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\textsuperscript{a,b,c,d}, Fei Lao\textsuperscript{a,b,c,d}, Xun Shi\textsuperscript{e}, Donghao Zhang\textsuperscript{a,b,c,d}, Jihong Wu\textsuperscript{a,b,c,d,*}

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

ARTICLE INFO

Keywords: 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 \cite{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 \cite{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 \cite{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 \cite{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.
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 it 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 [5,7]. 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 [3]. 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 hemiflber 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 CO2 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 jujube fruits 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-packaging 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 (Suyanzhongtian 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 Ultrasonic Cleaner; Kunshan Ultrasonic 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 CO2 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 CO2 outlet. The depressurization 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 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 red, and b* ranges from negative values for green to positive values for red, and b* ranges from negative values for green to positive values for yellow. The total color difference (ΔE) was calculated using the following equation:

\[ \Delta E = \sqrt{\left( L^* - L^*_0 \right)^2 + \left( a^* - a^*_0 \right)^2 + \left( b^* - b^*_0 \right)^2} \]  

where L^*o, a^*o, and b^*o 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:

\[ LRI = 100N + 100(t_RN - t_R)/\left( t_R(t_{n+1}) - t_R(t_n) \right) \]  

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_R is the RT of the compound, t_RN is the RT of n-alkanes immediately before the compound, and t_R(t_{n+1}) 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 KH₂PO₄ and 2.5 mmol/L L-CysH₂BrN (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 mM/L KH₂PO₄ (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
Aw 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 aw and MC of dried jujube slices after different pretreatments. Except for the BC pretreatment, the aw 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 aw values were observed between the US sample and other pretreated samples, except for BC. These results were consistent with the changes in aw 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 aw < 0.85 have not traditionally been considered capable of causing foodborne illness and microbial spoilage, foods with aw < 0.3 are largely protected against lipid oxidation, non-enzymatic browning, and enzymatic activity [34,35]. In the present work, all the samples had aw < 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 aw 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 CO₂, 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...
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 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),

| 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* | 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 ± 0.38b | 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 |
| Britteness (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*, water activity; MC, moisture content; RR, rehydration ratio.
followed by the HHP (3.08), CP (4.15), US (5.13), and BC (6.50) samples. It is generally considered that a ΔF 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\textsubscript{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 compounds 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 were 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-calamorene, and ketoisophorone were detected only in the pretreated samples, suggesting that these components are 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 antioxidative 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\textsubscript{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 treated 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 | LRI1 | Concentration (μg/kg)2 | Identification3 | Aroma descriptors4 |
|-----|--------------------------|-----|------|------------------------|-----------------|-------------------|
|     |                          |     |      | Control | CP | HHP | BC | US | HPCD |                       |                   |
| A1  | Ethyl valerate           | 539-82-2 | 1167 | 8.63 ± 0.29a | 6.45 ± 0.32b | n.d. | n.d. | 8.13 ± n.d. | MS, LRI | Fruity, apple         |
| A2  | Ethyl hexanoate          | 123-66-0 | 1253 | 325.16 ± 33.8b | 348.27 ± 24.02a | 429.35 ± 64.92ab | 62.09 ± 0.61c | 507.17 ± 171.54a | MS, LRI | Fruity, apple, green banana |
| A3  | Ethyl (2)-hex-3-enoate   | 64187-83-3 | 1316 | 39.84 ± 6.51ab | 46.91 ± 1.56a | 21.24 ± 0.48c | n.d. | 46.62 ± 17.66a | MS, LRI | Green, pear           |
| A4  | Ethyl (E)-hex-2-enoate   | 27829-72-7 | 1357 | n.d. | n.d. | n.d. | n.d. | 6.23 ± 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 ed | 8.00 ± 4.92 ed | 16.43 ± 4.32a |
| A6  | Ethyl nonanoate          | 123-29-5 | 1544 | n.d. | 11.09 ± 8.40ab | 9.85 ± 3.76ab | 2.24 ± 0.14bc | 8.00 ± 1.06ab | 2.39a      |
| 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 |
| 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 |
| A9  | 4-Hexanolide             | 695-06-7 | 1709 | n.d. | n.d. | 3.09 ± 0.16b | n.d. | 4.64 ± 0.71a | MS, LRI | Herbal, coconut        |
| A10 | Ethyl undecanoate        | 627-90-7 | 1748 | n.d. | 1.63 ± 0.07a | n.d. | n.d. | 1.24 ± 0.16a | MS, LRI | Soapy, coconut         |
| A11 | Ethyl laurate            | 106-33-7 | 1850 | n.d. | 91.58 ± 39.27ab | 16.44 ± 0.71c | 69.39 ± 13.59b | 114.7 ± 23.94a |
| A12 | Ethyl myristate          | 124-06-1 | 2056 | 2.38 ± 0.65b | 18.52 ± 14.33ab | 29.47 ± 13.26a | 6.06 ± 2.58ab | 18.74 ± 2.92ab | 33.99 ± 6.83a |
| 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 |
| A14 | Ethyl pentadecanoate     | 41114-00-5 | 2157 | n.d. | 2.85 ± 0.06a | 2.33 ± 0.70a | n.d. | 3.94 ± 0.77a | 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 ± 3.81a |
| A16 | Ethyl 9-hexadecanoate    | 54546-22-4 | 2292 | 3.83 ± 1.85a | 47.34 ± 9.59bc | 124.51 ± 41.08a | 14.17 ± 0.74c | 67.37 ± 24.08b | 120.94 ± 27.68a |
|     |                          |     |      |           |               |          |      |    |      |                      |                   |
| Aldehydes | (E)-2-Hexenal      | 6728-26-3 | 1238 | 88.28 ± 10.03a | 48.78 ± 2.71bc | 43.60 ± 8.57bc | 34.87 ± 11.92a | 56.04 ± 10.25b | 41.36 ± 2.37bc |
| B1  | 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 |
| C1  | Benzyalcohol             | 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 |
|     |                          |     |      |           |               |          |      |    |      |                      |                   |
| Terpenes | Ylangene               | 14912-44-8 | 1485 | n.d. | 2.03 ± 0.28a | n.d. | n.d. | n.d. | MS, LRI | Fruity, apple, green banana |
| D1  | Murolone                | 30021-74-0 | 1690 | n.d. | 2.90 ± 0.15a | 1.31 ± 0.04b | n.d. | n.d. | MS, LRI | Herbal, woody          |
| D2  | (+)-β-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 |
| D3  | α-Calacore              | 21391-99-1 | 1923 | n.d. | 1.18 ± 0.15b | 1.44 ± 0.07a | 0.81 ± 0.09c | 1.28 ± 0.03b | n.d. |
| D4  | Phenol                  | 108-95-2 | 2002 | 17.47 ± 11.74ab | 29.93 ± 21.31a | 3.34 ± 2.22b | 25.60 ± 27.33a | 35.90 ± 23.23a | MS, LRI | Phenolic              |
| E1  | Phenol                  | 78-59-1 | 1597 | 8.95 ± 8.87c | 29.83 ± 148.31bc | 983.77 ± 240.61ab | 237.04 ± 49.27d | 942.17 ± 307.43ab | 1070.15 ± 167.45a |
| F1  | Isophorone              | 47-0-5 | 2002 | 17.47 ± 4.36a | 29.93 ± 12.36a | 3.34 ± 0.50a | 25.60 ± 0.50a | 35.90 ± 27.33a | MS, LRI | Green, camphor         |
| G1  | Hexanoic acid           | 142-62-1 | 1845 | 38.18 ± 11.74ab | 50.90 ± 21.31a | n.d. | 22.38 ± 21.31a | n.d. | MS, LRI | Sour                  |

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;
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) difference that 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 [61], while the better retention of cAMP by HPCD may be due to the activating effect of CO₂ 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 latchi 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 α 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

### Table 3

| Bioactive compounds and antioxidant activity of vacuum freeze-dried jujube slices with different pretreatments. |
|---|
| Pretreatment | Control | CP | HHP | BC | US | HPCD |
| TPC (mg/g DW) | 22.49 ± 21.86 | 22.58 ± 19.72 | 22.12 ± 24.55 |
| GAE/AAE (mg/g DW) | 1.58ab | 1.51b | ± | ± | 0.09b | ± 0.47a |
| TFC (mg/g DW) | 20.99 ± 21.99 | 32.23 ± 16.27 | 29.37 ± 37.13 |
| RE/AAE (mg/g DW) | 4.40c ± 2.15abc | ± | ± | 12.21abc | ± |
| AA (mg/g DW) | 9.94 ± 4.52 | 5.94 ± 5.51 | 8.89 ± 10.14 |
| cAMP (mg/g DW) | 0.06a ± 0.01b | 0.03b | ± | ± | 0.15b | ± 0.64a |
| FRAP (mg/g DW) | 40.86 ± 42.81 | 43.63 ± 46.88 | 42.12 ± 48.87 |
| GAE/AAE (mg/g DW) | 9.39a ± 2.91a | ± | ± | 4.23a | ± 2.71a |
| ABTS (mg/g DW) | 51.01 ± 44.24 | 44.52 ± 51.01 | 46.03 ± 55.33 |
| GAE/AAE (mg/g DW) | 3.99ab ± 3.09b | ± | ± | 4.92b | ± 3.10a |
| DPPH (mg/g DW) | 31.73 ± 26.29 | 27.76 ± 30.46 | 30.13 ± 31.24 |
| GAE/AAE (mg/g DW) | 5.01a ± 1.53b | ± 0.95a | ± | ± 1.17a | ± 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. The total antioxidant capacity was characterized in terms of FRAP, ABTS⁺ scavenging activity, and DPPH scavenging capacity. 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. The total antioxidant capacity was characterized in terms of FRAP, ABTS⁺ scavenging activity, and DPPH scavenging capacity. 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. The total antioxidant capacity was characterized in terms of FRAP, ABTS⁺ scavenging activity, and DPPH scavenging capacity. 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.
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, 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 \( R^2 \) and \( Q^2 \) values of the PLS-DA model were 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 (+)-\( \delta \)-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.
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.ultrasint.2022.106219.
Z. Xu, Y. Guo, S. Ding, K. An, Z. Wang, Freezing by immersion in liquid CO₂.
C. Wang, D. Cheng, J. Cao, W. Jiang, Antioxidant capacity and chemical.
H.W. Xiao, Z.J. Gao, The application of scanning electron microscope (SEM) to.
C. Niamnuy, S. Devahastin, S. Soponronnarit, Some recent advances in.
A. Ciurzyńska, H. Kowalska, J. Kowalska, S. Galus, A. Marzec, E. Domian, The effect of pre-treatment (blanching, ultrasound and freezing) on.
A.F. Sanchez-Maldonado, A. Lee, J.M. Farber, Methods for the control of foodborne.
A.J. Fontana, Water activity: why it is important for food safety, Int. Conference.
yield: why it is important for food safety, Int. Conference.
T.G. Nam, D.O. Kim, S.H. Eom, Effects of light sources on major flavonoids and.
M. Nowacka, M. Dadan, U. Tylewicz, Current applications of ultrasound in fruit.
T. Bao, X. Hao, M.R.I. Shishir, N. Karim, W. Chen, Green alternative methods for.
Y. Feng, X. Yu, A.E.A. Yagoub, B. Xu, B. Wu, L. Zhang, C. Zhou, Vacuum.
R. Wang, S. Ding, D. Zhao, Z. Wang, J. Wu, X. Hu, Effect of dehydration methods on.
Y. Zhang, X. Sun, S.K. Vidyarthi, R. Zhang, Active components and antioxidant.
A.J. Yang, Evaluation of freeze-dried quince slices: physical properties and bioactive compounds, J. Food.
F. De Marchi, E. Aprea, I. Endrizzi, M. Charles, E. Betta, M.L. Corollaro, Effects of pasteurization.
S. Nantharatanakul, S. Momwong, P. Areecharoensak, The effect of pretreatment on.
A. Marzec, E. Domian, The effect of pre-treatment (blanching, ultrasound and freezing) on.
Y. Li, Y. Yang, S. Ding, K. An, Z. Wang, H. Wang, S. Cheng, G. Chen, Analysis of.
Y. Liu, Y. Sang, J. Guo, W. Zhang, T. Zhang, H. Wang, S. Cheng, G. Chen, Analysis of.
F. Zhao, D. Liu, Effect of freezing and storage on the volatile compounds of jujube fruit detected by.
Y. Liu, X. Fu, X. Yu, Y. Yang, H. Jin, L. Zhu, Comparative analysis and.
Y. Yuan et al. The effect of ultrasound pretreatment on quality attributes of freeze-dried quince slices: physical properties and bioactive compounds, J. Food.
Y. Zhang, X. Sun, S.K. Vidyarthi, R. Zhang, Active components and antioxidant.
A. Ciurzyńska, H. Kowalska, J. Kowalska, S. Galus, A. Marzec, E. Domian, The effect of pre-treatment (blanching, ultrasound and freezing) on.
A.F. Sanchez-Maldonado, A. Lee, J.M. Farber, Methods for the control of foodborne.
A.J. Fontana, Water activity: why it is important for food safety, Int. Conference.
T.G. Nam, D.O. Kim, S.H. Eom, Effects of light sources on major flavonoids and.
M. Nowacka, M. Dadan, U. Tylewicz, Current applications of ultrasound in fruit.
[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. Schrippel, 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 CO2 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.