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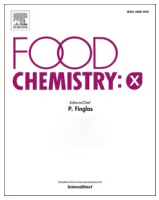

Effects of ultrasonication and freeze–thaw pretreatments on the vacuum freeze-drying process and quality characteristics of apricot (*Prunus armeniaca* L. cv. Diaoganxing)

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ABSTRACT

The combination of pretreatment and vacuum freeze-drying (VFD) technology is an effective technique for extending the shelf life of apricots, reducing costs and energy consumption. However, the impact of pretreatment on the freeze-drying and quality characteristics of apricots is still unclear. The effects of ultrasound (US), freeze-thaw (FT), and their combination (FT-US) on water migration and quality characteristics of apricot slices on VFD were studied. LR-NMR and SEM showed that pretreatment significantly reduced the time (19.05%–33.33%) and energy consumption (17.67%–35.66%) of the VFD process. Compared with the control group, the US, FT, and FT-US improved the color, texture, rehydration ability, and flavor of apricot slices. Among them, FT-US retained the most biologically active substances and antioxidant capacity, with the highest sensory score. Overall, FT-US pretreatment induced changes in the microstructure and chemistry of apricots, which contributed to the production of high-quality VFD apricot slices.

1. Introduction

Apricot (*Prunus armeniaca* L. cv. Diaoganxing) belongs to the subfamily *Amygdaloideae* of the *Rosaceae* family and is widely cultivated in Xinjiang, China (Yang et al., 2021). Apricot is rich in polyphenols, flavonoids, carotenoids, vitamins, and other health-related compounds, all of which contribute to its unique taste, color, nutrition, and functional characteristics (Wani, Masoodi, Ahmad, & Mir, 2018). However, apricot is prone to post-harvest softening, browning, and decay, severely restricting its transportability and commercial value (Gabioud Rebeaud, Cioli, Cotter, & Christen, 2023). Accordingly, appropriate processing methods that maintain the post-harvest quality of apricot and its market value are required.

Currently, drying is widely used in the field of food processing and is considered one of the most effective methods for retaining the nutritional components of fruit (Liu et al., 2022). The commonly used methods for drying apricots include natural drying, hot-air drying (HAD), microwave drying, and vacuum freeze-drying (VFD) (Hu et al., 2022). Owing to differences in their heat-transfer mechanisms, each drying method has its own advantages and disadvantages. HAD is the most widely used method, but due to its high temperature and the presence of oxygen, the bioactive substances in the fruit are prone to degradation. In contrast, VFD is performed under low-temperature conditions and under vacuum, which maximizes the retention of color, flavor, texture characteristics, and nutrients in the dried product (Cao, Zhang, Mujumdar, Zhong, & Wang, 2018). However, VFD technology has the disadvantages of long drying times and high energy consumption, limiting its widespread application in industrial production. To overcome the above problems, pretreatment methods such as blanching, ultrasound (US), ultrahigh pressure, freeze-thawing (FT), pulsed electric fields, and permeation are often used prior to VFD to improve drying rates and the quality of the dried products (Deng et al., 2019).

In recent years, non-thermal US treatment has been widely explored. In this method, the material undergoes contraction and expansion under the action of ultrasonic compression and tensile stresses, resulting in the formation of an internal spongiform structure and microscopic channels. This makes the water inside the material easier to transfer to the outside atmosphere, thereby improving the dehydration efficiency of the

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process, US treatment also maintains the original nutrient elements of the material (Waghmare et al., 2023). Accordingly, US pretreatment is widely used in the VFD of fruits and vegetables, such as carrot slices (Fan, Chitrakar, Ju, & Zhang, 2021), barley grass (Cao et al., 2018), and strawberry slices (Xu Chen, Sylvain Tiliwa, et al., 2021). For instance, Fan et al. (2021) reported that high-power US destroys the cell walls of carrots, forming pore structures and reducing the VFD time by 15%; while Xu, Chen, Sylvain Tiliwa, et al. (2021) reported that sequential pretreatment with dual frequency US significantly improves the color, flavor, and nutrient characteristics of freeze-dried strawberry slices. FT is another effective non-thermal pretreatment method that has been widely used for meat products (Jiang, Nakazawa, Hu, Osako, & Okazaki, 2019), fruits and vegetables (Bassey, Cheng, & Sun, 2022; Silva, Ramirez, Gavidia, & Miano, 2021). During the FT pretreatment process, the material is cooled, converting the water in the cells to ice crystals, which are then melted upon warming, resulting in more intracellular space and cavities in the cell structure (Silva et al., 2021). Xu, Zhang, Feng, et al. (2021) reported that FT pretreatment before VFD changes the microstructure of okra and shortens its drying time by 62.5%, and the dry-matter-content loss is only ~5.6%. The above studies showed that both US and FT pretreatment can be applied to dried products. However, in the field of fruit and vegetable processing, FT pretreatment is mostly applied to VFD of vegetables (Xu, Zhang, Yagoub, et al., 2021), seasonings (Feng et al., 2020), and small volume berries (Wang et al., 2023), and is rarely used in the processing of forest fruits and other fruits. In addition, previous applications have mostly used US and FT single pretreatment, and there are few reports on the combined application of the two methods in VFD processing. So we studied the combination of two pretreatment methods in VFD apricots to reduce the energy consumption and improve product quality.

Therefore, this study addressed (a) the effects of US, FT, and FT-US pretreatments on drying process of VFD apricot slices; (b) the effects of these pretreatments on the microstructure and water distribution of VFD apricot slices; and (c) the effects of the pretreatments on the color, texture, flavor, and bioactive substances of VFD apricot slices. Our overall findings provide an effective pretreatment strategy for the production of high-quality VFD apricot slices.

2. Materials and methods

2.1. Raw materials

Fresh apricots (*Prunus armeniaca* L. cv. Diaoganxing) were obtained from the Ili region of Xinjiang (30° 45′N, 105° 58′E) in July 2023, and those with uniform size and color, no mechanical damage, and no pests or diseases were selected as the experimental subjects. The initial moisture content of the apricots was 82% (wet basis), and the average soluble solid content was 24 ± 0.5%. Before pretreatment, the fruit was cleaned, pedicled, cored and cut into slices with a thickness of 6 ± 0.5 mm.

2.2. Pretreatment groups

2.2.1. US

The sealed apricot slices were placed in an ultrasonication device (KQ-400DE, Kunshan Ultrasonic Instrument Co., LTD., China). Based on the results of our previous US pretreatment single factor and response surface optimization experiments, the sealed apricot slices were treated with the optimized parameters of 200 W (40 kHz) ultrasonication for 25 min with 25 ± 0.5 °C water as the medium.

2.2.2. FT

Based on the results of our previous FT pretreatment single factor and response surface optimization experiments, The apricot slices were frozen at −20 ± 0.5 °C until the internal temperature of the sample remained constant (~24 h) (Xu, Zhang, Feng, et al., 2021) then immediately removed and defrosted at 25 ± 0.5 °C until the internal temperature of the sample was constant (Ando et al., 2019; Feng et al., 2020).

2.2.3. FT-US

Apricot slices were FT treated under the conditions stated above, then ultrasonic treatment was performed, also under the conditions stated above.

2.2.4. Control

Apricot slices were subjected to VFD without any pretreatment.

2.3. Vacuum freeze-drying (VFD)

The pretreated apricot slice samples were pre-frozen at −25 °C for 12 h, then transferred to a vacuum freeze-dryer (SCIENTZ-10ND, Ningbo Xinzhi Biotechnology Co., LTD., Zhejiang, China) and dried at 20 °C with a cold trap temperature of −40 °C and a vacuum pressure of 10 Pa. During the drying process, each sample was regularly weighed to determine its moisture content until it was <0.1 g/g, and the final drying time was recorded. The samples were then immediately placed in a sealed bag and stored away from light until analysis.

2.4. Analysis of drying characteristics

2.4.1. Moisture ratio (MR)

Plotting MR against drying time provides the drying curve for VFD apricot slices. MR was calculated according to the method reported by Liu et al. (2021):

$$MR = (M_t - M_e)/(M_0 - M_e) \quad (1)$$

where M_t represents the water content of the apricot slices at time t (dry basis, g/g), M_e is the water content of the apricot slice sample at equilibrium, and M_0 is the initial water content of apricot slices (dry basis, g/g).

2.4.2. Drying rate (DR)

DR refers to the water loss per unit time (Zhang et al., 2020). The expression for DR is:

$$DR = (M_{t1} - M_{t2})/(t_1 - t_2) \quad (2)$$

where M_{t1} and M_{t2} represent the moisture content (dry basis, g/g) of apricot slices at t_1 and t_2, and DR represents the drying rate (g/(g·min)).

2.4.3. Moisture content

The moisture content of the samples was determined by the method proposed by the oven method. ~5 g of apricot slice samples were weighed and then dried in 102 °C (DGG-9053A, Shanghai Sen Xin Experimental Instrument Co., LTD., Shanghai, China) to a constant weight (Cao et al., 2018).

2.4.4. Total energy consumption (TEC)

The TEC of the processing of fresh apricot slices (pretreatment and VFD) was calculated using expression (3) (Zhang, Yu, Arun, & Zhou, 2022):

$$TEC\ (kWh/kg, FW) = E_p + E_d/m \quad (3)$$

where m represents the weight (kg) of the apricot slice samples to be dried, E_p and E_d represent the energy consumption during pretreatment and drying, respectively, as measured by an electricity meter connected to the vacuum freeze-dryer.

2.4.5. LF-NMR analysis

Relaxation times were determined by low-field NMR (PQ001–20-

025 V, Suzhou Nuemai Analytical Instruments Co., LTD., Suzhou, China). Approximately 4 g freeze-dried apricot slice samples were placed in a cylindrical glass tube (diameter 2 cm; height 4 cm) and held at 25 °C for 30 min. Measurement of transverse relaxation time (T2) was performed using the Carl-Purcell-Mebron-Gill pulse train (Zhang et al., 2020). The parameters were as follows: P1 = 5.10 ms; P2 = 9.04 ms; proton corresponding resonance frequency SF = 20 MHz; spectrum width SW = 100 kHz; digital gain DRG1 = 3; waiting time TW = 2000 ms.

2.4.6. Microstructure analysis

The microstructures for each group of apricot slices were observed using SEM with a Sigma 300 field emission-scanning electron microscope (Zeiss GmbH, Germany) at a voltage of 5.0 kV and magnifications of 30× and 200 × .

2.5. Quality characteristics of apricot slices

2.5.1. Rehydration rate (RR)

The RRs of the apricot slices were determined with reference to the method of Bassey et al. (2022) with slight modifications. Briefly, ~5 g of apricot slice samples (R_1) were taken and placed in a beaker containing 500 mL of distilled water at 25 °C for 1 h. Then, the material was removed, wiped dry, and weighed (R_2). The ratio of R_2 to R_1 was taken as the value of RR.

2.5.2. Hardness

The hardness (g) of the apricot slice samples were determined using a texture analyzer (TA-XT plus, Vienna Court, Lammas Rod, Godalming, Surrey GU7 1YL, UK). A P/5 cylindrical probe was used with a testing speed of 1.0 mm/s in the early stage, 3.0 mm/s in the middle stage, and 3.0 mm/s in the later stage. The triggering force and penetration distance were 5 g and 15.0 mm, respectively (Jiang et al., 2017). Each group of samples was analyzed three times, and the results were averaged.

2.5.3. Color parameters

A portable colorimeter (YS3060, Shenzhen 3NH Testing Instrument Co., Ltd., Shenzhen, China) was used to determine the color parameters, L* (lightness), a* (red/green), and b* (yellow/blue), of the apricot slice samples at room temperature (Wu, Guo, Guo, Ma, & Zhou, 2021). ΔE values were determined using:

$$\Delta E = \sqrt{\left(L^* - L^0\right)^2 + \left(a^* - a^0\right)^2 + \left(b^* - b^0\right)^2} \quad (4)$$

where L^0, a^0, and b^0 are the values for the fresh apricot slices. The measurements were repeated three times for each group of samples, and the average value was taken as the result.

2.5.4. Electronic nose analysis

The changes in volatile substance content of the dried apricot samples were determined using an electronic nose (Airsense PEN3, Germany) (Zhang et al., 2020). ~5.00 g apricot slice samples were placed in a 20 mL bottle with a poly (tetrachloroethylene) cap, held at 40 °C for 30 min, and then the headspace gas was injected for testing. The electronic nose was equipped with 10 sensors with serial numbers 1, 2, 3, 4, 5, 6, 7, 8, 9, and 10 representing W1C: aromatic, W5S: broad-range, W3C: aromatic, W6S: hydrogen, W5C: arom-aliph, W1S: broad-methane, WIW: sulfur-organic, W2S: broad-alcohol, W2W: sulph-chlor, and W3S: methane-aliph.

2.5.5. Total carotenoid content

The carotenoid contents of the apricot slices were determined using a carotenoid assay kit (Suzhou Gris Biotechnology Co., LTD., Jiangsu, China) according to the manufacturer's instructions with reference to the sample absorption at 470 nm. The results are expressed in mg/g.

2.5.6. Ascorbic acid (AA) content

The method of Yuan, Lao, Shi, Zhang, and Wu (2022). with some modifications was used to determine the AA contents of the apricot slices Briefly, 1 g of apricot powder was accurately weighed, ground in 5 mL 0.2% metaphosphate cooled in an ice bath, and centrifuged at 8000 rpm for 10 min at 4 °C. The supernatant was removed, and the sold residue was added to 4 mL 0.2% metaphosphate and extracted again in the same manner. The supernatants were combined, made up to 10 mL with 0.2% metaphosphate, and filtered through a 0.45 μm filter membrane.

An HPLC system equipped with an XBridge C18 column (4.6 × 250 mm, 5 μm; ANPEL Laboratory Technologies Inc.) and an ultraviolet detector was employed. The mobile phase consisted of methanol and 50 mmol/L KH_2PO_4 at a flow rate of 0.7 mL/min. AA was detected at 245 nm and quantified using a standard curve. The results are expressed in milligrams AA per g sample (mg AA/g).

2.5.7. Total phenols content (TPC) and total flavonoids content (TFC)

The samples were prepared according to the method of Yuan et al. (2022) with slight modifications. Briefly, 2 g of apricot powder was weighed into a 50 mL beaker, then 20 mL of 70% ethanol solution (*v/v*) was added, and the mixture was agitated in an ultrasonication bath (KQ-400DE, Kunshan Ultrasonic Instrument Co., Ltd., China) for 30 min (100 W, 40 kHz). The resulting mixture was centrifuged at 8000 rpm for 15 min at 4 °C and the supernatant was collected. Extraction of the residue was repeated three times in the same manner. The three supernatants were combined and stored at 4 °C.

TPC was determined by the Folin-Ciocalteu method, and the results are expressed as milligram gallic acid equivalents per g sample (mg GAE/g) (Wu et al., 2021).

TFC was determined by a colorimetric method and the results are expressed as milligram rutin equivalents per g sample (mg RE/g) (Jiang et al., 2017).

2.5.8. Antioxidant capacity

DPPH, ABTS, and FRAP kits (Suzhou Gris Biotechnology Co., LTD., Jiangsu, China) were used to determine the antioxidant capacities of apricot slices *in vitro* according to the manufacturer's instructions using absorption values of 517, 734, and 590 nm, respectively. Standard curves were prepared using Trolox. The results are expressed as milligram Trolox per g sample (mg Trolox/g).

2.6. Sensory evaluation

The sensory evaluation was conducted using the method of Yuan et al. (2022) with slight modifications, and a total of 12 experienced group members from Shihezi University (Xinjiang, Shihezi) were invited for sensory evaluation. The appearance, aroma, taste, texture, and overall quality of apricot slices were evaluated using a nine point hedonic method. All four test samples were provided in random order, and each group member was required to complete three copies of each sample, with the data presented as the average.

2.7. Statistical analysis

The data were statistically analyzed using SPSS 22.0 and Origin Pro 8.0. Pearson correlation test was used to analyze the correlation between bioactive substances and antioxidant capacity. Data are expressed as mean ± standard error ($n = 3$), and $p < 0.05$ is considered significant.

3. Results and discussion

3.1. Drying characteristics

The variation of MR over time is shown in Fig. 1A. MR for all four groups decreases rapidly in the initial drying stage and then more slowly in the later drying stage until reaching the end. The drying times for the

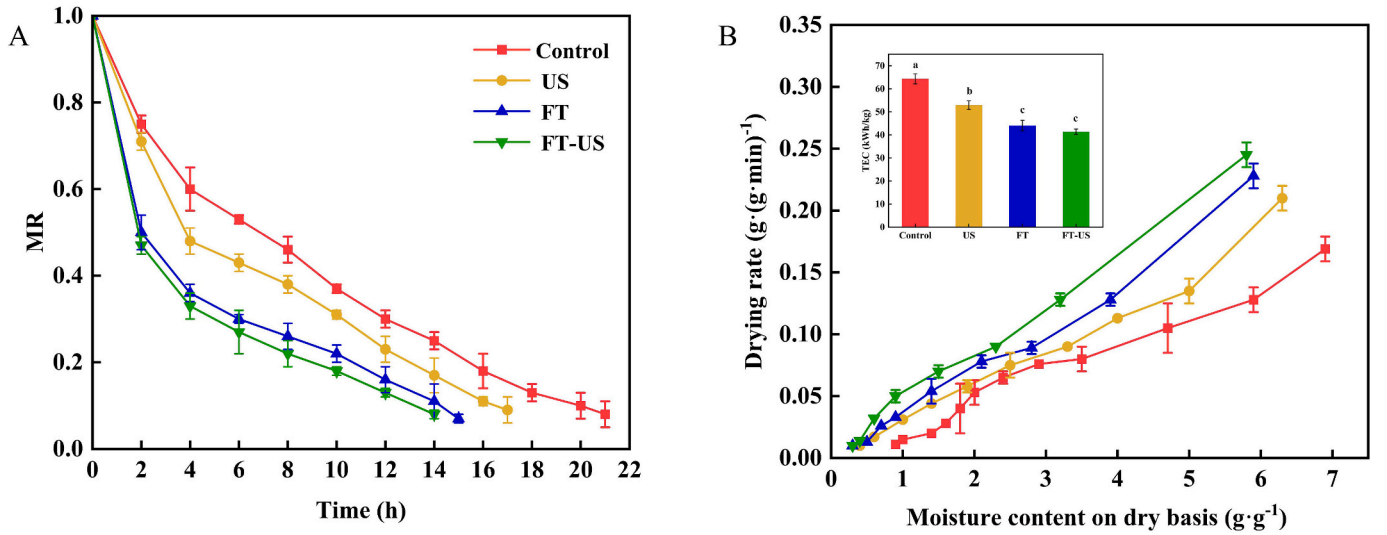

Fig. 1. Drying curves of vacuum freeze-drying (VFD) apricot slices with different pretreatments. The relationship between moisture ratio (MR) and drying time (A); Drying rate in relation to moisture content and total energy consumption (B). Different letters in the same row indicated significant differences between pretreatments ($p < 0.05$).

samples in the control, US, FT, and FT-US groups are 21, 17, 15, and 14 h, respectively. Thus, pretreatment effectively shortens the VFD time for apricot slices. Liu, Wang, et al. (2021) also reported that US and FT effectively reduce the drying time of *Platycodon grandiflorum*. We believe that the rapid decrease in MR in the early stage is due to the rapid removal of free water from the apricot slices by VFD, while the slower decline in the later stage of drying is mainly due to the difficulty in removing the remaining bound and non-flowing water (Xu, Zhang, Feng, et al., 2021). Furthermore, US and FT may affect water distribution in the apricot slices, resulting in significant differences in drying time.

The DR of apricot slices in the pretreatment groups are significantly higher than that in the control group (Fig. 1B), with that for the FT-US group being highest, followed by the FT group and then the US group. We believe that the mechanical and cavitation effects of US treatment cause the apricot slices to shrink and expand repeatedly, forming microscopic pores that facilitate the migration and loss of water. FT treatment also disrupts the cell structure, changes the cell permeability, and is conducive to water diffusion. Separately, both FT and US are effective pretreatment methods, but the FT-US group shows the highest dehydration efficiency, suggesting a strong synergistic effect between the two pretreatment methods. Accordingly, combined treatment has a lower time cost.

The TEC for the control, US, FT, and FT-US groups are 64.33, 52.96, 44.06, and 41.39 kWh/kg (Fig. 1B), respectively. Compared with that of the control group, the TEC of the US, FT, and FT-US treatment groups are decreased by 17.67%, 31.51%, and 35.66%, respectively. Thus, the TEC results also indicate that FT-US is an efficient pretreatment method for low-energy VFD of apricot slices.

3.2. LF-NMR

LF-NMR is an effective method for determining the moisture contents, states, and distributions of samples (Chen et al., 2018). The relaxation times and moisture distributions of the samples are shown in Fig. 2. The peaks appearing at different relaxation times represent water in three different states in the apricot slice samples; T21 (cell-wall-polysaccharide-bound water; 1–10 ms), T22 (non-mobile water interacting with proteins and other macromolecules; 10–100 ms), and T23 (highly mobile free water; 100–1000 ms) (Feng et al., 2020). The water distributions for the four groups are roughly the same, but the relaxation times and peak areas are slightly different. The peak areas for T21 and T22 are relatively high (Table S1). Therefore, combined and non-

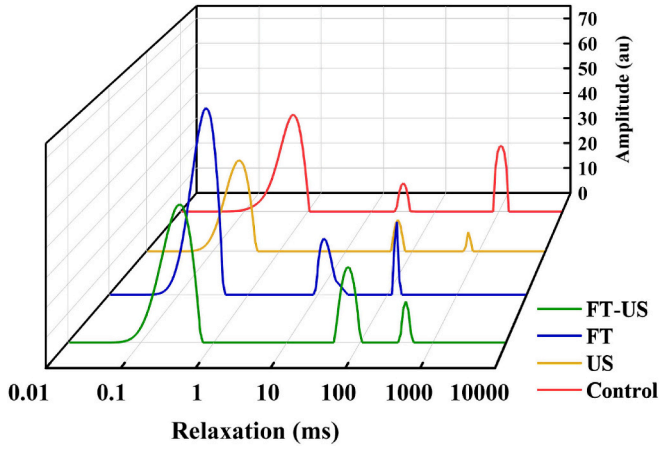

Fig. 2. Low field NMR T2 relaxation time of vacuum freeze-drying (VFD) apricot slices with different pretreatments.

flowing water are the main water components in the dry samples. Compared with the control group, the T23 peaks for the US, FT, and FT-US groups are all shifted to the left, and the A23 peak area is decreased while that of the A22 peak is increased, indicating that pretreatment accelerates the removal of free water, increases the relative content of non-flowing water, and thus reduces water fluidity. This is because during the US and FT pretreatment processes, the cells of the apricot slices are damaged to varying degrees, forming porous structures, enhancing the diffusion of free water in the cells, promoting water migration during the VFD process, accelerating the drying rate (Zhang et al., 2020). And the pretreatment also accelerated the decline rate of MR (Section 3.1). As is well known, fresh apricot slices are mainly composed of free water, so the LF-NMR results were consistent with the MR change results. A study by Zhang et al. (2020) also showed that US pretreatment can effectively shorten the lateral relaxation time of free water in VFD strawberry slices. In summary, US, FT, and FT-US pretreatment all change the state and distribution of water in apricot slices in a manner that decreases drying time and energy consumption.

3.3. Microstructure

Changes in the quality characteristics of dried foods, such as texture, color, and rehydration rate, are closely related to changes in their microstructure during drying (Yuan et al., 2022). SEM images of the samples are shown in Fig. 3. Unlike the control group, the US, FT, and FT-US samples all show porous tissue structures and irregular cell arrangements. These results are consistent with our expectation that pretreatment would change the microstructure of the apricot slices, facilitating water diffusion and improving VFD efficiency. The porous structure observed for the US pretreatment group may be due to the inertial flow and sponge effect caused by ultrasonic cavitation, forming microscopic channels that enhance mass and water transfer (Feng et al., 2019). Furthermore, FT causes the interior of the apricot slices to fill with holes and adopt an extremely irregular tissue arrangement, which is related to the rapid formation of irregular ice crystals during the FT process, which disrupts the internal structure of the cells (Zhang et al., 2022). It is almost impossible to observe a complete cell-wall structure for the FT-US group, and evidence of cell-wall fragmentation can even be observed in the 200× magnified image. This indicates that FT-US treatment causes the greatest degree of damage to the microstructure of apricot slices and confirms the feasibility of combined FT and US treatment. In summary, the pretreatments lead to changes in microstructure that affect the dehydration efficiency of apricot slices. The formation of microchannels, changes in pore size, and an increase in the number of pores all improve the drying rate and shorten the drying time.

3.4. RR

The rehydration characteristics of dried products are important indicators of their quality and the efficiency of the drying process. The higher the RR, the closer the moisture content of the rehydrated fruit is to that of the fresh fruit (Zhou et al., 2021). RR mainly depends on the pore structure of the dry fruit, and the higher the porosity, the better the rehydration effect (Ricce, Rojas, Miano, Siche, & Augusto, 2016). As shown in Fig. 4A, the RR for the US, FT, and FT-US groups are higher than that of the control group, especially that for FT-US (5.58%). US pretreatment promotes the transfer of external moisture to the interior of the sample due to the microchannels generated by ultrasonic cavitation (Yuan et al., 2022), improving the rehydration ability of the dried apricot slices. The formation of ice crystals during FT pretreatment results in disruption of the internal structure of the sample, and the resulting porous network structure also benefits rehydration owing to the capillary effect. Similar findings have been reported for US pretreatment of VFD strawberry slices (Xu, Chen, Sylvain Tiliwa, et al., 2021) and FT-US pretreatment of dried maca (Chen et al., 2018). In conclusion, different pretreatments can improve the rehydration of apricot slices by changing their microstructures, thus improving product quality.

3.5. Hardness

Hardness is an important parameter used to measure the quality of dry products, affecting the taste and overall acceptability of food (Chen et al., 2017). Freeze-dried apricot slices with moderate hardness are more popular with consumers. As shown in Fig. 4B, the hardness of the samples in the control, US, FT, and FT-US groups are 530.63, 612.22, 1356.29, and 980.72 g, respectively. Thus, the hardness of the FT and FT-US samples are significantly higher than that of the control sample ($p < 0.05$). In general, the hardness of a product is determined by a combination of factors such as moisture content and microstructure (Xu, Zhang, Feng, et al., 2021; Zhang et al., 2022). For instance, US pretreatment has been reported to increase the hardness of jujube slices (Yuan et al., 2022), and dried jackfruit (Wu et al., 2021) but decrease the hardness of dried plum (Li et al., 2021). This indicates that the influence of US pretreatment on hardness is related to different treatment conditions (time, frequency, temperature, *etc.*) and plant properties. Zhang et al. (2022) reported that the hardness of lotus root slices increases with the number of FT cycles. Therefore, different pretreatment methods and conditions have different effects on sample hardness. In this study, FT and FT-US pretreatments were observed to increase the hardness of apricot slices. Considering the SEM results, this may be attributed to the stronger disruptive effect of FT on the cell walls, with the irregular cell-wall arrangement resulting in higher sample hardness.

3.6. Color and carotenoids

Color is an essential organoleptic quality for evaluating fresh fruits and vegetables and their dried products (Li et al., 2021). The L*, a*, b*, ΔE values and appearance of the samples are shown in Fig. 4C, D, and we also recorded the metrics for the undried samples as a reference. The results show that the different pretreatments have significant effects on the color of apricot slices, with the L* values of the dried apricot slices increasing ($p < 0.05$) compared with that of the undried apricot slices, indicating that VFD increases the brightness of apricot slices. Similar results have been reported for dried black mulberries (Chen et al., 2017). In addition, compared with the undried apricot slices, the a* and b* values of the dried apricot slices are significantly decreased ($p < 0.05$),

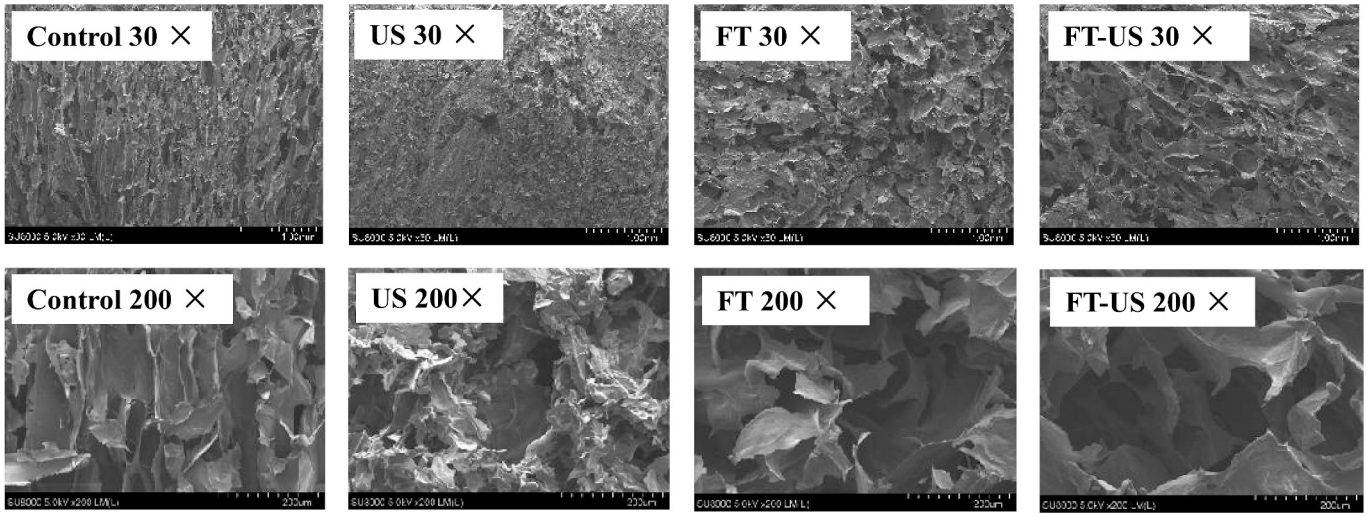

Fig. 3. Scanning electron micrographs of vacuum freeze-drying (VFD) apricot slices with different pretreatments.

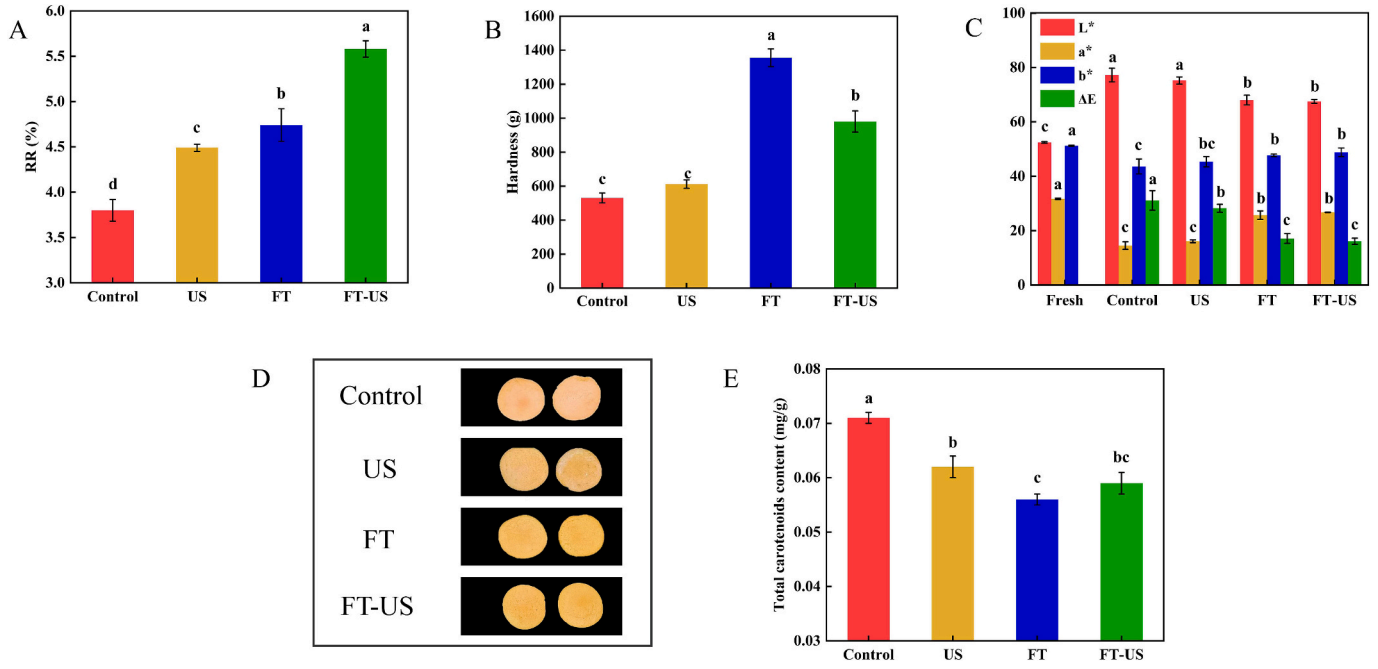

Fig. 4. Rehydration rate of vacuum freeze-drying (VFD) apricot slices with different pretreatments (A); hardness and moisture content (B); color (C); appearance (D) and carotenoid content (E). Different letters in the same row indicated significant differences between pretreatments ($p < 0.05$).

which were presented following the order: FT-US and FT > US > Control, indicating that the VFD process causes color loss of apricot slices. However, FT-US and FT pre-treatment are more helpful in maintaining the original color of apricot slices. The ΔE value of FT-US group is the lowest (16.07), followed by those of the FT (17.10), US (28.24), and control (31.11) groups. Xu, Zhang, Feng, et al. (2021) also found that the FT-US pre-treatment had the smallest color difference in okra compared to the single treatment of US and FT. The less extensive color change for the FT-US group may be related to the shorter drying time required following this pretreatment, better maintaining fruit color (Xu, Chen, Sylvain Tiliwa, et al., 2021).

The characteristic orange-yellow color of apricots is due to carotenoids, which are highly susceptible to isomerization and degradation during the drying process (İncedayi, Tamer, Sinir, Suna, & Çopur, 2016; Nora et al., 2014). The carotenoid contents of the apricot slices in the control, US, FT, and FT-US groups are 0.071, 0.062, 0.056, and 0.059 mg/g, respectively (Fig. 4E). Thus, the different pretreatments result in more extensive degradation of carotenoids during VFD, and this is related to cell damage caused by US and FT (Medeiros et al., 2016). Silva et al. (2016) also reported that US pretreatment results in a decrease in carotenoid content during melon drying. In addition, carotenoid contents are related to ultrasonication time, power, thawing time, and temperature. Liu et al. (2019) reported that high-strength US-assisted thawing of mango puree at 25 °C resulted in a higher carotenoid retention rate compared to that at 4 °C with lower US strength. This was partly attributed to the longer thawing time required at 4 °C, which is more likely to cause carotenoid degradation. However, ultrasound penetration at 25 °C is deeper than that at 4 °C, resulting in more carotenoids being released from the tissue.

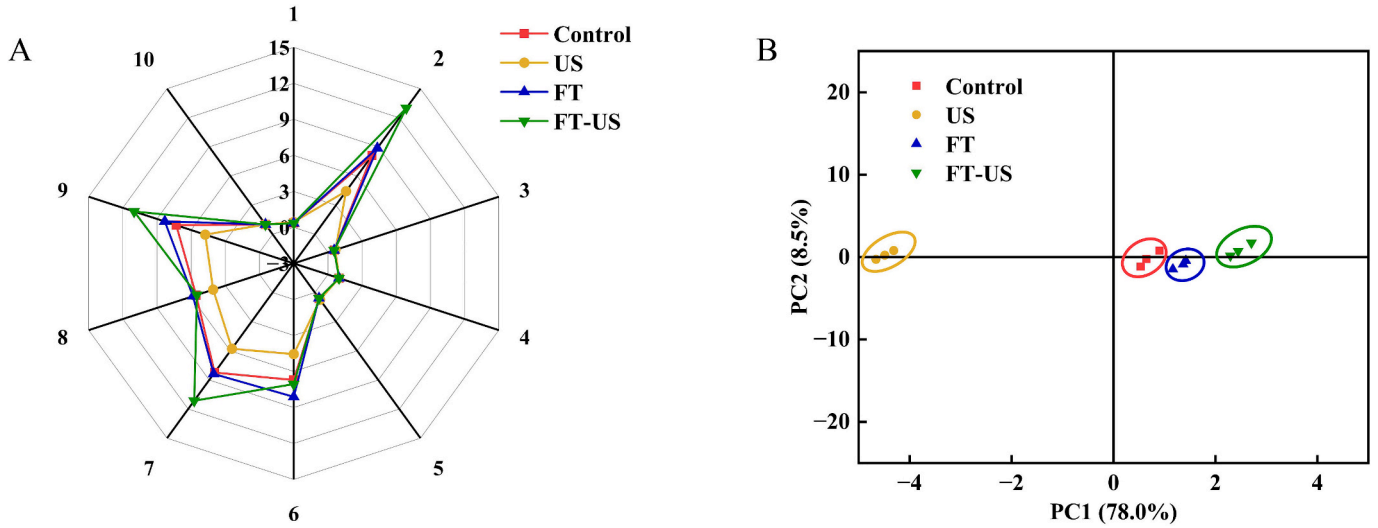

Fig. 5. Radar images (A) and principal component analysis (PCA) (B) of vacuum freeze-drying (VFD) apricot slices with different pretreatments.

3.7. Electronic nose analysis

In order to more intuitively visualize the electronic nose response values for differently pretreated freeze-dried apricot slices, radar images based on the response data are presented (Fig. 5A). The response profile shapes for the different pretreatments are similar, but the signal intensities are different. This may be due to changes in the types and concentrations of characteristic volatile components caused by pretreatments (Boateng & Yang, 2021). As shown in Fig. 5A, the W2W (sensitive to aromatic compounds and organic sulfides), W1W (sensitive to inorganic sulfides), and W5S (sensitive to nitrogen oxides) sensors are most responsive to the apricot slices, followed by the W1S (sensitive to methyl groups) and W2S (sensitive to alcohols and aldehydes) sensors. However, the response values of the W1C, W3S, W6S, W5C, and W3C sensors are not significant, and there are no significant differences between the different pretreatment groups. Similar results have been observed in fruit flavor studies on *ginkgo biloba* (Boateng & Yang, 2021) and strawberry (Zhang et al., 2020). In addition, the sensor response values for the FT-US pretreatment group are generally higher than those for the control, US, and FT groups, indicating that more of the volatile components of apricot slices are retained upon VFD after FT-US pretreatment.

We speculate that FT-US causes greater damage to the microstructure of the sample, resulting in the formation of new volatile substances. Simultaneously, FT-US pretreatment leads to the shortest drying time, which is beneficial to the retention of flavor substances during the drying process (Feng et al., 2020). However, it is difficult to distinguish the different pretreatments from the sizes of the sensor response values alone, so it is necessary to further process the sensor response values to identify the inherent differences between samples.

Principal component analysis (PCA) is an effective dimensionality-reduction method that can preserve the original data information to the maximum extent possible while regrouping observations. The PCA results for the sensor values are shown in Fig. 5B. The contribution rates of the first principal component (PC1) and the second principal component (PC2) are 78% and 8.5%, respectively, and the cumulative variance contribution rate is 86.5% (> 85%), so the two principal components include most of the information regarding the volatile substances in the apricot slice samples. The PCA results for the apricot slices in the control, FT, and FT-US groups are close to each other, indicating that their volatile components are similar. The distance between the US group and the other three groups indicates that they are significantly different, and this result may be related to the loss of nitrogen oxides (W5S); methyl compounds (W1S); sulfides (W1W); alcohols, aldehydes, and ketones (W2S); and organic sulfides (W2W). These results indicate that US pretreatment alone has a negative effect on the flavor of freeze-dried apricot slices, while FT and FT-US pretreatments preserve their flavors.

3.8. Biological activity and antioxidant capacity

3.8.1. AA

AA is heat sensitive and easily decomposes under conditions such as light, heat, and oxygen during the drying of fruits and vegetables (Krzykowski et al., 2018). The AA contents of apricot slices from the different groups are shown in Table 1. The AA content of apricot slices in the control group is 2.50 mg/g, followed by FT-US (2.11 mg/g), US (2.05 mg/g), and FT (1.94 mg/g). Thus, compared with the US and FT pretreatment, the FT-US pretreatment have better AA-retention effects on the apricot slices. The decrease in AA content caused by pretreatments may be related to various factors: (a) US and FT damage the cell wall and vacuole structures of plants, making it easier for AA to come into contact with ascorbic acid oxidase and ascorbic acid peroxidase, leading to AA degradation (Cao et al., 2020); (b) US pretreatment can lead to a large accumulation of free radicals in apricot slices, promoting rapid oxidation of AA (Xu, Chen, Sylvain Tiliwa, et al., 2021); (c) during US, light, heat, and other factors can cause a decrease in AA content (Krzykowski et al., 2018); (d) during FT, AA and other active substances may be lost. In this study, compared to a single pretreatment, the higher retention rate of AA in FT-US pretreatment may be due to cell damage during the FT process (Wang et al., 2023), which helps US extract more AA and offsets some of the loss from a single pretreatment (Belgheisi & EsmaeilZadeh Kenari, 2019). On the other hand, FT-US pretreatment may result in shorter VFD time, leading to better AA retention. Xu, Chen, Sylvain Tiliwa, et al. (2021) demonstrated in their study on freeze-dried strawberry slices that US at different frequencies cause a decrease in AA content. Furthermore, in the studies on infrared and convective drying of lotus root, Zhang et al. (2022) observed that drip loss during FT pretreatment adversely affects AA in lotus root, with the AA retention rate gradually decreasing with the number of FT cycles.

Table 1
Bioactive components (AA, TFC, TPC) and antioxidant activities (DPPH, ABTS, FRAP) of vacuum freeze-drying (VFD) apricot slices with different pretreatment.

	Control	US	FT	FT-US
AA (mg AA/g)	2.50 ± 0.05a	2.05 ± 0.10b	1.94 ± 0.09b	2.11 ± 0.11b
TFC (mg RE/g)	1.44 ± 0.02bc	1.39 ± 0.05c	1.49 ± 0.06b	1.67 ± 0.08a
TPC (mg GAE/g)	7.82 ± 0.12a	7.01 ± 0.24b	7. 57± 0.06ab	7.70 ± 0.17a
DPPH (mg Trolox/g)	0.33 ± 0.05ab	0.31 ± 0.03b	0.34 ± 0.03a	0.33 ± 0.02ab
ABTS (mg Trolox/g)	1.49 ± 0.11bc	1.45 ± 0.09c	1.56 ± 0.12b	1.72 ± 0.12a
FRAP (mg Trolox/g)	6.48 ± 0.21b	6.47 ± 0.29b	6.88 ± 0.13ab	7.12 ± 0.32a

Different letters in the same row indicated significant differences between pretreatments ($p < 0.05$).

3.8.2. TPC and TFC

Phenols and flavonoids are important bioactive components in apricots (Wani et al., 2018). The TPC and TFC values for the different groups are shown in Table 1. The TPC for the control group is 7.82 mg GAE/g, which is not significantly different to those of the FT (7.57 mg GAE/g) and FT-US (7.70 mg GAE/g) groups. However, the US group has a significantly reduced TPC content (7.01 mg GAE/g; $p < 0.05$). Several studies have shown that US treatment is conducive to the extraction of phenolic compounds. This is attributed to the cavitation effect generated by ultrasonic waves, releasing phenolic compounds and deactivating enzymes related to the degradation of phenolic substances (Xu, Chen, Sylvain Tiliwa, et al., 2021). However, improper US treatment times and intensities can also have adverse effects on phenolic compounds (Chu, Wei, Ding, Mei, & Xie, 2021). Therefore, US has both positive and negative effects on the retention of phenolic substances. In this study, the reasons for phenolic changes may be the same as those for AA changes. Xu, Zhang, Feng, et al. (2021) reported that US pretreatment reduces the TPC of vacuum freeze-dried okra; whereas Wang, Xiao, Ye, Wang, and Raghavan (2019) reported that the TPC of kiwifruit slices pretreated with US for 30 min is significantly increased.

In terms of TFC, that of the FT-US group (1.67 mg RE/g) is higher than that of the control group (1.44 mg RE/g; $p < 0.05$), indicating that FT-US has a positive effect on the retention of flavonoids. In the study on the VFD of okra, Xu, Zhang, Feng, et al. (2021) found that FT and FT-US pretreatment freezing, and thawing processes have a positive impact on the retention of flavonoids, which is related to the cell damage caused by ice formation contributing to the release and extraction of flavonoids. Yuan et al. (2022) also reported that US, high static pressure, and high-pressure carbon dioxide treatment effectively retain the flavonoids when VFD jujube slices.

In summary, FT-US has a positive effect on the retention of bioactive substances during the VFD of apricot and is an ideal pretreatment method.

3.8.3. Antioxidant capacity

DPPH, ABTS, and FRAP kits were used to explore the antioxidant capacities of the samples (Table 1). There are no significant differences in the results for DPPH among the four groups ($p > 0.05$). However, the FT-US group shows higher ABTS (1.72 mg Trolox/g) and FRAP (7.12 mg Trolox/g) values than the other groups. Generally speaking, the antioxidant capacities of dried fruits and vegetables result from the joint action of phenols and flavonoids. In this study, the TPC and TFC values for the FT-US group are higher than those for the other groups, thus maintaining a higher antioxidant activity (Wu et al., 2021). In summary, FT-US pretreatment not only improved VFD efficiency, it also showed advantages in the maintenance of antioxidant capacity.

3.8.4. Correlation analysis

In order to further explore the relationship between bioactive substances and antioxidant capacity, we conducted correlation analysis between bioactive substances and antioxidant capacity in the US, FT, and FT-US groups of apricot slices (Supplementary Fig. S1). Pearson correlation analysis showed that the content of AA, TPC, and TFC was positively correlated with the antioxidant capacity of DPPH, ABTS, and FRAP (The coefficients r of AA and DPPH, ABTS, and FRAP were 0.94, 0.81, and 0.91, respectively. The coefficients r of TPC and DPPH, ABTS, and FRAP were 0.65, 0.99, and 0.99, respectively. And the coefficients r of TPC and DPPH, ABTS, and FRAP were 0.52, 1.00, and 0.96, respectively). The correlation between carotenoid content and ABTS and FRAP antioxidant capacity was not significant. The results indicated that AA, TPC, and TFC play important roles in the antioxidant capacity of freeze-dried apricot slices.

3.9. Sensory analysis

To evaluate the popularity of pretreatment vacuum freeze-dried apricot slices, sensory evaluations were conducted on the samples from five aspects (Fig. 6). In terms of appearance and texture parameters, the control group achieved higher scores due to color deviation, indicating that brightness determines the popularity of the appearance of apricot slices. The FT and FT-US groups achieved higher texture scores due to the increased hardness of apricot slices after pretreatment. The ranking order of aroma scores is FT-US > US > FT > Control. The higher aroma score of the pretreatment group compared to the control group is due to the increased variety and quantity of volatile substances caused by pretreatment. Moreover, it is interesting that group members have inconsistent ratings on taste, which may be due to their different preferences for sour and sweet flavors. In summary, the FT-US pretreatment group and the control group have relatively higher overall acceptability of apricot slices.

4. Conclusions

This study explored the effects of US, FT, and FT-US on the VFD characteristics and quality of freeze-dried apricots in order to shorten the VFD time, reduce VFD energy consumption, and improve product quality. The results showed that US, FT, and FT-US change the microstructure and moisture state of apricot slices, which has an effect on their VFD characteristics and product quality, effectively reducing the VFD time and energy consumption. Compared with FT and US pretreatments alone, combined FT-US presents the advantages of both pretreatments, improving the RR, hardness, color, and flavor of freeze-dried apricot slices. In addition, the combined pretreatment has obvious advantages in terms of the retention of bioactive substances (carotenoids, AA, TPC, TFC), antioxidant capacity (DPPH, ABTS, FRAP) and sensory evaluation of the sample. Therefore, FT-US pretreatment saves time and energy during VFD and has the potential to produce high-quality VFD apricot slices.

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fochx.2024.101357.

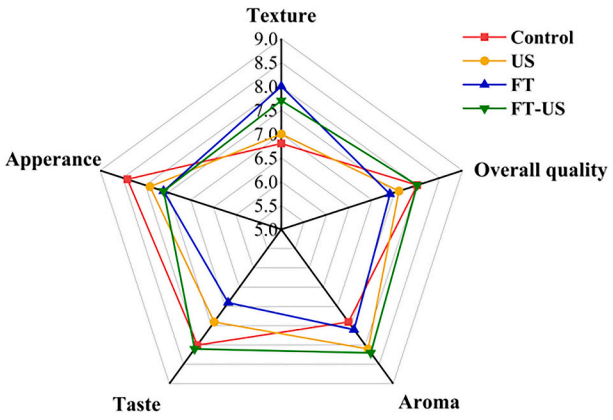

Fig. 6. Sensory evaluation of vacuum freeze-drying apricot slices with different pretreatments.

CRediT authorship contribution statement

Xin Li: Conceptualization, Investigation, Methodology, Writing – original draft. **Yan Zhou:** Investigation, Methodology. **Hao Dong:** Data curation, Methodology. **Tongrui Sun:** Data curation, Formal analysis, Validation. **Yuxing Liu:** Formal analysis, Validation. **Shaobo Cheng:** Conceptualization, Project administration, Supervision, Validation. **Guogang Chen:** Conceptualization, Formal analysis, Funding acquisition, Resources, Supervision, Writing – review & editing.

Declaration of competing interest

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

Data availability

Data will be made available on request.

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