes in the cell wall and cell membrane structure induced by HHP promote mass exchange and water diffusion during drying, thereby increasing moisture transfer rates and rehydration capacity [37,38]. US has been demonstrated to enhance mass transfer and moisture transport by creating microscopic channels in solid materials through unique mechanical fluctuations and cavitation effects [39]. This explains the decreased a_w and increased RR in the samples pretreated with US because of the formation of porous structures that facilitated moisture removal. Water in HPCD-pretreated samples comes into contact with CO_2, forming carbonic acid and releasing H^+ ions, which causes a decrease in extracellular pH. This leads to structural damage and increased permeability of the cell membrane, which promotes water diffusion [16,40]. Overall, these five pretreatments had a positive effect on the drying characteristics of freeze-dried jujube slices.

3.2. Microstructure

Changes in many quality attributes of dried foods, such as rehydration, color, texture, flavor, and nutrition, are closely related to microstructural changes during processing [41]. SEM images of freeze-dried jujube slices pretreated with CP, HHP, BC, US, and HPCD, and control samples are shown in Fig. 1. It can be seen that the unpretreated freeze-dried jujube has a dense structure and intact structure with essentially no visible microscopic pores. In contrast, the pretreated samples exhibited porous structures with different pore sizes and numbers. The increase and expansion of the pore structure promotes the diffusion and evaporation of internal water during drying [42]. The largest pore sizes were observed in the HPCD samples, and the highest number of pores was observed in the US samples. The increased pore size of the HPCD samples may be related to damage to cell walls and cell membranes by

Table 1
Water activity, moisture content, and textural parameters of vacuum freeze-dried jujube slices with different pretreatments.

	Control	CP	HHP	BC	US	HPCD
a_w	0.22 ± 0.00a	0.18 ± 0.00b	0.16 ± 0.00c	0.20 ± 0.01a	0.16 ± 0.01bc	0.15 ± 0.02c
MC (%)	7.65 ± 0.07b	5.75 ± 0.06c	4.81 ± 0.09d	9.08 ± 0.08a	4.56 ± 0.09e	3.80 ± 0.08f
RR (%)	1.82 ± 0.27c	2.53 ± 0.38bc	3.53 ± 0.13a	2.32 ± 0.49c	3.29 ± 0.25ab	4.08 ± 0.49a
Hardness (g)	7400.53 ± 294.60c	6828.09 ± 334.81c	12,506.26 ± 412.28ab	6108.22 ± 1188.32c	10,694.13 ± 81.65b	13,827.39 ± 2634.16a
Brittleness (g)	98.29 ± 8.18d	381.59 ± 228.14d	454.24 ± 134.01d	5772.25 ± 1035.89c	10,590.29 ± 82.87a	7162.58 ± 722.76b

Different letters in the same row indicate significant differences among the pretreatments ($p < 0.05$). CP, cold plasma; HHP, high hydrostatic pressure; BC, blanching; US, ultrasound; HPCD, high-pressure carbon dioxide. a_w, water activity; MC, moisture content; RR, rehydration ratio.

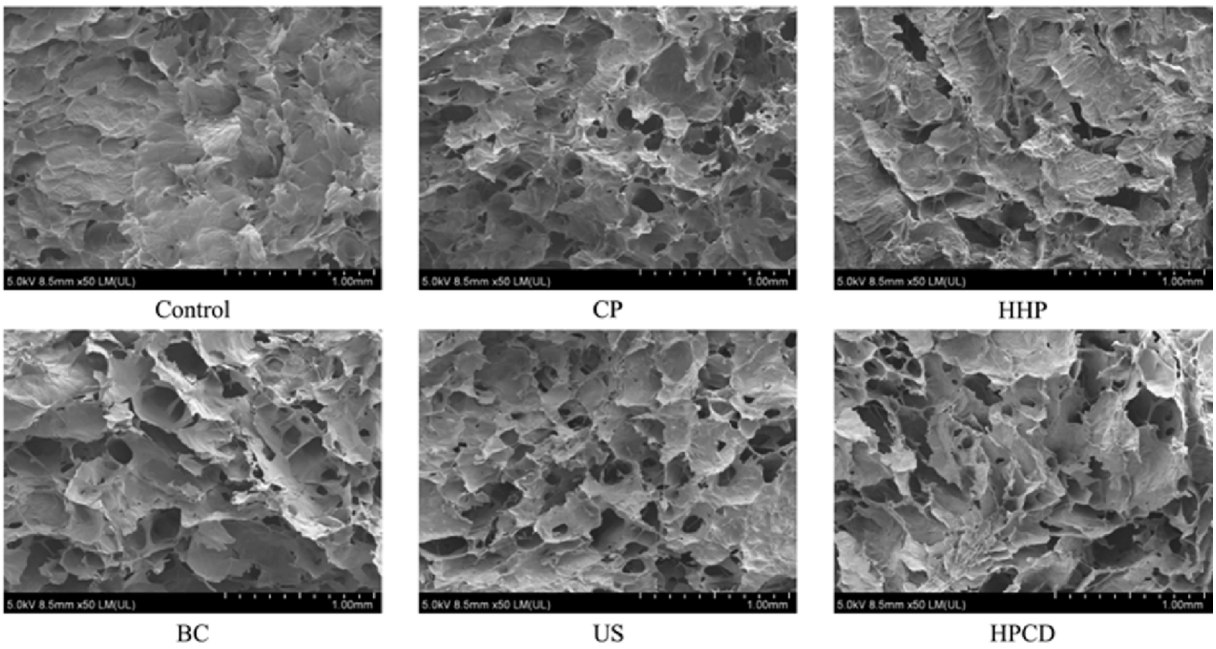

Fig. 1. Scanning electron micrographs of vacuum freeze-dried jujube slices with different pretreatments. CP, cold plasma; HHP, high hydrostatic pressure; BC, blanching; US, ultrasound; HPCD, high-pressure carbon dioxide.

high-pressure and acidic environments, which leads to cell separation and collapse [43]. The increased porosity of US samples may be due to the inertial flow and sponge effect induced by ultrasonic cavitation, which triggers the formation of intercellular micropores [44]. These results are consistent with the microstructural changes in whole jujubes subjected to different pretreatments before hot air drying [45].

3.3. Color and texture

Color plays a critical role in consumer acceptance of dried foods and may also reflect visual quality and nutritional quality. The color is mainly determined by the color parameters L* (lightness/darkness), a* (redness/greenness), b* (yellowness/blueness), and ΔE (total color difference). In this study, different pretreatments resulted in color changes (Fig. 2). Except for the HPCD pretreatment, the L* values increased after the other four pretreatments, indicating that the pretreated freeze-dried jujube slices were significantly ($p < 0.05$) brighter and whiter than the untreated samples. Fresh jujube has a green pulp, so all freeze-dried samples have negative a* values; the smaller the a* value, the greener the sample. As shown in Fig. 2, the freeze-dried jujube slices with CP pretreatment (− 1.13) displayed a significant ($p < 0.05$) increase in a* compared to the control, followed by the BC, HHP, and US samples (− 2.38, − 2.44, and − 3.08, respectively), with no significant ($p > 0.05$) change in a* for the HPCD samples (− 3.41). These results suggest that HPCD pretreatment may protect green color from damage caused by vacuum freeze-drying. The maximum reduction in greenness caused by CP may be due to the non-enzymatic browning promoted by high surface

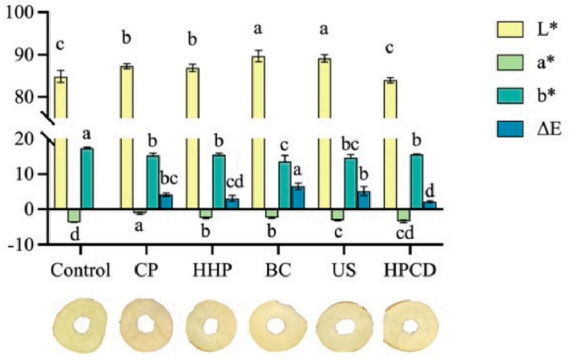

Fig. 2. Color and appearance of vacuum freeze-dried jujube slices with different pretreatments. Different letters in the same row indicate significant differences among treatments ($p < 0.05$). CP, cold plasma; HHP, high hydrostatic pressure; BC, blanching; US, ultrasound; HPCD, high-pressure carbon dioxide.

temperatures [19]. In contrast, the b* value of freeze-dried jujube slices without pretreatment (17.38) was significantly ($p < 0.05$) higher than that of the pretreated samples, indicating that the pretreated samples had significantly lower yellow values than the control. This demonstrates the positive effect of pretreatment on inhibiting the activity of enzymes that promote the browning reaction. In addition, compared with the control, the HPCD samples had the smallest ΔE value (2.12),

followed by the HHP (3.08), CP (4.15), US (5.13), and BC (6.50) samples. It is generally considered that a ΔE value >3 indicates a very distinct color difference between the pretreated and control samples [46]. These results are also directly reflected in the appearance of the freeze-dried jujube slices (Fig. 2), which is consistent with the color parameters detected using the instrument. The control samples were characterized by low brightness and a tendency toward green color, while the HPCD, HHP, CP, US, and BC samples were progressively brighter but lighter in green. The color changes induced by US and BC pretreatment in previous studies on freeze-dried green okra and apple slices were consistent with the color changes observed in this study [9,11]. The effect of pretreatment on color may be explained by cell deformation, membrane rupture, and textural changes, which lead to changes in internal scattered light and surface reflection, making it easier to eliminate pigments from plant tissues [47,48].

This unique texture, which is different from that of fresh agro-products, is also an important characteristic of dried foods. In this study, the a_w of all samples is lower than 0.4 (Table 1), so the hardness and brittleness, which can directly reflect the textural properties of pretreated freeze-dried jujube slices, could be effectively measured [11]. As shown in Table 1, compared with the control samples, the hardness values of the HPCD, HHP, and US samples were significantly ($p < 0.05$) higher than those of the control, CP, and BC samples. However, the differences among the control, CP, and BC samples were not significant ($p > 0.05$). The changes in hardness may be attributed to the combined effect of different MC and microstructures caused by pretreatments. It has been reported that a low MC, a large number of pores, and large pore sizes result in low hardness values of the product [15,42]. In terms of brittleness (frangibility), the brittleness of the samples obtained by US, HPCD, and BC pretreatment was better than that of the HHP, CP, and control. Pretreatment resulted in different changes in the hardness and brittleness of freeze-dried foods. For example, US pretreatment resulted in increased brittleness and unchanged hardness of freeze-dried carrot slices [49], whereas in another study on freeze-dried shiitake mushroom chips, US pretreatment resulted in decreased hardness and brittleness [50]. This may be caused by different substrates and pretreatment conditions, such as power and time. Fan et al. [49] found that the brittleness of freeze-dried carrot slices increased with increasing ultrasonic power used for pretreatment. Hulle et al. [38] observed that the hardness of dehydrated *aloe vera* cubes increased with increasing HHP pressure during pretreatment, whereas the holding time had no significant effect on the hardness.

3.4. Volatile compounds

Aroma is another major determinant of the quality of dried jujube slices, affecting their flavor and commercial appeal [7]. The volatile compounds in control and pretreated freeze-dried jujube slices are listed in Table 2. Fourteen volatile compounds were identified in the control freeze-dried jujube slices, whereas 19, 22, 19, 21, and 18 volatile compounds were identified in the CP, HHP, BC, US, and HPCD samples, respectively (Fig. 3). Aldehydes, acids, esters, alcohols, ketones, furans, terpenoids, naphthalene, and phenols have been reported as the main components of the aroma of fresh jujubes [24]. In this study, 16 esters, 2 aldehydes, 1 alcohol, 4 terpenes, 1 phenol, 1 ketone, and 1 acid were detected in freeze-dried jujube slices, of which 20 components have been reported in previous studies on fresh jujubes and their products [24,51–54]. Six volatile components were identified in pretreated freeze-dried jujubes for the first time. This may be related to the geographical and varietal differences. However, these compounds have been previously reported in soybean [55], jelly palm [56], mushroom [57], rose flower [58], grape [59], and tea [60].

As shown in Fig. 3, the ester content was higher than 50% of the total volatile compounds in six samples (55.03% in BC to 93.37% in HPCD). Among the 16 ester compounds, ethyl hexanoate, which has been identified as a key odor-active compound, had an astonishingly high content in freeze-dried jujubes with different pretreatments, from 62.09 μg/kg (BC) to 507.17 μg/kg (US). Ethyl hexanoate exhibited a pleasant fruity aroma, similar to the smell of apples and green bananas [24]. However, there was no significant ($p > 0.05$) difference in the ethyl hexanoate content among the pretreatments and the control group, except for BC and US, which had the lowest and highest ethyl hexanoate contents, respectively. Compared with the control, CP, HHP, US, and HPCD pretreatments increased the content and type of esters, such as ethyl nonanoate (fruity, rose-like), ethyl caprate (fruity, grape-like), diethyl succinate (fruity, apple-like), 4-hexanolide (herbal, coconut-like), ethyl undecanoate (soapy, coconut-like), ethyl laurate (floral, soapy), ethyl pentadecanoate (honey, sweet), and ethyl hexadecanoate (fruity). This result was consistent with the study of [61–64], which reported an increase in esters in CP-treated fresh-cut cantaloupe, HHP-treated passion fruit puree, US-treated freeze-dried strawberry slice, and HPCD-treated coconut water, respectively. Most esters identified in freeze-dried jujubes are saturated esters, which are synthesized by alcohols and acyl-CoAs catalyzed by alcohol acyltransferases (AAT) [65]. The differential rates of substrate metabolism due to AAT activity and selectivity under different pretreatments may explain the increased esters contents [66]. In contrast, some esters, such as ethyl valerate, ethyl hexanoate, and ethyl (*Z*)-hex-3-enoate, were not detected or were significantly ($p < 0.05$) decreased in BC samples, which may be due to the intensive heat treatment resulting in rapid hydrolysis of these esters by hydrolases during processing [67]. In terms of the other volatile compounds, the content of (*E*)-2-hexenal in the pretreated samples was significantly ($p < 0.05$) lower than that of the control. The terpenes ylangene, γ-muurolene, α-calacorene, and ketone isophorone were detected only in the pretreated samples, suggesting that these components were specific to pretreated freeze-dried jujubes and were formed by reactions that occurred during pretreatment. There were no significant ($p > 0.05$) differences in the contents of benzaldehyde, benzyl alcohol, and (+)-δ-cadinene between the pretreatment samples and the control, indicating that pretreatment had no effect on them. Overall, compared to the control, HPCD, HHP, and US significantly ($p < 0.05$) increased the total content of volatile compounds, CP had no significant ($p > 0.05$) effect on total volatile compounds, and BC significantly ($p < 0.05$) decreased the content of volatile compounds.

3.5. Bioactive compounds and antioxidant activity

Phenolic compounds, especially flavonoids, are considered the main determinants of the antioxidant and anticancer effects of jujubes [2]. Differences in the phenolic compounds of the freeze-dried jujube slices are shown in Table 3. Compared to the control (22.49 mg GAE/g DW), the TPCs of HHP (22.58 mg GAE/g DW) and HPCD (24.55 mg GAE/g DW) pretreatments increased, and there was no significant ($p > 0.05$) difference. In contrast, significantly ($p < 0.05$) low TPC was observed in dried samples pretreated with BC (19.72 mg GAE/g DW). This indicates that the phenolic compounds were effectively preserved by pretreatment, except for BC. Similar results were found by Chen et al. [68], who compared TPCs in dried blueberries under CO_2 laser perforation, US, and freezing-thawing as pretreatments. The TFCs of the pretreated freeze-dried jujubes are listed in Table 3. Compared with the control sample (20.99 mg RE/g DW), the sample pretreated with HPCD (37.13 mg RE/g DW) showed the significantly ($p < 0.05$) highest TFC. In addition, the TFCs in the samples pretreated with CP (21.99 mg RE/g DW), HHP (32.23 mg RE/g DW), and US (29.37 mg RE/g DW) were slightly higher than those in the control sample, although the difference was not significant ($p > 0.05$). In contrast, the BC-pretreated samples (16.27 mg RE/g DW) retained the least amount of flavonoids, indicating that the non-thermal pretreatments effectively retained the flavonoids. This is possibly attributed to the lower thermal degradation, depolymerization, and polymerization of flavonoids resulting from the low-temperature environment of the pretreatment [69]. On the one hand, the structural damage to plant tissues caused by pretreatment facilitates

Table 2
Volatile profiles of vacuum freeze-dried jujube slices with different pretreatments.

No.	Compounds	CAS	LRI[1]	Concentration (μg/kg)[2]						Identification[3]	Aroma descriptors[4]
				Control	CP	HHP	BC	US	HPCD		
	Esters										
A1	Ethyl valerate	539-82-2	1167	8.63 ± 0.29a	6.45 ± 0.32b	n.d.	n.d.	8.13 ± 2.26ab	n.d.	MS, LRI	Fruity, apple
A2	Ethyl hexanoate	123-66-0	1253	325.16 ± 33.8b	348.27 ± 24.02ab	429.35 ± 64.92ab	62.09 ± 0.61c	507.17 ± 171.54a	423.21 ± 40.38ab	MS, LRI	Fruity, apple, green banana
A3	Ethyl (*Z*)-hex-3-enoate	64187-83-3	1316	39.84 ± 6.51ab	46.91 ± 1.56a	21.24 ± 0.48c	n.d.	46.42 ± 17.68a	26.72 ± 0.54bc	MS, LRI	Green, pear
A4	Ethyl (*E*)-hex-2-enoate	27829-72-7	1357	n.d.	n.d.	n.d.	n.d.	6.23 ± 1.76a	n.d.	MS, LRI	Green, fruity
A5	Ethyl octanoate	106-32-1	1446	7.54 ± 1.32d	19.65 ± 6.84c	32.41 ± 7.11b	9.86 ± 0.47 cd	17.17 ± 4.92 cd	46.51 ± 4.32a	MS, LRI	Fruity, banana
A6	Ethyl nonanoate	123-29-5	1544	n.d.	11.09 ± 8.40ab	9.85 ± 3.76ab	3.24 ± 0.14bc	8.00 ± 1.06ab	14.63 ± 2.39a	MS, LRI	Fruity, rose
A7	Ethyl caprate	110-38-3	1647	n.d.	9.23 ± 6.10c	46.85 ± 13.59b	3.95 ± 0.32c	12.79 ± 2.25c	73.55 ± 19.46a	MS, LRI	Fruity, grape
A8	Diethyl succinate	123-25-1	1684	n.d.	19.70 ± 8.41b	15.13 ± 0.89b	2.96 ± 0.15c	22.17 ± 5.39ab	30.43 ± 4.47a	MS, LRI	Fruity, apple
A9	4-Hexanolide	695-06-7	1709	n.d.	n.d.	n.d.	3.09 ± 0.16b	4.64 ± 0.71a	n.d.	MS, LRI	Herbal, coconut
A10	Ethyl undecanoate	627-90-7	1748	n.d.	n.d.	1.63 ± 0.07a	n.d.	n.d.	1.24 ± 0.16a	MS, LRI	Soapy, coconut
A11	Ethyl laurate	106-33-2	1850	n.d.	n.d.	91.58 ± 39.27ab	16.44 ± 0.71c	69.39 ± 13.59b	114.71 ± 23.94a	MS, LRI	Floral, soapy
A12	Ethyl myristate	124-06-1	2056	2.38 ± 0.65b	18.52 ± 14.33ab	29.47 ± 13.26a	6.06 ± 2.58b	18.74 ± 2.92ab	33.99 ± 6.83a	MS, LRI	Sweet, violet
A13	Ethyl (*E*)-9-octadecenate	6114-18-7	2093	2.40 ± 0.49b	14.87 ± 12.33b	77.48 ± 31.72a	5.05 ± 0.89b	21.70 ± 4.96b	91.22 ± 24.62a	MS	
A14	Ethyl pentadecanoate	41114-00-5	2157	n.d.	2.85 ± 0.06a	2.33 ± 0.70a	n.d.	3.94 ± 0.77a	3.75 ± 1.66a	MS, LRI	Honey, sweet
A15	Ethyl hexadecanoate	628-97-7	2258	1.43 ± 0.69c	11.59 ± 5.71ab	17.50 ± 6.03a	3.53 ± 0.31bc	12.80 ± 4.93a	18.24 ± 3.83a	MS, LRI	Fruity
A16	Ethyl 9-hexadecenoate	54546-22-4	2292	3.83 ± 3.34c	47.34 ± 9.59bc	124.51 ± 41.08a	14.17 ± 0.74c	67.37 ± 24.08b	120.94 ± 27.68a	MS, LRI	
	Aldehydes										
B1	(*E*)-2-Hexenal	6728-26-3	1238	88.28 ± 10.03a	48.78 ± 2.71bc	43.60 ± 8.57bc	34.87 ± 11.92c	56.04 ± 10.25b	41.36 ± 2.37bc	MS, LRI	Green banana
B2	Benzaldehyde	100-52-7	1532	15.77 ± 1.85a	13.63 ± 6.04a	10.09 ± 4.69a	9.03 ± 3.77a	12.20 ± 2.84a	8.70 ± 2.90a	MS, LRI	Sweet, cherry
	Alcohols										
C1	Benzyl alcohol	100-51-6	1882	3.65 ± 0.98a	8.05 ± 6.48a	2.44 ± 1.51a	3.09 ± 0.66a	3.43 ± 0.82a	2.10 ± 0.61a	MS, LRI	Floral, rose
	Terpenes										
D1	Ylangene	14912-44-8	1485	n.d.	n.d.	2.03 ± 0.28a	n.d.	n.d.	n.d.	MS, LRI	
D2	γ-Muurolene	30021-74-0	1690	n.d.	n.d.	2.90 ± 0.15a	1.31 ± 0.04b	n.d.	n.d.	MS, LRI	Herbal, woody
D3	(+)-δ-Cadinene	483-76-1	1761	7.66 ± 2.32b	15.89 ± 1.89a	9.64 ± 2.38b	9.52 ± 0.84b	6.66 ± 0.56b	6.68 ± 0.69b	MS, LRI	Thyme, herbal
D4	α-Calacorene	21391-99-1	1923	n.d.	1.18 ± 0.15b	1.44 ± 0.07a	0.81 ± 0.09c	1.28 ± 0.03b	n.d.	MS, LRI	Woody
	Phenols										
E1	Phenol	108-95-2	2002	17.47 ± 14.36a	29.93 ± 12.36a	3.34 ± 0.50a	25.60 ± 27.33a	35.90 ± 34.14a	n.d.	MS, LRI	Phenolic
	Ketones										
F1	Isophorone	78-59-1	1597	n.d.	n.d.	8.95 ± 0.68b	n.d.	n.d.	12.15 ± 1.32a	MS, LRI	Green, camphor
	Acids										
G1	Hexanoic acid	142-62-1	1845	38.18 ± 11.74ab	50.90 ± 21.31a	n.d.	22.38 ± 2.22b	n.d.	n.d.	MS, LRI	Sour
	Total			562.22 ± 87.88c	724.83 ± 148.31bc	983.77 ± 240.61ab	237.04 ± 49.27d	942.17 ± 307.43ab	1070.15 ± 167.45a		

[1] LRI: calculated linear retention index on the DB-WAX column. [2] Values are represented as the mean ± standard deviation (SD; $n = 3$). n.d.: not detected as the concentration of the compound was below the detection limit. Different lowercase letters in the same row indicate significant differences ($p < 0.05$). CP, cold plasma;

HHP, high hydrostatic pressure; BC, blanching; US, ultrasound; HPCD, high-pressure carbon dioxide. [3] Identification: volatile compounds were identified by the following abbreviations: LRI, linear retention index; MS, mass spectrum. [4] Reference aroma descriptors from the LRI & Odor Database (http://www.odour.org.uk/).

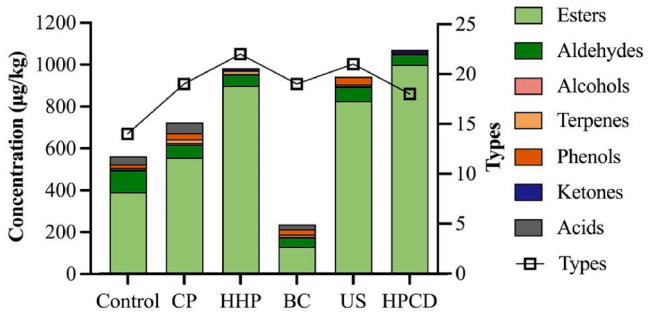

Fig. 3. Concentrations and types of volatile compounds in vacuum freeze-dried jujube slices with different pretreatments. CP, cold plasma; HHP, high hydrostatic pressure; BC, blanching; US, ultrasound; HPCD, high-pressure carbon dioxide.

Table 3
Bioactive compounds and antioxidant activity of vacuum freeze-dried jujube slices with different pretreatments.

	Control	CP	HHP	BC	US	HPCD
TPC (mg GAE/g DW)	22.49 ± 1.58ab	21.86 ± 1.51b	22.58 ± 0.36ab	19.72 ± 1.74c	22.12 ± 0.09b	24.55 ± 0.47a
TFC (mg RE/g DW)	20.99 ± 4.40bc	21.99 ± 2.15abc	32.23 ± 8.54ab	16.27 ± 0.70c	29.37 ± 12.21abc	37.13 ± 11.66a
AA (mg/g DW)	9.94 ± 0.35ab	4.52 ± 0.31c	9.54 ± 0.05ab	5.51 ± 0.21c	8.89 ± 0.16b	10.14 ± 1.35a
cAMP (mg/d DW)	2.03 ± 0.06a	0.75 ± 0.01b	0.66 ± 0.03b	0.54 ± 0.15b	0.77 ± 0.21b	1.93 ± 0.64a
FRAP (mg AAE/g DW)	40.86 ± 9.39a	42.81 ± 2.91a	43.63 ± 4.47a	46.88 ± 2.31a	42.12 ± 4.23a	48.87 ± 2.71a
ABTS (mg AAE/g DW)	51.01 ± 3.99ab	44.24 ± 3.09b	44.52 ± 4.11ab	51.01 ± 2.54b	46.03 ± 4.92b	55.33 ± 3.10a
DPPH (mg AAE/g DW)	31.73 ± 0.51a	26.29 ± 1.53b	27.76 ± 0.95a	30.46 ± 0.45b	30.13 ± 1.17a	31.24 ± 0.78a

Different lowercase letters in the same row indicate significant differences ($p < 0.05$). CP, cold plasma; HHP, high hydrostatic pressure; BC, blanching; US, ultrasound; HPCD, high-pressure carbon dioxide. TPC, total phenol content; TFC, total flavonoid content; AA, L-ascorbic acid; cAMP, cyclic adenosine monophosphate; FRAP, ferric reducing antioxidant power; ABTS: 2,2′-azinobis-(3-ethylbenzothiazoline-6-sulfonate), DPPH: 2,2-diphenyl-1-picrylhydrazyl.

the extraction of bioactive substances, and on the other hand, bioactive compounds leak along with juice because of the lack of a barrier in the form of a compact cell layer on the surface [70].

Jujube fruits are an excellent source of high levels of AA compared to other fruits [2]. There was no significant ($p > 0.05$) change in AA contents of dried samples pretreated with HHP (9.54 mg/g DW), US (8.89 mg/g DW), and HPCD (10.14 mg/g DW) compared to the control sample (9.94 mg/g DW), whereas AA contents of the CP (4.52 mg/g DW) and BC (5.51 mg/g DW) samples were significantly ($p < 0.05$) lower (Table 3). Decreased AA in cashew apple juice, orange juice, and strawberry after CP treatment has been reported in the literature, and both treatment time and applied voltage of CP have a significant effect on AA content [71–73]. This decrease may be attributed mainly to the reaction of ozone and free radicals generated by CP with AA [72,74]. In contrast, the reduction in AA content in BC samples was attributed to the water solubility and thermal instability of AA, which has been reported in several previous studies [10,75].

Jujubes are the only fruits and vegetables that carry the active cAMP form and thus, have been used as traditional Chinese medicine for asthma and allergic reactions [76]. As shown in Table 3, the cAMP content of the HPCD sample (1.93 mg/g DW) was not significantly ($p > 0.05$) different from that of the control sample (2.03 mg/g DW). In contrast, the cAMP contents of all other pretreated samples were significantly lower than that of the control sample, which were presented following the order: US (0.77 mg/g DW) > CP (0.75 mg/g DW) > HHP (0.66 mg/g DW) > BC (0.54 mg/g DW). However, there was no significant ($p > 0.05$) difference among the CP, HHP, BC, and US samples. The content of cAMP in jujubes has been reported to be influenced by geography, cultivar, ripeness, and processing conditions [25,77–80]. The different pretreatments in this study may have caused different stimulation of adenylate cyclase activity [81], while the better retention of cAMP by HPCD may be due to the activating effect of CO_2 on cAMP [82].

The total antioxidant capacity was characterized in terms of FRAP, $ABTS^+$ scavenging activity, and DPPH scavenging capacity. None of the pretreatments significantly ($p > 0.05$) affected the FRAP and ABTS assays of freeze-dried jujube slices compared to the control (Table 3). These results are in accordance with previous reports that the antioxidant activity of fresh and dried walnuts [83] and dried jujube [45] did not change significantly after CP, US, and BC pretreatment. In terms of DPPH scavenging capacity, the HHP, US, HPCD, and control samples revealed significantly ($p < 0.05$) higher DPPH free radical scavenging capacities than the samples pretreated with CP and BC. In general, antioxidant activity is linearly correlated with the presence of bioactive compounds in a sample [28]. To investigate the effect of bioactive compounds on the antioxidant capacity of freeze-dried jujube slices, the correlation coefficients (r) between the TPC, TFC, AA content, cAMP content, and FRAP, ABTS, and DPPH antioxidant activities are presented in Table S1. A significant correlation was found between TPC and FRAP ($r = 0.571$). The results also showed that the TPC, TFC, and AA contents in the samples were extremely highly ($p < 0.01$) correlated with ABTS activity ($r = 0.760$, 0.599, and 0.700, respectively). The DPPH scavenging capacity was found to be highly significantly ($p < 0.01$) and positively correlated with TFC and AA content ($r = 0.638$ and 0.881, respectively), and significantly ($p < 0.05$) and positively correlated with TPC ($r = 0.520$). These results are consistent with those of previous studies [26,78]. In the pretreatment used in this study, HPCD was able to better maintain the antioxidant activity of freeze-dried jujube slices, mainly because the oxygen exclusion and low temperature in HPCD processing could effectively maintain the bioactive components in the samples, which is consistent with previous research on HPCD-treated litchi juice [84].

3.6. Sensory evaluation

To evaluate the acceptability of the vacuum freeze-dried jujube slices with different pretreatments, the sensory qualities of the samples were analyzed in five aspects, and the results are shown in Fig. 4. The CP sample scored the lowest in all sensory attributes, whereas the HPCD and US samples received positive scores from the panelists for all five sensory attributes and were closer to the control sample, especially in taste and overall quality. Interestingly, the scores of the panelists for the appearance of freeze-dried jujube slices corresponded to the a* values of the color measurements, indicating that the degree of greenness determined the popularity of the dried jujube slices. However, the scores of the panelists for aroma did not agree with the total amount of volatile compounds because not all volatile compounds are perceived [85]. The

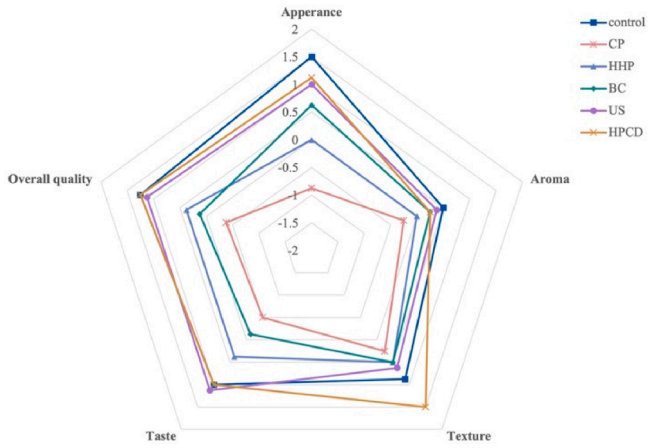

Fig. 4. Hedonic sensory test of vacuum freeze-dried jujube slices with different pretreatments. CP, cold plasma; HHP, high hydrostatic pressure; BC, blanching; US, ultrasound; HPCD, high-pressure carbon dioxide.

texture score, in contrast, may be attributed to the combined effect of hardness and brittleness. In conclusion, sensory evaluation tests showed that HPCD and US pretreatments had a more desirable appearance, texture, and taste than CP, BC, and HHP pretreatments, which is consistent with previous studies [63,86].

3.7. Multivariate analysis

To analyze differences between the control and pretreated freeze-dried jujube slices, data on quality characteristics obtained for the six samples were subjected to PCA for exploratory data analysis; the score plot is shown in Fig. 5A. In the PCA models, R^2 indicates goodness of fit and Q^2 indicates the predictive power, respectively. In this study, the R^2 and Q^2 in the PCA model were 0.913 and 0.584, respectively. As shown in Fig. 5A, the first two principal components (PC1 and PC2, respectively) explained 57.60% of the variance in the sample data. Freeze-dried jujube slices with CP and BC pretreatments were located in the same region of PC1 and PC2. This can be explained by the fact that the sharp increase in surface temperature due to CP pretreatment is similar to the variation of the thermal BC pretreatment compared to the other pretreatments [19]. However, the control, HHP, US, and HPCD samples were not located in the same region of PC1 and PC2, indicating that they could be well distinguished.

To fully understand the differences in quality characteristics and explore the potential of physicochemical properties as indicators of freeze-dried jujube slices pretreated by different methods, PLS-DA was performed with physicochemical properties considered as X-variables and different pretreatments, including control, CP, HHP, BC, US, and HPCD as categorical Y-variables. Six latent variables (LVs) were selected as optimal to describe the physicochemical properties, and explained 96.83% of the Y-variance. The R^2X, R^2Y, and Q^2 values of the PLS-DA model are 0.907, 0.968, and 0.890, respectively, which are all close to 1, proving that the model yields satisfactory results. In addition, the PLS-DA model was subjected to a 200-times permutation trial, and the results showed that the R^2 and Q^2 values on the left side of the validation model were lower than those on the right side (Fig. S2). Usually, the model was validated when the Y-axis intercepts of R^2 and Q^2 were between 0.3 and 0.4 and <0.05, respectively. In this study, the PLS-DA model was validated without overfitting because the Y-axis intercepts of R^2 and Q^2 were within the acceptable range (0.390 and − 0.711, respectively) (Fig. S2).

To visually compare the effects of different pretreatments compared to control samples, a PLS-DA biplot was constructed for the first two LVs (LV1 and LV2) (Fig. 5B). In the biplot, groupings or separations among the different pretreated freeze-dried jujube slice classes were observed, similar to the PCA plot. In addition, the importance of physicochemical properties for classification can be indicated by their position and distance from the center. The inner, middle, and outer ellipses represent the correlation coefficients of 50, 75, and 100%, respectively. This indicates that for physicochemical properties located between the inner and middle ellipses, 50–75% of the variability is explained by LV1 and LV2, and for those located between the middle and outer ellipses, 75–100% of the variability is explained by LV1 and LV2. Moreover, the further away from the center and the closer to a group of classes, the more highly positively the physicochemical properties are correlated with the corresponding class. Therefore, it can be clearly observed that the volatile compounds (*E*)-2-hexenal (B1), ylangene (D1), and ethyl (*E*)-hex-2-enoate (A4) are more closely correlated with the control, HHP, and US samples, respectively. The CP sample showed a high correlation with benzyl alcohol (C1) and (+)-δ-cadinene (D3), whereas for the HPCD sample, the close correlations were ethyl hexanoate (A2) and isophorone (F1). Although some information can be inferred from the biplot, this is not a simple way to assess the importance of each variable. Therefore, the variable importance in projection (VIP) scores were analyzed. The VIP score shows the importance of the variable in the predictive ability of the PLS-DA model and can be used as a criterion for variable selection [87]. When VIP $>$ 1, the variable is considered important for the model

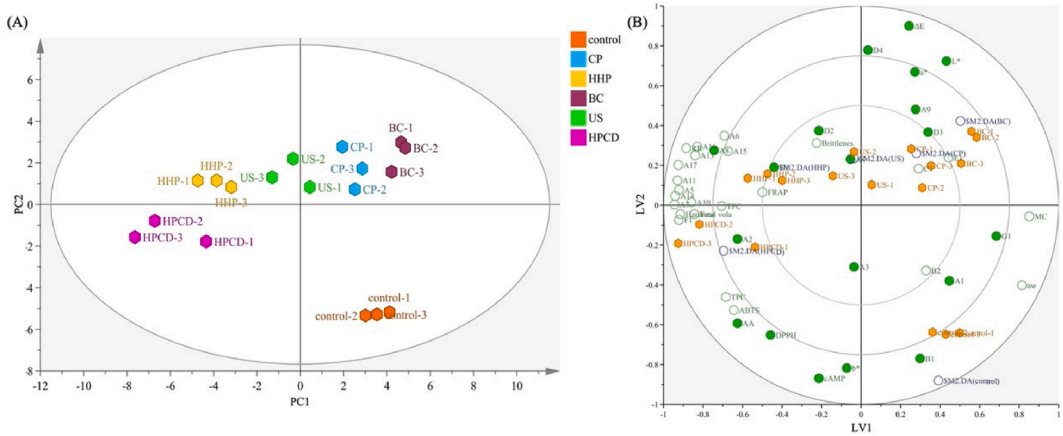

Fig. 5. Principal component analysis (PCA) score scatter plot (A) and partial least squares-discriminant analysis (PLS-DA) biplots (B) describing the comparison of treatment impact of freeze-dried jujube slices with different pretreatments. Open circles represent the physicochemical properties; indicators with variable importance in projection (VIP) $>$ 1 are represented as solid circles. Inner, middle, and outer ellipses represent the correlation coefficients of 50, 75, and 100%, respectively. CP, cold plasma; HHP, high hydrostatic pressure; BC, blanching; US, ultrasound; HPCD, high-pressure carbon dioxide.

[88] and is labelled as a solid green circle in Fig. 5B. In total, there were 19 markers, of which 12 were volatile compounds, 4 were color parameters, and 3 were bioactive compounds. Among these, physicochemical properties, including volatile compounds (ethyl hexanoate (A2), (*E*)-2-hexenal (B1), and hexanoic acid (G1)), bioactive compounds (cAMP and AA), and color parameters have been reported as discriminating indicators in jujube fruits of different origins, cultivars, processing methods, and storage times [89–93]. Terpene discrimination indicators included all the terpenes detected in this study, indicating that the terpenes are more sensitive to processing. Among them, ylangene (D1) was unique to HHP pretreatment, whereas γ-muurolene (D2) was specific to HHP and BC pretreatments. Therefore, these compounds were used as pretreatment indicators for freeze-dried jujube slices.

4. Conclusions

The quality characteristics of freeze-dried jujube slices were significantly affected by CP, HHP, BC, US, and HPCD pretreatments. All pretreatments decreased the water activity and b* values and increased the a* values and brittleness owing to changes in the microstructure and inactivation of enzymes. Compared to other pretreated samples, freeze-dried jujube slices pretreated with HPCD protected the color, sensory properties, and contents of total phenols, total flavonoids, AA, and cAMP, and antioxidant capacity, while increasing the concentration of volatile compounds. In contrast, the greatest deterioration in quality characteristics (color, volatile compounds, and bioactive compounds) was found in the freeze-dried samples pretreated with conventional BC owing to the intensive heat treatment. PLS-DA revealed the color parameters, some volatile compounds, AA, and cAMP as discriminator indicators to confirm the differences among the five selected pretreated samples and control samples. In conclusion, these pretreatments helped improve the original quality of freeze-dried jujube slices. Notably, HPCD pretreatment has the potential to be used for the production of vacuum freeze-dried jujube products. However, further process optimization is needed to shorten the drying time and energy consumption after non-thermal pretreatment to improve the vacuum freeze-drying efficiency, as well as physiological experiments to explore the benefits of freeze-dried jujube slices.

CRediT authorship contribution statement

Lin Yuan: Data curation, Methodology, Writing – original draft, Writing – review & editing. **Fei Lao:** Project administration, Writing – review & editing. **Xun Shi:** Project administration. **Donghao Zhang:** Writing – review & editing. **Jihong Wu:** Conceptualization, Project administration, 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

Data will be made available on request.

Acknowledgments

The authors are grateful for the support provided by the Support Plan for Innovation and Development of Key Industries of the Xinjiang Production and Construction Corps (No. 2022DB021).

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.ultsonch.2022.106219.

References

[1] Q.H. Gao, C.S. Wu, M. Wang, The jujube (*Ziziphus Jujuba* Mill.) fruit: a review of current knowledge of fruit composition and health benefits, J. Agric. Food Chem. 61 (2013) 3351–3363.
[2] Y. Lu, T. Bao, J. Mo, J. Ni, W. Chen, Research advances in bioactive components and health benefits of jujube (*Ziziphus jujuba* Mill.) fruit, J. Zhejiang Univ-Sci. B 22 (2021) 431–449.
[3] B. Deng, M. Guo, H. Liu, S. Tian, X. Zhao, Inhibition of autophagy by hydroxychloroquine enhances antioxidant nutrients and delays postharvest fruit senescence of *Ziziphus jujuba*, Food Chem. 296 (2019) 56–62.
[4] J. Bi, Q. Chen, Y. Zhou, X. Liu, X. Wu, R. Chen, Optimization of short- and medium-wave infrared drying and quality evaluation of jujube powder, Food Bioprocess Technol. 7 (2014) 2375–2387.
[5] A. Wojdylo, A. Figiel, P. Legua, K. Lech, A.A. Carbonell-Barrachina, F. Hernandez, Chemical composition, antioxidant capacity, and sensory quality of dried jujube fruits as affected by cultivar and drying method, Food Chem. 207 (2016) 170–179.
[6] Q.H. Gao, C.S. Wu, M. Wang, B.N. Xu, L.J. Du, Effect of drying of jujubes (*Ziziphus jujuba* Mill.) on the contents of sugars, organic acids, α-tocopherol, β-carotene, and phenolic compounds, J. Agric. Food Chem. 60 (2012) 9642–9648.
[7] J. Song, Q. Chen, J. Bi, X. Meng, X. Wu, Y. Qiao, Y. Lyu, 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 (2020), 127201.
[8] L.Z. Deng, A.S. Mujumdar, Q. Zhang, X.H. Yang, J. Wang, Z.A. Zheng, Z.J. Gao, H. W. Xiao, Chemical and physical pretreatments of fruits and vegetables: effects on drying characteristics and quality attributes - a comprehensive review, Crit. Rev. Food Sci. Nutr. 59 (2019) 1408–1432.
[9] H.O. Wang, Q.Q. Fu, S.J. Chen, Z.C. Hu, H.X. Xie, Effect of hot-water blanching pretreatment on drying characteristics and product qualities for the novel integrated freeze-drying of apple slices, J. Food Qual. 2018 (2018) 1347513.
[10] A. Krzykowski, D. Dziki, S. Rudy, U. Gawlik-Dziki, R. Polak, B. Biernacka, Effect of pre-treatment conditions and freeze-drying temperature on the process kinetics and physicochemical properties of pepper, LWT Food Sci. Technol. 98 (2018) 25–30.
[11] X. Xu, L. Zhang, Y. Feng, C. Zhou, A.E.A. Yagoub, H. Wahia, H. Ma, J. Zhang, Y. Sun, Ultrasound freeze-thawing style pretreatment to improve the efficiency of the vacuum freeze-drying of okra (*Abelmoschus esculentus* (L.) Moench) and the quality characteristics of the dried product, Ultrason. Sonochem. 70 (2021), 105300.
[12] B.A. Niemira, Cold plasma decontamination of foods, Annu. Rev. Food Sci. Technol. 3 (2012) 125–142.
[13] I. Oey, M. Lille, A. Van Loey, M. Hendrickx, Effect of high-pressure processing on colour, texture and flavour of fruit- and vegetable-based food products: a review, Trends Food Sci. Technol. 19 (2008) 320–328.
[14] S. Wang, Y. Liu, Y. Zhang, X. Lü, L. Zhao, Y. Song, L. Zhang, H. Jiang, J. Zhang, W. Ge, Processing sheep milk by cold plasma technology: impacts on the microbial inactivation, physicochemical characteristics, and protein structure, LWT Food Sci. Technol. 153 (2022), 112573.
[15] L. Zhang, L. Liao, Y. Qiao, C. Wang, D. Shi, K. An, J. Hu, Effects of ultrahigh pressure and ultrasound pretreatments on properties of strawberry chips prepared by vacuum freeze drying, Food Chem. 303 (2020), 125386.
[16] L. Garcia-Gonzalez, A.H. Geeraerd, S. Spilimbergo, K. Elst, L. Van Ginneken, J. Debevere, J.F. Van Impe, F. Devlieghere, High pressure carbon dioxide inactivation of microorganisms in foods: the past, the present and the future, Int. J. Food Microbiol. 117 (2007) 1–28.
[17] H. Liao, X. Hu, X. Liao, F. Chen, J. Wu, Inactivation of *Escherichia coli* inoculated into cloudy apple juice exposed to dense phase carbon dioxide, Int. J. Food Microbiol. 118 (2007) 126–131.
[18] X. Bi, J. Wu, Y. Zhang, Z. Xu, X. Liao, High pressure carbon dioxide treatment for fresh-cut carrot slices, Innov. Food Sci. Emerg. Technol. 12 (2011) 298–304.
[19] T. Bao, X. Hao, M.R.I. Shishir, N. Karim, W. Chen, Cold plasma: an emerging pretreatment technology for the drying of jujube slices, Food Chem. 337 (2021), 127783.
[20] G.I. Denoya, S.R. Vaudagna, G. Polenta, Effect of high pressure processing and vacuum packaging on the preservation of fresh-cut peaches, LWT Food Sci. Technol. 62 (2015) 801–806.
[21] Y.Y. Liu, Y. Wang, W.Q. Lv, D. Li, L.J. Wang, Freeze-thaw and ultrasound pretreatment before microwave combined drying affects drying kinetics, cell structure and quality parameters of *Platycodon grandiflorum*, Ind. Crop. Prod. 164 (2021), 113391.
[22] G. Rajkumar, S. Shanmugam, M.D.S. Galvão, M.T.S. Leite Neta, R.D. Dutra Sandes, A.S. Mujumdar, N. Narain, Comparative evaluation of physical properties and aroma profile of carrot slices subjected to hot air and freeze drying, Dry. Technol. 35 (2017) 699–708.
[23] M.S.M. Wee, A.T. Goh, M. Stieger, C.G. Forde, Correlation of instrumental texture properties from textural profile analysis (TPA) with eating behaviours and macronutrient composition for a wide range of solid foods, Food Funct. 9 (2018) 5301–5312.
[24] Q. Chen, J. Song, J. Bi, X. Meng, X. Wu, Characterization of volatile profile from ten different varieties of Chinese jujubes by HS-SPME/GC–MS coupled with E-nose, Food Res. Int. 105 (2018) 605–615.

[25] R. Wang, S. Ding, D. Zhao, Z. Wang, J. Wu, X. Hu, Effect of dehydration methods on antioxidant activities, phenolic contents, cyclic nucleotides, and volatiles of jujube fruits, Food Sci. Biotechnol. 25 (2016) 137–143.
[26] Y. Zhang, X. Sun, S.K. Vidyarthi, R. Zhang, Active components and antioxidant activity of thirty-seven varieties of Chinese jujube fruits (*Ziziphus jujuba* Mill.), Int. J. Food Prop. 24 (2021) 1479–1494.
[27] H. Zhang, L. Jiang, S. Ye, Y. Ye, F. Ren, Systematic evaluation of antioxidant capacities of the ethanolic extract of different tissues of jujube (*Ziziphus jujuba* Mill.) from China, Food Chem. Toxicol. 48 (2010) 1461–1465.
[28] C. Wang, D. Cheng, J. Cao, W. Jiang, Antioxidant capacity and chemical constituents of Chinese jujube (*Ziziphus jujuba* Mill.) at different ripening stages, Food Sci. Biotechnol. 22 (2013) 639–644.
[29] T.G. Nam, D.O. Kim, S.H. Eom, Effects of light sources on major flavonoids and antioxidant activity in common buckwheat sprouts, Food Sci. Biotechnol. 27 (2018) 169–176.
[30] X. Mohammadi, Y. Deng, G. Matinfar, A. Singh, R. Mandal, A. Pratap-Singh, Impact of three different dehydration methods on nutritional values and sensory quality of dried broccoli, oranges, and carrots, Foods 9 (2020) 1464.
[31] A. Tarafdar, N.C. Shahi, A. Singh, R. Sirohi, Artificial neural network modeling of water activity: a low energy approach to freeze drying, Food Bioprocess Technol. 11 (2018) 164–171.
[32] G. Yildiz, G. Izli, The effect of ultrasound pretreatment on quality attributes of freeze-dried quince slices: physical properties and bioactive compounds, J. Food Process Eng. 42 (2019), e13223.
[33] A. Ciurzyńska, J. Falacińska, H. Kowalska, J. Kowalska, S. Galus, A. Marzec, E. Domian, The effect of pre-treatment (blanching, ultrasound and freezing) on quality of freeze-dried red beets, Foods 10 (2021) 132.
[34] A.F. Sanchez-Maldonado, A. Lee, J.M. Farber, Methods for the control of foodborne pathogens in low-moisture foods, Annu. Rev. Food Sci. Technol. 9 (2018) 177–208.
[35] A.J. Fontana, Water activity: why it is important for food safety, Int. Conference Food Safety (1998) 177–185.
[36] J.F. Bi, X. Wang, Q.Q. Chen, X. Liu, X.Y. Wu, Q. Wang, J. Lv, A.J. Yang, Evaluation indicators of explosion puffing Fuji apple chips quality from different Chinese origins, LWT Food Sci. Technol. 60 (2015) 1129–1135.
[37] J.G. Ling, X.T. Xuan, N. Yu, Y. Cui, H.T. Shang, X.J. Liao, X.D. Lin, J.F. Yu, D.H. Liu, High pressure-assisted vacuum freeze drying: a novel, efficient way to accelerate moisture migration in shrimp processing, J. Food Sci. 85 (2020) 1167–1176.
[38] N.R.S. Hulle, P.S. Rao, Effect of high pressure pretreatments on structural and dehydration characteristics of aloe vera (*Aloe barbadensis* Miller) cubes, Dry. Technol. 34 (2016) 105–118.
[39] K.J. Mothibe, M. Zhang, J. Nsor-atindana, Y.C. Wang, Use of ultrasound pretreatment in drying of fruits: drying rates, quality attributes, and shelf life extension, Dry. Technol. 29 (2011) 1611–1621.
[40] D. Zhao, Y. Wang, Y. Zhu, Y. Ni, Effect of carbonic maceration pre-treatment on the drying behavior and physicochemical compositions of sweet potato dried with intermittent or continuous microwave, Dry. Technol. 34 (2016) 1604–1612.
[41] C. Niamnuy, S. Devahastin, S. Soponronnarit, Some recent advances in microstructural modification and monitoring of foods during drying: a review, J. Food Eng. 123 (2014) 148–156.
[42] H.W. Xiao, Z.J. Gao, The application of scanning electron microscope (SEM) to study the microstructure changes in the field of agricultural products drying, in: V. Kazmiruk (Ed.), Scanning electron Microscopy, IntechOpen, Rijeka, Croatia, 2012, pp. 213–226.
[43] Z. Xu, Y. Guo, S. Ding, K. An, Z. Wang, Freezing by immersion in liquid CO_2 at variable pressure response surface analysis of the application to carrot slices freezing, Innov. Food Sci. Emerg. Technol. 22 (2014) 167–174.
[44] Y. Feng, X. Yu, A.E.A. Yagoub, B. Xu, B. Wu, L. Zhang, C. Zhou, Vacuum pretreatment coupled to ultrasound assisted osmotic dehydration as a novel method for garlic slices dehydration, Ultrason. Sonochem. 50 (2019) 363–372.
[45] T. Bao, X. Hao, M.R.I. Shishir, N. Karim, W. Chen, Green alternative methods for pretreatment of whole jujube before the drying process, J. Sci. Food Agric. 102 (2022) 1030–1039.
[46] F.M. Silva, C.L.M. Silva, Colour changes in thermally processed cupuacu (*Theobroma grandiflorum*) puree: critical times and kinetics modelling, Int. J. Food Sci. Technol. 34 (1999) 87–94.
[47] M. Nowacka, M. Dadan, U. Tylewicz, Current applications of ultrasound in fruit and vegetables osmotic dehydration processes, Appl. Sci. Basel 11 (2021) 1269.
[48] Y. Deng, Y. Zhao, Effect of pulsed vacuum and ultrasound osmopretreatments on glass transition temperature, texture, microstructure and calcium penetration of dried apples (Fuji), LWT Food Sci. Technol. 41 (2008) 1575–1585.
[49] D. Fan, B. Chitrakar, R. Ju, M. Zhang, Effect of ultrasonic pretreatment on the properties of freeze-dried carrot slices by traditional and infrared freeze-drying technologies, Dry. Technol. 39 (2021) 1176–1183.
[50] D. Shi, C. Yin, X. Feng, R. Zhou, X. Fan, Y. Qiao, F. Yao, W. Huang, Y. Liu, H. Gao, W. Cheng, Effect of ultrasound and cellulase pre-treatment on the water distribution, physical properties, and nutritional components of *Lentinula edodes* chips, Food Bioprocess Technol. 13 (2020) 625–636.
[51] Y. Xia, Y. Liu, J. Wang, Q. Shuang, Assessment of key aroma compounds in fresh jujube brandy by GC-O-MS and odor activity value, J. Food Process. Preserv. 44 (2020), e14494.
[52] T. Li, T. Jiang, N. Liu, C. Wu, H. Xu, H. Lei, Biotransformation of phenolic profiles and improvement of antioxidant capacities in jujube juice by select lactic acid bacteria, Food Chem. 339 (2021), 127859.
[53] Y. Pu, T. Ding, R. Lv, H. Cheng, D. Liu, Effect of drying and storage on the volatile compounds of jujube fruit detected by electronic nose and GC-MS, Food Sci. Technol. Res. 24 (2018) 1039–1047.
[54] Y. Liu, Y. Sang, J. Guo, W. Zhang, T. Zhang, H. Wang, S. Cheng, G. Chen, Analysis of volatility characteristics of five jujube varieties in Xinjiang Province, China, by HS-SPME-GC/MS and E-nose, Food Sci. Nutr. 9 (2021) 6617–6626.
[55] F. Yuan, X. Fu, X. Yu, Q. Yang, H. Jin, L. Zhu, Comparative analysis and development of a flavor fingerprint for volatile compounds of vegetable soybean seeds based on headspace-gas chromatography-ion mobility spectrometry, Front. Plant Sci. 12 (2021), 768675.
[56] M.C.S. Aguiar, F.O. Silvério, G.P. de Pinho, P.S.N. Lopes, P.H. Fidêncio, S. J. Ventura, Volatile compounds from fruits of Butia capitata at different stages of maturity and storage, Food Res. Int. 62 (2014) 1095–1099.
[57] S. Rapior, G. Konska, J. Guillot, C. Andary, J.M. Bessiere, Volatile composition of *Laetiporus sulphureus*, Cryptogam. Mycol. 21 (2000) 67–72.
[58] J. Li, C. Qian, T. Duan, T. Cai, Z. Xiang, Determination of the volatiles in *Rosa chinensis* cultivars by comprehensive two-dimensional gas chromatography (GC × GC) and quadrupole time-of-flight (QTOF) mass spectrometry (MS), Anal. Lett. 54 (2021) 573–589.
[59] M. Parker, A.P. Pollnitz, D. Cozzolino, I.L. Francis, M.J. Herderich, Identification and quantification of a marker compound for 'pepper' aroma and flavor in Shiraz grape berries by combination of chemometrics and gas chromatography−mass spectrometry, J. Agric. Food Chem. 55 (2007) 5948–5955.
[60] Q. Li, Y. Li, Y. Luo, L. Xiao, K. Wang, J. Huang, Z. Liu, Characterization of the key aroma compounds and microorganisms during the manufacturing process of Fu brick tea, LWT Food Sci. Technol. 127 (2020), 109355.
[61] D. Zhou, T. Li, K. Cong, A. Suo, C. Wu, Influence of cold plasma on quality attributes and aroma compounds in fresh-cut cantaloupe during low temperature storage, LWT Food Sci. Technol. 154 (2022), 112893.
[62] H. Niu, L. Yuan, H. Zhou, Y. Yun, J. Li, J. Tian, K. Zhong, L. Zhou, Comparison of the effects of high pressure processing, pasteurization and high temperature short time on the physicochemical attributes, nutritional quality, aroma profile and sensory characteristics of passion fruit puree, Foods 11 (2022) 632.
[63] C. Wang, L. Zhang, Y. Qiao, L. Liao, D. Shi, J. Wang, L. Shi, Effects of ultrasound and ultra-high pressure pretreatments on volatile and taste compounds of vacuum freeze dried strawberry slice, LWT Food Sci. Technol. 160 (2022), 113232.
[64] F. De Marchi, E. Aprea, I. Endrizzi, M. Charles, E. Betta, M.L. Corollaro, M. Cappelletti, G. Ferrentino, S. Spilimbergo, F. Gasperi, Effects of pasteurization on volatile compounds and sensory properties of coconut (*Cocos nucifera* L.) water: thermal vs. high-pressure carbon dioxide pasteurization, Food Bioprocess Technol. 8 (2015) 1393–1404.
[65] W. Schwab, R. Davidovich-Rikanati, E. Lewinsohn, Biosynthesis of plant-derived flavor compounds, Plant J. 54 (2008) 712–732.
[66] O.J. Caleb, K. Ilte, W.B. Herppich, M. Geyer, P.V. Mahajan, Impacts of minimal processing and hot water dipping of 'Sonata' strawberries on volatiles emitted during storage, Sci. Hortic. 243 (2019) 385–391.
[67] S. Bi, S. Sun, F. Lao, X. Liao, J. Wu, Gas chromatography-mass spectrometry combined with multivariate data analysis as a tool for differentiating between processed orange juice samples on the basis of their volatile markers, Food Chem. 311 (2020), 125913.
[68] F. Chen, M. Zhang, S. Devahastin, D. Yu, Comparative evaluation of the properties of deep-frozen blueberries dried by vacuum infrared freeze drying with the use of CO_2 laser perforation, ultrasound, and freezing-thawing as pretreatments, Food Bioprocess Technol. 14 (2021) 1805–1816.
[69] M. Ahmed, J.B. Eun, Flavonoids in fruits and vegetables after thermal and nonthermal processing: a review, Crit. Rev. Food Sci. Nutr. 58 (2018) 3159–3188.
[70] S.N. Zhao, O.D. Baik, Y.J. Choi, S.M. Kim, Pretreatments for the efficient extraction of bioactive compounds from plant-based biomaterials, Crit. Rev. Food Sci. Nutr. 54 (2014) 1283–1297.
[71] L. Xu, A.L. Garner, B. Tao, K.M. Keener, Microbial inactivation and quality changes in orange juice treated by high voltage atmospheric cold plasma, Food Bioprocess Technol. 10 (2017) 1778–1791.
[72] N.N. Misra, S.K. Pankaj, J.M. Frias, K.M. Keener, P.J. Cullen, The effects of nonthermal plasma on chemical quality of strawberries, Postharvest Biol. Technol. 110 (2015) 197–202.
[73] O. Rodriguez, W.F. Gomes, S. Rodrigues, F.A.N. Fernandes, Effect of indirect cold plasma treatment on cashew apple juice (*Anacardium occidentale* L.), LWT – Food Sci. Technol. 84 (2017) 457–463.
[74] N.K. Mahnot, C.L. Mahanta, K.M. Keener, N.N. Misra, Strategy to achieve a 5-log *Salmonella* inactivation in tender coconut water using high voltage atmospheric cold plasma (HVACP), Food Chem. 284 (2019) 303–311.
[75] E. Xanthakis, E. Gogou, P. Taoukis, L. Ahrné, Effect of microwave assisted blanching on the ascorbic acid oxidase inactivation and vitamin C degradation in frozen mangoes, Innov. Food Sci. Emerg. Technol. 48 (2018) 248–257.
[76] J.C. Cyong, K. Hanabusa, Cyclic adenosine monophosphate in fruits of *Zizyphus jujuba*, Phytochemistry 19 (1980) 2747–2748.
[77] S. Guo, J.A. Duan, Y. Zhang, D. Qian, Y. Tang, Z. Zhu, H. Wang, Contents changes of triterpenic acids, nucleosides, nucleobases, and saccharides in jujube (*Ziziphus jujuba*) fruit during the drying and steaming process, Molecules 20 (2015) 22329–22340.
[78] X. Kou, Q. Chen, X. Li, M. Li, C. Kan, B. Chen, Y. Zhang, Z. Xue, Quantitative assessment of bioactive compounds and the antioxidant activity of 15 jujube cultivars, Food Chem. 173 (2015) 1037–1044.
[79] C. Wang, R. Wang, C. Fu, X. Jiang, X. Li, G. Han, J. Zhang, Combining bioactive compounds and antioxidant activity profiling provide insights into assessment of geographical features of Chinese jujube, Food Biosci. 46 (2022), 101573.
[80] Q. Zhang, L. Wang, Z. Wang, Z. Liu, Z. Zhao, G. Zhou, M. Liu, P. Liu, Variations of the nutritional composition of jujube fruit (*Ziziphus jujuba* Mill.) during maturation stages, Int. J. Food Prop. 23 (2020) 1066–1081.

[81] D.J.M. Peters, M. Cammans, S. Smit, W. Spek, M.M. Van Lookeren Campagne, P. Schaap, Control of cAMP-induced gene expression by divergent signal transduction pathways, Dev. Genet. 12 (1991) 25–34.
[82] T. Klengel, W.J. Liang, J. Chaloupka, C. Ruoff, K. Schröppel, J.R. Naglik, S. E. Eckert, E.G. Mogensen, K. Haynes, M.F. Tuite, L.R. Levin, J. Buck, F. A. Mühlschlegel, Fungal adenylyl cyclase integrates CO_2 sensing with cAMP signaling and virulence, Curr. Biol. 15 (2005) 2021–2026.
[83] M. Amini, M. Ghoranneviss, Effects of cold plasma treatment on antioxidants activity, phenolic contents and shelf life of fresh and dried walnut (*Juglans regia* L.) cultivars during storage, LWT Food Sci. Technol. 73 (2016) 178–184.
[84] L. Liu, Q. Zeng, R. Zhang, Z. Wei, Y. Deng, Y. Zhang, X. Tang, M. Zhang, Comparative study on phenolic profiles and antioxidant activity of litchi juice treated by high pressure carbon dioxide and thermal processing, Food Sci. Technol. Res. 21 (2015) 41–49.
[85] X. Pang, D. Chen, X. Hu, Y. Zhang, J. Wu, Verification of aroma profiles of Jiashi muskmelon juice characterized by odor activity value and gas chromatography-olfactometry/detection frequency analysis: aroma reconstitution experiments and omission tests, J. Agric. Food Chem. 60 (2012) 10426–10432.
[86] F. Li, J. Cao, Q. Liu, X. Hu, X. Liao, Y. Zhang, Acceleration of the Maillard reaction and achievement of product quality by high pressure pretreatment during black garlic processing, Food Chem. 318 (2020), 126517.
[87] M. Perez-Enciso, M. Tenenhaus, Prediction of clinical outcome with microarray data: a partial least squares discriminant analysis (PLS-DA) approach, Hum. Genet. 112 (2003) 581–592.
[88] M.R. Almeida, C.H.V. Fidelis, L.E.S. Barata, R.J. Poppi, Classification of Amazonian rosewood essential oil by Raman spectroscopy and PLS-DA with reliability estimation, Talanta 117 (2013) 305–311.
[89] S. Sun, Y. Zhang, P. Li, H. Xi, L. Wu, J. Zhang, G. Peng, Y. Su, Direct analysis of volatile components from intact jujube by carbon fiber ionization mass spectrometry, BMC Chem. 13 (2019) 125.
[90] Y. Qiao, Q. Chen, J. Bi, X. Wu, X. Jin, M. Gou, X. Yang, G. Purcaro, Investigation of the volatile profile of red jujube by using GC-IMS, multivariate data analysis, and descriptive sensory analysis, Foods 11 (2022) 421.
[91] Y. Song, F. Wang, R. Zhang, S.K. Vidyarthi, X. Sun, Z. Pan, Comparison of volatile compounds and fatty acids of jujubes (*Ziziphus jujuba* mill.) before and after blackening process, Int. J. Food Prop. 25 (2022) 1079–1098.
[92] G. Hui, J. Jin, S. Deng, X. Ye, M. Zhao, M. Wang, D. Ye, Winter jujube (*Zizyphus jujuba* Mill.) quality forecasting method based on electronic nose, Food Chem. 170 (2015) 484–491.
[93] L. Wu, L. Li, G. Zhang, N. Jiang, X. Ouyang, M. Wang, Geographical and varietal traceability of Chinese jujubes based on physical and nutritional characteristics, Foods 10 (2021) 2270.