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Effects of variable-temperature drying on the qualities and sweet-substance profile of *Zizyphus jujuba* Mill. cv. Junzao

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ABSTRACT

The changes in the qualities and sweet-substance levels of Junzao jujube during variable-temperature drying (VTD) were investigated. The results showed that VTD retains the original color of jujube, reduces its hardness and chewiness, and decreases its wrinkling while shortening the drying time by 13.2% compared with that of constant temperature drying (CTD). "Electronic-tongue" taste analysis showed that the sweetness of VTD jujube is significantly higher than that for CTD. This is shown to be related to the contents of sucrose, fructose, and glucose, as well as the activities of invertase and sucrose synthase enzymes. In addition, the content trends for sweet amino acids are correlated with the temperature gradient used in VTD. Thus, the present study elucidates the factors governing the transformation of sugar substances in jujube during VTD, as well as providing a practical reference for the application of VTD in the jujube industry.

1. Introduction

The jujube fruit species *Ziziphus jujuba* Mill. cv. Junzao is a traditional medicine and health food in China. As well as being delicious, it is rich in nutrients and functional ingredients. Accordingly, it has become known as the 'the king of fruits' and is widely favored by consumers. However, fresh jujube is prone to post-harvest moisture loss, softening, and rot, which are not conducive to its storage and transportation. Therefore, it is often collected and stored after natural air drying on trees. This kind of natural drying effectively removes most of the moisture from fresh jujube, but ~21% of the water typically remains. This presents the risk of rot and mildew, and it does not meet the moisture-content requirements for storage and sale. Therefore, effective drying methods are usually required to extend the storage period of jujube (Liu et al., 2021).

Drying is an important means of maintaining product quality and extending shelf life. It reduces the weight and volume of a food by removing moisture and thus preventing the growth of microorganisms, effectively prolonging the storage period of a foodstuff and reducing its storage and transportation costs. With the continued development and expansion of the deep-processing industry, jujube-production and -processing technologies have also seen continuous improvement.

At present, the most commonly used jujube-drying technologies are microwave drying, vacuum freeze drying, and hot-air drying. Of these, microwave drying has several drawbacks, such as high initial cost, uneven heating, limited microwave penetration, and mass loss (Chen, Achkar, Liu, & Bennacer, 2021); while, although vacuum-freeze-dried products are of high quality, the process is characterized by high energy consumption, low drying rates, and high cost (Zhang, Yu, Arun, & Zhou, 2022). Accordingly, hot-air drying, which is characterized by its low cost, easy operation, and simple equipment, is the most common drying method. However, improper hot-air-drying conditions, such as excessively high drying temperatures and long drying times, can lead to accumulation of fruit pigments, hardening of the material surface, local overheating, and shrinkage, all of which can lead to a severe degradation of product quality (Kumar, Karim, & Joardder, 2014). Therefore,

Abbreviations: VTD, variable temperature drying; CTD, constant temperature drying; SEM, scanning electron microscope; α-Amy, α-amylase; NI, neutral invertase; AI, acid invertase; SS-s, sucrose synthase- synthesis; SS-c, sucrose synthase- cleavage; FK, fructokinase; HK, hexokinase; SPS, sucrose phosphate synthase; F6P, fructose 6 phosphate; G6P, glucose 6 phosphate; UDPG, uridine 5′-diphosphoglucose; HPLC, High-performance liquid chromatography; SEM, scanning electron microscopy; ANOVA, analysis of variance.

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the jujube industry urgently needs new methods or processes to overcome these problems.

Variable temperature drying (VTD) is a technology by which the dehydration rate of a material is controlled by constantly changing the temperature during hot-air drying according to its characteristics. The application of VTD to beetroot (Kowalski & Szadzinska, 2014), yam (Sahoo, Titikshya, Aradwad, Kumar, & Naik, 2022), lotus root (Zhang, Wang, & Du, 2022; Zhang, Yu, et al., 2022), garlic (Lopez-Ortiz, Rodriguez-Ramirez, Mendez-Lagunas, Martynenko, & Pilatowsky-Figueroa, 2018), and potato (Ho, Chou, Chua, Mujumdar, & Hawlader, 2002), among others, has been investigated, and it has been shown to increase drying efficiency and preserve the original food qualities to a greater extent compared with conventional air drying. However, the effects of VTD on the product quality and storage behavior of jujube are hitherto unreported.

The "electronic tongue" is a means of quantitative and qualitative analysis of the overall properties of a sample that exploits the response sensitivities of different sensors to different substances, analyzing the collected signals through a pattern recognition system. Electronic-tongue technology is widely used in the detection and analysis of food, medicine, and beverages, and it can also be used to evaluate the quality, flavor, taste, and other indicators of products. For example, Veloso, Dias, Rodrigues, Pereira, and Peres (2016) reported that an electronic tongue can be thought of as a taste sensor, and demonstrated that it can distinguish between different sensory intensities of olive oil; while Phat, Moon, and Lee (2016) used high-performance liquid chromatography (HPLC), sensory evaluation, and electronic-tongue technology to analyze the umami flavors of mushroom extract. These studies showed that the results obtained using electronic tongues are close to human sensory scores, indicating that they can be used as a means of objective taste evaluation.

Sweetness is an important sensory attribute in fruit and vegetable products, having an important influence on their quality and taste. Numerous studies have shown that electronic tongues can be used to assess the effects of drying processes on the sweetness of food materials. For instance, Wang et al. (2022) studied the effects of ultrasonic-assisted vacuum freeze-drying on the volatile components and taste components in strawberry slices using an electronic tongue and sensory analysis. Their results showed that the drying treatment increases the fruity and sweet taste of strawberries. Zhang et al. (2024) used metabonomic and sensory histology techniques to analyze the non-volatile substances in Lu'an Guapian tea samples subjected to second and pulley-liquefied-gas drying. Their electronic-tongue results showed that the sweet taste of the tea dried using pulley liquefied gas is more intense. Hong et al. (2021) used different drying methods to treat radishes, and subsequent electronic-tongue analysis showed that freeze-dried radish has higher sourness, umami, and sweetness values. However, most current research on dried jujube focuses on the changes of volatile substances and bioactive substances during drying (Liu et al., 2022; Song et al., 2020), and little is based on electronic-tongue technology.

Accordingly, we preliminarily developed a VTD process that can realize the rapid dehydration of jujube. However, the effects of this technology on the sweet substances and sugar transformation of jujube remained unclear. Therefore, liquid chromatography–mass spectrometry (LCMS) and electronic-tongue technology were used to explore changes in the physical and chemical indexes of jujube during VTD and its effects on the transformation of jujube sugars.

2. Materials and methods

2.1. Raw material

Semi-air-dried jujube (*Ziziphus jujuba* Mill. cv. Junzao; moisture content, 21 ± 1% dry basis) was picked in December 2022 in Hotan, Xinjiang, China. Jujube fruit of uniform size with no mechanical damage and no rot or mildew were selected and immediately sent to the laboratory of the Food College of Shihezi University. Fruit was stored at 0 ± 1 °C prior to analysis.

2.2. Drying conditions and equipment

Samples (about 1500 g) were evenly spread in a drying oven (BGZ-246, Shanghai Boxun Medical Biological Instrument Corp., Shanghai, China) at 50 g/dm^2. The relative humidity of the oven was kept at 60 ± 5% and the air speed was 1 m/s. The samples were weighed every 30 min, and the drying end point was when the moisture content was 15% dry basis.

For constant temperature drying (CTD), the samples were dried at 60 °C for 14.17 h (~850 min). For VTD, the samples were dried at 44 °C for 2.7 h (first stage); at 65 °C for 2.1 h (second stage), and at 49 °C for 7.5 h (third stage).

2.3. Drying curves

Moisture content (*MC*) (dry basis) was calculated using Eq. (1), and the drying rate (*DR*) was calculated using Eq. (2):

$$MC(\%) = \frac{m_t - m}{m} \times 100\% \tag{1}$$

$$DR(\mathrm{g/g \bullet h^{-1}}) = \frac{M_{t_1} - M_{t_2}}{t_2 - t_1} \tag{2}$$

where m and m_t represent the dry base mass at constant weight and the dry base mass at time t (g), respectively; and M_{t1}, M_{t2}, M_t, and M_0 represent dry base moisture content at time t_1, t_2, t, and initial.

2.4. Quality determination

2.4.1. Surface color

The surface color of the jujube was measured at different drying times with a colorimeter (YS3060, Shenzhen Sanensi Technology Co., Ltd., Shenzhen, China). The L^* value represents brightness, the a^* value represents red-green, and the b^* value represents yellow-blue. The total color difference (ΔE) was calculated using Eq. (3), and the hue angle (h) was estimated directly from the colorimeter (Zia & Alibas, 2021).

$$\Delta E = \sqrt{\left(L^* - L^0\right)^2 + \left(a^* - a^0\right)^2 + \left(b^* - b^0\right)^2} \tag{3}$$

where L^*, a^*, and b^* are the brightness, red-green value, and blue-yellow values of jujube after drying; and L^0, a^0, and b^0 are the brightness, red-green, and blue-yellow values of jujube before drying.

2.4.2. Texture characteristics

The texture characteristics of the jujube were evaluated according to Liu et al. (2021). A texture analyzer (Surface Measurement Systems, Ltd., UK) was used to determine the texture characteristics of the samples before and after being subjected to different drying methods. The hardness (g), springiness (mm), gumminess (g), chewability (g), cohesiveness (mm), and recoverability (mm) of the samples were determined.

2.4.3. Microstructure

The microstructures of the jujube fruits were evaluated according to Liu, Zhang, et al. (2021). The internal microstructures of the dried samples were observed using scanning electron microscopy (SEM; S-3400 N, Hitachi Ltd., Japan). Jujube cut into 1 cm × 1 cm × 0.3 cm and fixed in glutaraldehyde at 4 °C for 10 h. Then, the samples were then dehydrated by gradient ethanol solutions (volume fractions: 30%, 50%, 70%, 90%, and 100%) for 15 min. Finally, freeze-dried to obtain SEM samples. Prior to observation, the dehydrated samples were coated with gold–palladium in an ion sputter coater.

2.4.4. Organic acids

The contents of organic acids were determined according to Yang, Kang, Liu, Guo, and Chen (2022). Briefly, 1 g jujube was mixed with 10 mL deionized water, homogenized, ultrasonicated for 30 min, and centrifuged at 10,000 ×*g* for 10 min. The supernatant was collected, the extraction process was repeated, and the two supernatants are combined as the extraction solution. After passing through a 0.45 μm membrane filter, the extract was analyzed by UPLC (Thermo Fisher Scientific Inc., USA). The injection volume was 1 μL; mobile phase A was acetonitrile/water (84:16, *v*/*v*); mobile phase B was 10 mM ammonium formate; the flow rate was 0.8 mL/min; the column temperature was 70 °C; and the injector temperature was 4 °C. The results are expressed as mg kg^{-1} dry basis.

2.4.5. Soluble sugars

The method reported by Pei et al. (2014) was adopted. Briefly, 1 g jujube powder was extracted with 4 mL ethanol at room temperature for 2 h. The residue was extracted again. Next, the solution was centrifuged at 10,000 ×*g* for 20 min. The supernatant was then evaporated to dryness using a rotary evaporator, and the resulting precipitate was taken up in 0.5 mL deionized water. Finally, the solution was centrifuged for 10 min and the supernatant was filtered for HPLC analysis. Separation and measurement of soluble sugars was performed using a Varian ProStar 210 HPLC system (Varian, USA) equipped with a Wyatt Optilab T-rEX differential index detector (Wyatt, USA) and a Shodex SUGAR SP0810 column (8.0 mm × 300 mm, 6 μm, Shodex, Japan). The mobile phase was deionized water and the flow rate was 0.6 mL/min. The results are expressed as g kg^{-1} dry basis.

The contents of fructose 6 phosphate (F6P), glucose 6 phosphate (G6P), and uridine 5′-diphosphoglucose (UDPG) were determined using the corresponding assay kits according to the manufacturer's instructions (Suzhou Grace Biotechnology Co., Ltd., Suzhou, China). All sugar content are presented as mg kg^{-1} dry basis.

2.4.6. Determination of taste using electronic tongue

The samples were analyzed using an electronic tongue (SA402B, Insent, Japan). The sample was diluted at a ratio of 1:10 with water, mixed using a food processor, centrifuged at 8000 ×*g* for 5 min, and the supernatant was filtered. The filtrate (70 mL) was then subjected to electronic tongue testing.

2.4.7. Sweet free amino acid

A 0.5-g jujube sample was accurately weighed and soaked in 20 mL boiling ultra-pure water for 10 min. After cooling to room temperature, the sample was centrifuged at 10,000 ×*g* for 10 min, and the supernatant volume was made up to 50 mL. For HPLC analysis, the mixture was filtered through a 0.22-μm MCE microporous membrane and analyzed using a Waters-AccQ-Tag amino acid column (3.9 mm × 150 mm, 4 μm). The results are expressed as g kg^{-1} dry basis.

2.4.8. Determination of enzymes associated with sugar conversion

The activities of neutral invertase (NI), acid invertase (AI), sucrose synthase–synthesis (SS-s), sucrose synthase–cleavage (SS-c), sucrose phosphate synthase (SPS), α-amylase (α-Amy), fructokinase (FK), and hexokinase (HK) were determined using assay kits according to the manufacturer's instructions (Suzhou Grace Biotechnology Co., Ltd., Suzhou, China). All enzyme activities are presented as U kg^{-1} dry basis.

2.5. Statistical analysis

All experiments were performed three times and the data expressed as mean ± standard deviation. SPSS 22.0 was used for data analysis. Origin software was used for data visualization. The differences between groups were determined by one-way analysis of variance (ANOVA) with Duncan's test at a significance level of $p < 0.05$.

3. Results and discussion

3.1. Drying characteristics

The jujube drying and drying-rate curves for different drying methods are shown in Fig. 1. CTD and VTD achieve their drying end points after 14.17 and 12.30 h, respectively (Fig. 1A). Thus, the drying time for VTD is shortened by 13.2% compared with that for CTD, which is in concert with Kowalski and Szadzinska (2014) results on sugar beet drying. VTD may effectively shortens the drying time by reducing the collapse of internal moisture exchange channels. It can be seen from the drying-rate curve (Fig. 1B) that the dehydration of jujube occurs mainly in the deceleration period (Liu, Zhang, et al., 2021). With the extension of drying time, the drying rate decreases due to most surface water being lost and it being more difficult to remove internal water. In the later drying period, the drying rate for VTD is higher, which may be caused by the increased partial pressure of water vapor in the air and the difference between the partial pressure of water vapor on the surface and that inside the material.

3.2. Surface color

Color is an important parameter used to evaluate the quality of food, and it has an important effect on consumer choice. The color change observed upon drying products is affected by enzymatic browning, non-enzymatic browning, pigment degradation, and ascorbic acid oxidation (Sahoo et al., 2022). Fig. 2 shows the changes in color components of a jujube sample during drying. Sharp decreases in L^* (Fig. 2A), a^* (Fig. 2B), and b^* (Fig. 2C) values are observed during the early and middle drying periods. The decreases in a^* and b^* values means that the red coloration fades along with an increase in green and yellow coloration, resulting in a yellowish appearance developing on the jujube surface during the drying process. This is closely related to the thermal oxidation and degradation of chlorophyll, carotenoids, anthocyanins, and some phenolic substances caused by high temperature (Shi, Zhang, Su, Zhou, & Li, 2018). Of all the color parameters, the a^* value shows the most significant change. Compared with that of undried jujube, the a^* values for CTD and VTD decrease by 19.96% and 13.85%, respectively. The smaller decrease in the a^* value for VTD indicates that the short and high temperature VTD process effectively reduces the degradation of pigments and the oxidation of phenols. In addition, the decrease in L^* value is mainly caused by moisture loss from the sample skin. Liu, Zhang, et al. (2021) obtained similar results when exploring the effects of different temperatures on the drying kinetics, color, and texture of jujube. In the present study, the color properties of the VTD sample fluctuate significantly during the middle drying period, tending to stabilize during the later drying period. The higher temperature conditions accelerate the decomposition of pigments during the middle drying period.

The color difference value ΔE is used to quantify the overall color change of a dried product, and the lower the value of ΔE, the better the product quality (Liu, Zhang, et al., 2021). Here, ΔE increases with drying time (Fig. 2D). At the end of drying, the ΔE values for CTD and VTD jujube are 4.94 and 3.89, respectively. Thus, ΔE for VTD is 21.26% lower than that for CTD, which is related to the effective shortening of exposure to high temperature the shorter overall drying time compared with that for CTD. When jujube are heated at high temperatures for long periods of time, enzymatic and non-enzymatic browning reactions are intensified, darkening the surface of the fruit and destroying its natural color (Karabacak, 2019), thus accelerating changes in ΔE.

Hue angle is a parameter used to describe color in color space. It is a measure of the position of hue on the color wheel. When the hue angle is 0°, the color is close to red, 90° is yellow, 180° is green, and 270° is blue. The hue angle of undried jujube is 26.95°, i.e., showing red color. The hue angles of jujube following CTD and VTD increase slightly (Fig. 2E) upon drying, indicating that the surface color of the sample gradually

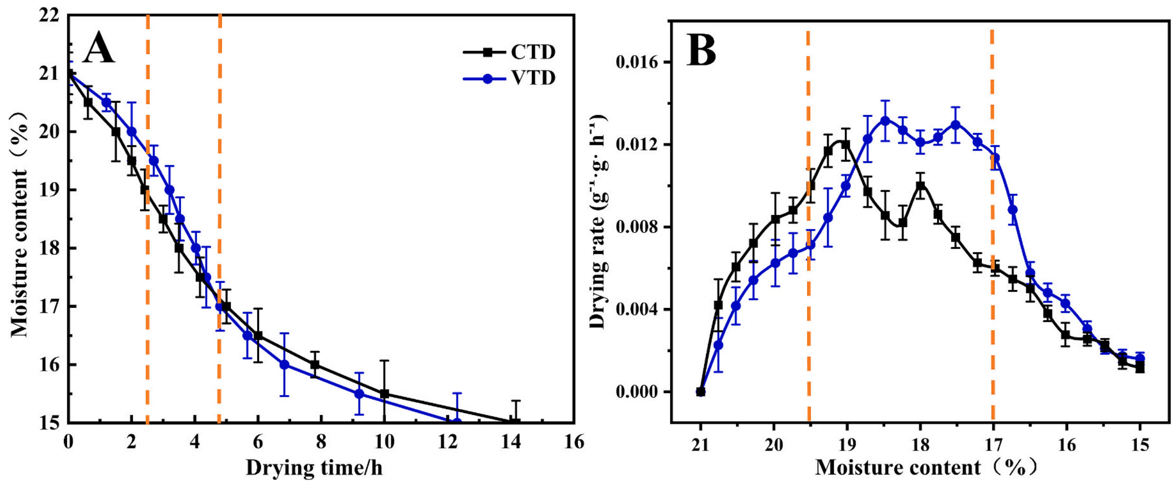

Fig. 1. Drying curve and drying rate curve of jujube with variable temperature drying (VTD)and constant temperature drying (CTD).

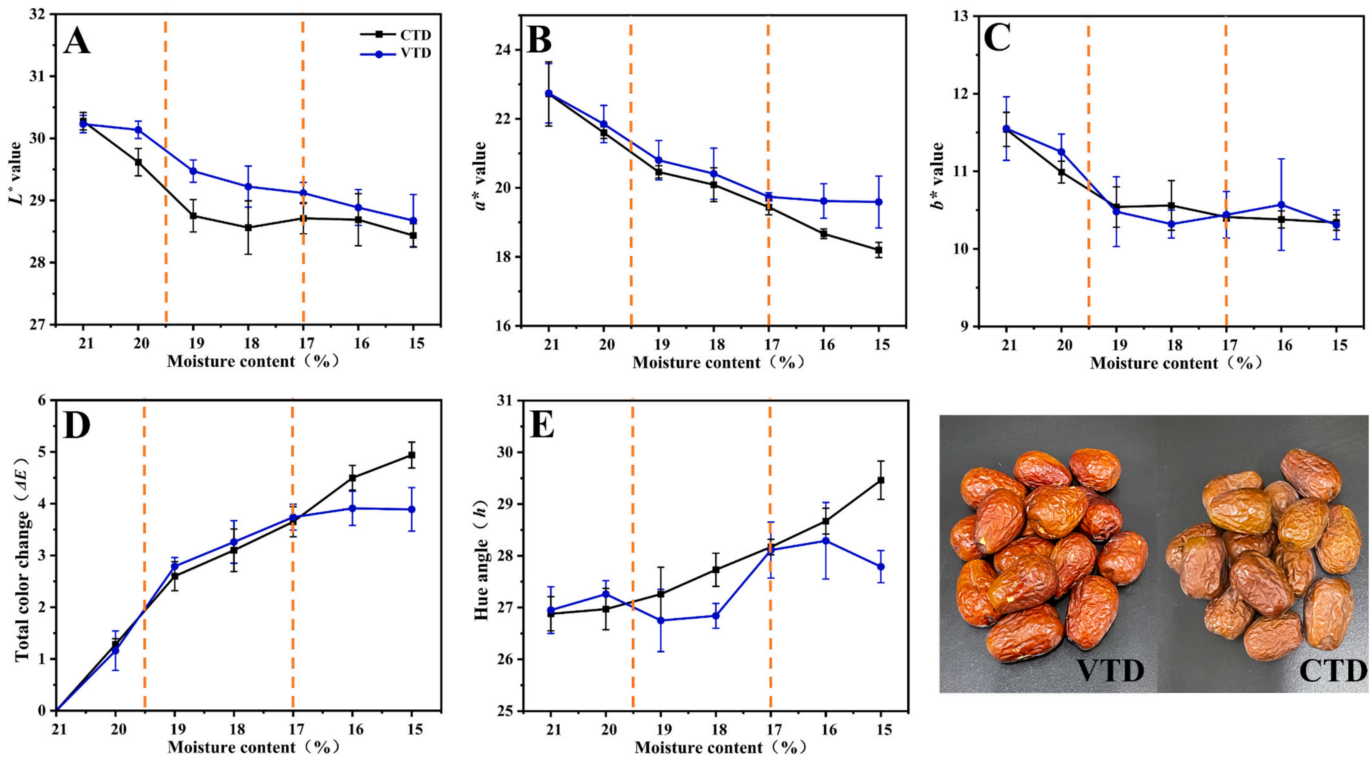

Fig. 2. L^*value (A), a^* value (B), b^* value (C), ΔE (D) and hue angle (E) of jujube dried by variable temperature drying (VTD)and constant temperature drying (CTD).

turns yellow. In summary, VTD better retains the original color of jujube.

3.3. Texture characteristics

Texture profile analysis is a method to evaluate the sensory quality of food by mechanical compression of samples and simulated chewing (Liu, Zhang, et al., 2021). Wojdylo et al. (2016) used it to study the effects of different drying methods on the contents of bioactive substances and the sensory qualities of jujube and found that dried jujube with a soft tissue structure and low chewability are most popular among consumers. In the present study, the dried samples all show a decrease in hardness, chewiness, and gumminess ($p < 0.05$) (Fig. 3), but no significant changes in cohesiveness, recoverability, and springiness ($p > 0.05$). At the end of drying, the hardness, chewiness, and gumminess of the CTD and VTD jujubes are decreased by 16.81% and 27.56%, 16.47% and 33.92%, and 26.16% and 36.06%, respectively, compared with the original sample (moisture content 21% dry basis). As the temperature increases, the water evaporates rapidly and the loss of moisture leads to large holes between the cells in the early drying period, thus rapidly decreasing the fruit's hardness and chewiness. As the external moisture of the jujube is gradually lost, the surface forms a hard shell (Kamal, Song, Tan, Zhu, & Tan, 2019), so that the hardness and chewiness gradually increase in the middle and later drying periods. The temperature control of VTD effectively delays the further formation of the surface crust and product shrinkage, resulting in a product with lower hardness and chewiness.

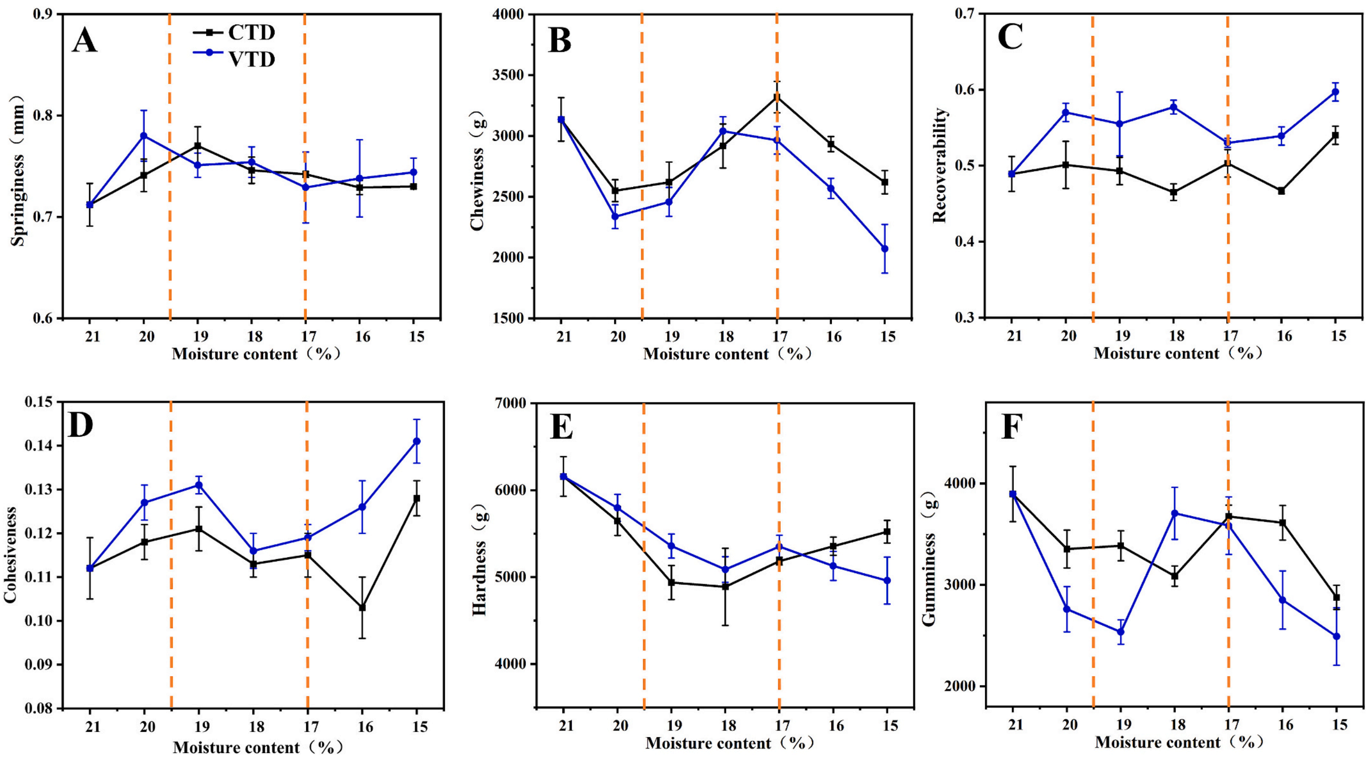

Fig. 3. Springiness (A), chewiness (B), recoverability (C), cohesiveness (D), hardness (E) and gumminess (F) of jujube dried by variable temperature drying (VTD)and constant temperature drying (CTD).

3.4. Microstructure

In order to explain the changes in hardness and chewability mentioned above, SEM was used to observe the internal microstructures of the samples after different drying periods (Fig. 4). The undried jujube (Fig. 4A) shows uniform honeycomb shapes, a uniform cell size, clear cell outlines, and no obvious cell deformation. Pores are observed inside the samples, with those of the CTD jujube being larger and more densely packed, while those of the VTD jujube being smaller in the early drying period (Fig. 4B,E). The lower temperature in the early drying period of VTD slows down the degradation of cell walls and prolongates the period of shrinkage and collapse of tissue structure caused by moisture gradient and enzyme action (Yao et al., 2022).

Fig. 4C,F shows the microscopic characteristics of the sample in the middle drying period. The pores formed in the early drying period gradually became larger and more compact in the middle drying period, and the cell structure shows obvious shrinkage and deposition. At this time, the cell wall support function of the jujube is damaged by long-term high temperature drying, and the moisture-exchange channels contract, which explains the increasing hardness observed in the middle drying period.

Fig. 4D,G shows the microstructure of the jujube in the later drying period. The pores are larger and more uniform in the VTD jujube, while the internal gaps, adhesions, and reconstructions of CTD jujube appear larger compared with those in the middle drying period, because the lower temperature reduces the temperature difference between the inside and outside of the jujube in the later period of VTD, at which point the internal tissue reaches a phase-transition temperature, generating water vapor and thus an increase in the internal pressure. While liquid water at the surface prevents water vapor from escaping directly to the external environment, this pressure gradient provides a driving force that causes the water to migrate outward (Chen et al., 2021). However, as CTD jujube is in a high temperature state for a long time, its surface forms a hard shell due to rapid water loss, and the water vapor and energy in the internal tissue accumulate excessively, resulting in larger holes and extensive interior collapse. This may also be why the VTD drying rate is faster in the later drying period.

3.5. Organic acid

Organic acid is an important indicator of the ripeness, portability, and storage stability of horticultural products. As shown in Table S1, 26 different organic acids were detected in jujube, among which lactic acid, malic acid, oxalic acid, and citric acid account for 86.37% of the total organic acids. The content of total organic acids in CTD and VTD jujube are increased significantly upon drying. The organic acid content of CTD jujube is slightly higher than that of VTD jujube, which is due to the fact that the fruit is exposed to high temperature for a long time in the drying process, generating organic acids via the Maillard reaction, while the basic groups in the amino acids are consumed, further increasing the titratable acid content (Liu, Cao, Wang, & Liao, 2010).

3.6. Taste analysis

Electronic tongues have been successfully applied to the qualitative and quantitative study of food taste characteristics (Zhang, Wang, & Du, 2022). In the present study, the taste of jujube was analyzed using an electronic tongue, and the results are shown in Fig. S1. All the tastes except astringent aftertaste, bitterness, and bitterness aftertaste, are significantly different between the samples ($p < 0.05$). The sweetness, acidity, and saltiness of dried jujube are significantly increased compared with those of the undried fruit, suggesting that proper drying increases the sweetness of jujube. This is consistent with previous findings by Wang et al. (2022), who reported that the sweetness of strawberry slices increases upon vacuum freeze-drying. Furthermore, Oikonomopoulou, Krokida, and Karathanos (2013) reported that the perception of saltiness and sweetness can be enhanced by adjusting the microstructure of a food. A higher the porosity increases the specific surface area of a food and therefore its contact with the taste receptors, thus increasing the perception of sweetness and saltiness.

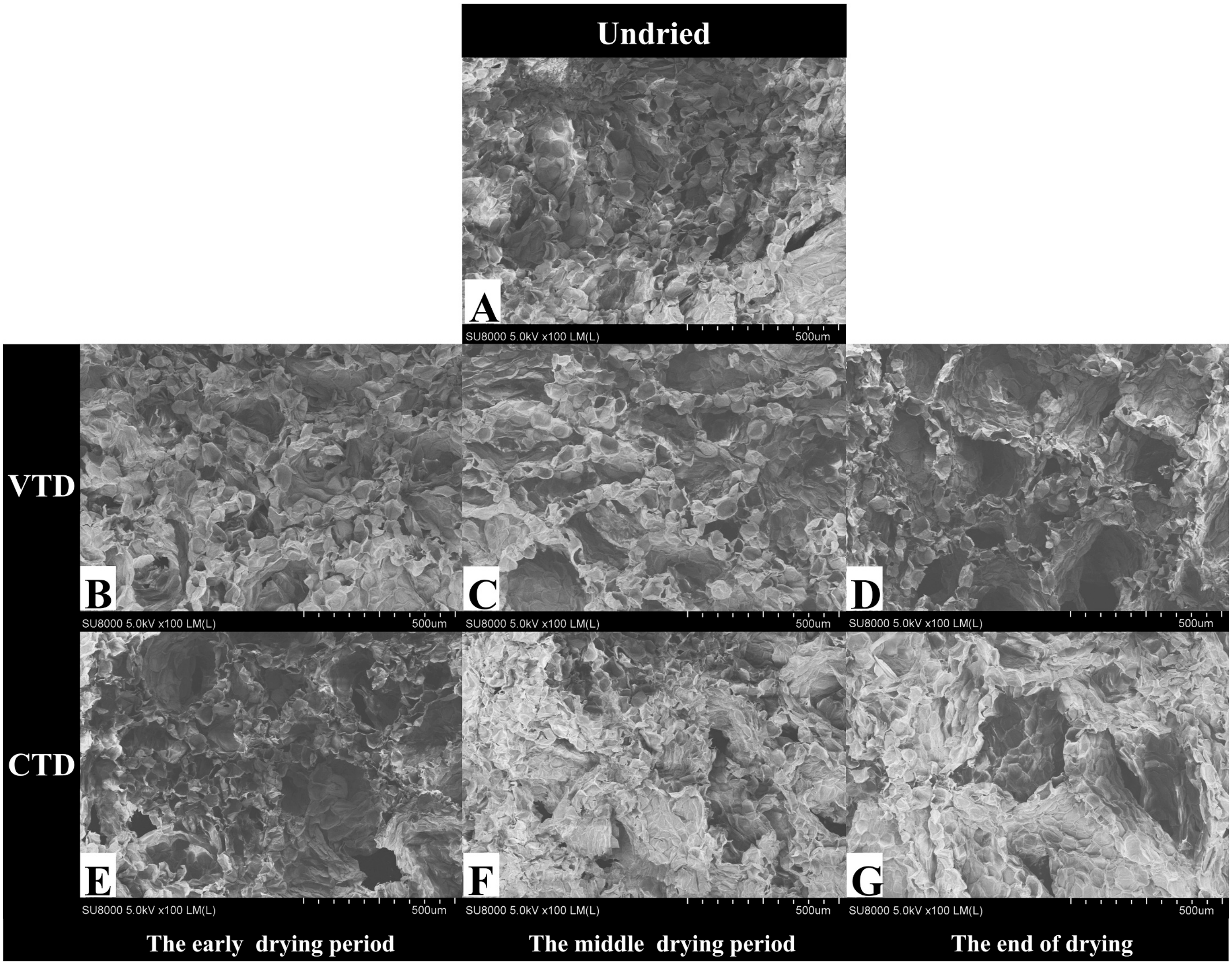

Fig. 4. SEM images of the initial sample (A) before drying and the samples of variable temperature drying (VTD)and constant temperature drying (CTD). Jujube in the early stage of VTD (B); jujube in the middle stage of VTD (C); jujube at the end of VTD (D); jujube in the early stage of CTD (E); jujube in the middle stage of CTD (F); jujube at the end of CTD (G); SEM: scanning electron microscope.

In the present study, VTD jujube presents higher sweet and umami tastes and lower sour, bitter, and astringent tastes compared with CTD jujube. This increase in umami flavor may be related to the contents of umami amino acids in jujube. The short, high-temperature drying process of VTD lessens the degradation of amino acids, thus maintaining the umami flavor of jujube. In terms of bitterness, Pu et al. (2018) reported that the Maillard reaction is the main source of the bitterness of dried jujube and that the degree of bitterness is related to its shrinkage. The temperature strategy of VTD and the resulting uniform internal structure (Fig. 4) limit the formation of bitter compounds in jujube.

3.7. Soluble sugars and related enzymes

The composition and contents of sugars in jujube significantly affect its sweetness and flavor, and an appropriate carbohydrate composition and content can also increase the sensory interactions responsible for sweetness and aroma perception (Liu, Zhang, et al., 2021; Song et al., 2019). Sugars account for ~80% of the dry weight of jujube, among which, sucrose, fructose, and glucose are the main sugars (Li, Fan, Ding, & Ding, 2007). In order to determine why jujube is sweeter after VTD, the changes in its sugar profile and related enzyme activities during CTD and VTD were investigated.

The changes in the main carbohydrate substances and activities of enzymes related to sugar conversion in jujube upon drying are shown in Fig. 5. The sucrose level in jujube decreases slowly in the early and later stages of VTD and rapidly in the middle high-temperature stage (Fig. 5A), while sucrose shows a relatively stable downward trend during CTD (Fig. 5B). During the VTD process, fructose and glucose show the same trend but with the glucose level being slightly lower (between 117.338 and 206.284 g kg^{-1}) than that of fructose (136.944 to 248.722 g kg^{-1}). As the moisture content of jujube decreases, fructose and glucose show an initial slow increase and then a more rapid increase. During CTD, fructose and glucose show the same upward trend as they do in VTD, but the glucose content is significantly higher than that of fructose in the middle and later stages of CTD. However, the changes in maltose content during the whole drying process are not significant ($p <$ 0.05). Furthermore, for VTD, the starch content decreases slowly in the early and middle stages and then more rapidly in the later stage (Fig. 5C), but it shows a uniform downward trend in CTD (Fig. 5D). We also found that the changes in sucrose, fructose, and glucose are not significant in the later stage of CTD, and the fructose content of VTD jujube is significantly higher than that of CTD jujube at the end of drying, giving the jujube a sweeter taste. This result is consistent with the sweetness measurement obtained using the electronic tongue

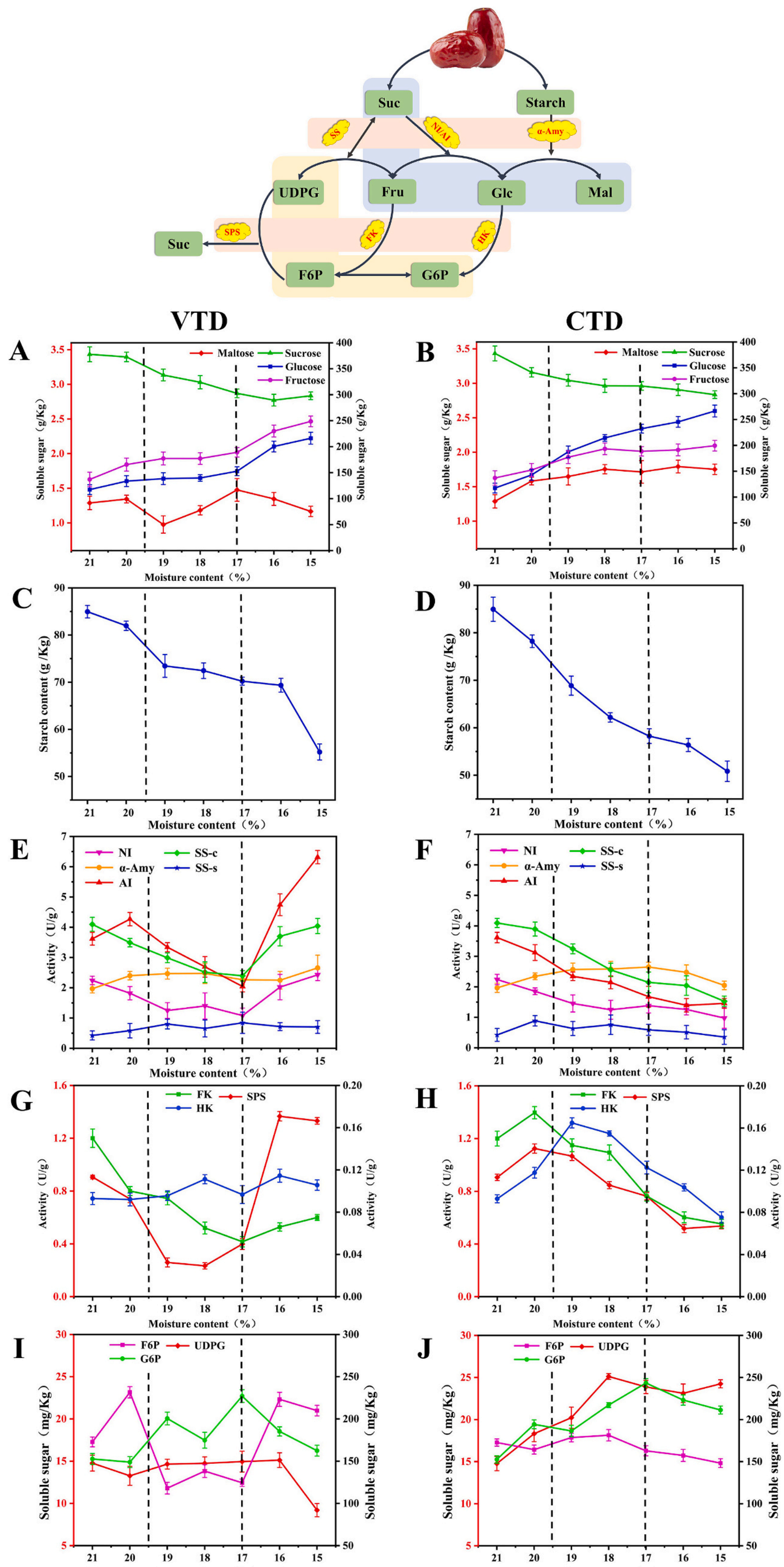

Fig. 5. Effects of variable temperature drying (VTD)and constant temperature drying (CTD) on the soluble sugar (A, B, I, J), starch (C, D) and related enzymes (E, F, G, H) of jujube samples. α-Amy, α-amylase; NI, neutral invertase; AI, acid invertase; SS-s, sucrose synthase- synthesis; SS-c, sucrose synthase- cleavage; FK, fructokinase; HK, hexokinase; SPS, sucrose phosphate synthase; F6P, fructose 6 phosphate; G6P, glucose 6 phosphate; UDPG, uridine 5′-diphosphoglucose.

(Fig. 5).

Sucrose can be converted to fructose and glucose by NI and AI. It can also be reversibly converted to fructose and UDPG by SS-s and SS-c. Fructose and glucose are further phosphorylated to F6P and G6P by FK and HK. F6P and UDPG can be synthesized into sucrose by SPS (Zhang, Bian, Hou, & Li, 2018). Starch is converted into glucose and maltose under the action of α-Amy. These enzymes play important roles in the conversion of sucrose and hexose. As can be seen from Fig. 5E,G, the activities of NI, AI, SPS, and SS-c as a whole show a trend of decreasing first and then increasing, maintaining a low level in the middle high-temperature drying stage in the VTD process (NI: 1.079–2.426 U/g, AI: 2.043–6.317 U/g, SPS: 0.233–1.367 U/g, SS-c: 2.394–4.095 U/g). There are no significant changes in α-Amy, SS-s, HK, and FK activities ($p < 0.05$). In the CTD process, except for that of α-Amy, the activities of the enzymes increase slightly in the initial drying stage, then decrease and eventually stabilize (Fig. 5F,H). VTD improves the activity of some enzymes compared with CTD, among which, the activities of NI, AI, and SS-c are higher, reaching 2.426, 6.371, and 1.332 U/g, respectively, at the end of drying.

The increased temperature stimulates the activities of related enzymes and promotes the conversion of sucrose to fructose and glucose in the early stage of VTD. Fig. 5 shows that sucrose is converted into UDPG and fructose, fructose, and glucose, respectively, by SS-c, NI, and AI. The lower UDPG content and higher NI and AI activities indicate that NI and AI play greater roles in the conversion of sucrose to fructose and glucose. During the VTD process, the activities of all the enzymes decrease, except or those of SS-c and α-Amy in the middle stage. The sucrose content decreases rapidly during this period, primarily due to the elevated temperature facilitating the thermal degradation of sucrose, which is also affected by SS-c. In addition, starch is also converted to glucose under the action of α-Amy. These effects increase the glucose and fructose contents, with the glucose content increasing significantly. However, no significant increase in glucose is observed during this period (Fig. 5A) owing to its consumption as a substrate in the Maillard reaction.

According to isotope labeling experiments by Locas and Yaylayan (2008), 5-hydroxymethylfural is partially produced by glucose when sucrose is referred to in acidic methanol at 65 °C. This means that at 65 °C, the carbonyl substrate of the Maillard reaction is predominantly glucose, which is why the glucose content is lower than that of fructose. In this process, the synthesis and consumption of fructose and glucose reach a dynamic balance. As a result, fructose and glucose levels are relatively stable in this stage. In the later stage of VTD, NI, AI, and SS-c activities are enhanced due to the higher temperature in the previous stage causing the cell structure to be destroyed, resulting in the leakage of cell contents and the release of more enzyme substances, while the lower temperature increases the activity of these enzymes in the later stage, accelerating the conversion of sucrose to fructose and glucose. As a result, sucrose levels are lower, and glucose and fructose levels are higher in this stage. The temperature is kept high and constant during the CTD process, stimulating the activities of relevant enzymes and thus the conversion between sugars, leading to significant changes in the early drying stage. While the sugar material gradually tends to stabilize as the drying time increases, as the activities of enzymes involved in sugar conversion are largely suppressed when jujube are exposed to high temperatures for long periods of time. Furthermore, the contact area between enzymes and sugars is reduced due to severe shrinkage and collapse in the interior of the jujube at high temperature (Fig. 4).

In the whole drying process, we also found that F6P and G6P show a trade-off relationship (Fig. 5I,J) that is more apparent in the VTD process. The content of G6P is higher in the middle stage of VTD, while the content of F6P is higher in the early and later stages of VTD (Fig. 5I). This may be because high temperatures promote the conversion of F6P to G6P, or that HK activity is higher during the middle stage, which promotes the conversion of glucose to G6P. In addition, the low content of UDPG, low activity of SPS, and relatively stable F6P content indicate that the contribution of SPS to sucrose synthesis is not significant.

Combining the above results, it appears that NI and AI play significant roles in promoting the conversion of sucrose to fructose and glucose, and that the effects of SPS and SS-s on sucrose synthesis are not significant. In addition, changes in temperature alter the internal spatial structure of jujube, increasing the area of contact between the enzyme and the sugar and facilitating interconversion between different sugar components. At the same time, temperature changes also affect the activities of enzymes involved in sugar conversion, and dynamic changes in these activities directly or indirectly affect the sugar content and composition of jujube. The temperature strategy of VTD changes the activity of enzymes involved in sugar conversion and promotes the mutual conversion of sugars more than CTD, giving the jujube a sweeter taste.

3.8. Sweet amino acids

Amino acids can be categorized according to flavor characteristics into MSG amino acids, sweet amino acids, bitter amino acids, and tasteless amino acids (Hu, Feng, Huang, Ibrahim, & Liu, 2020). The sweet amino acids include alanine (Fig. 6A), glycine (Fig. 6B), proline (Fig. 6C), serine (Fig. 6D), and threonine (Fig. 6E). Fig. 6 shows the changes of sweet amino acid levels in jujube during drying. Among these five amino acids, the content of proline is the highest, and the contents of glycine and alanine are low. The contents of the five sweet amino acids decreases slightly upon drying, which is consistent with Liu et al. (2022). The changes in serine, proline, and threonine levels show a similar trend, increasing first and then decreasing, reaching their maximum values at 19% moisture content (2.989, 12.644, and 0.46 g kg^{-1}, respectively) during the VTD process. The higher temperature during the middle stage stimulates protein degradation, resulting in the production of a large number of free amino acids. In addition, high temperature can cause amino acid pyrolysis, dehydrogenation, decarboxylation, amino transfer, and the Maillard reaction (Liu et al., 2022), which may account for the slight decrease in amino acid content in the later stage.

For the CTD process, the sweet amino acids all show a trend of increasing first and then decreasing, remaining stable at the later drying period. The higher temperature stimulated protein degradation and produced a large number of free amino acids at the beginning of drying. With the increase of drying time, the Maillard reaction intensified and the content of free amino acids decreased, and the increase and consumption of free amino acids reached a balance at the later drying period.

4. Conclusions

The effects of CTD and VTD on the color, texture, microstructure, and organic acids of jujube were studied, and the changes in its sweet-substance profile during the drying process were thoroughly explored. The results showed that VTD retains the original color of jujube more than CTD, reduces its hardness and chewiness, reduces its shrinkage, and shortens the drying time by 13.2%. The results of electronic-tongue analysis showed that VTD jujube has a higher sweet and umami tastes, and lower sour, bitter, and astringent tastes. This change in sweetness is related to the decrease of sucrose, the increase of fructose and glucose, and the accumulation of soluble total sugars in the VTD process. NI, AI, and SS-c play important roles in this process. A certain drying temperature enhances the activities of NI, AI, and SS-c, and promotes the accumulation of soluble total sugar. In addition, the fluctuation trend of sweet amino acids also corresponds to the VTD temperature. Overall, this study provides a scientific basis and practical guidelines for the popularization and application of VTD for jujube drying.

This paper only studies from the perspective of sugar conversion, but organic acids are also important factors affecting the taste of fruits. It was also found that the organic acid content of jujube changed

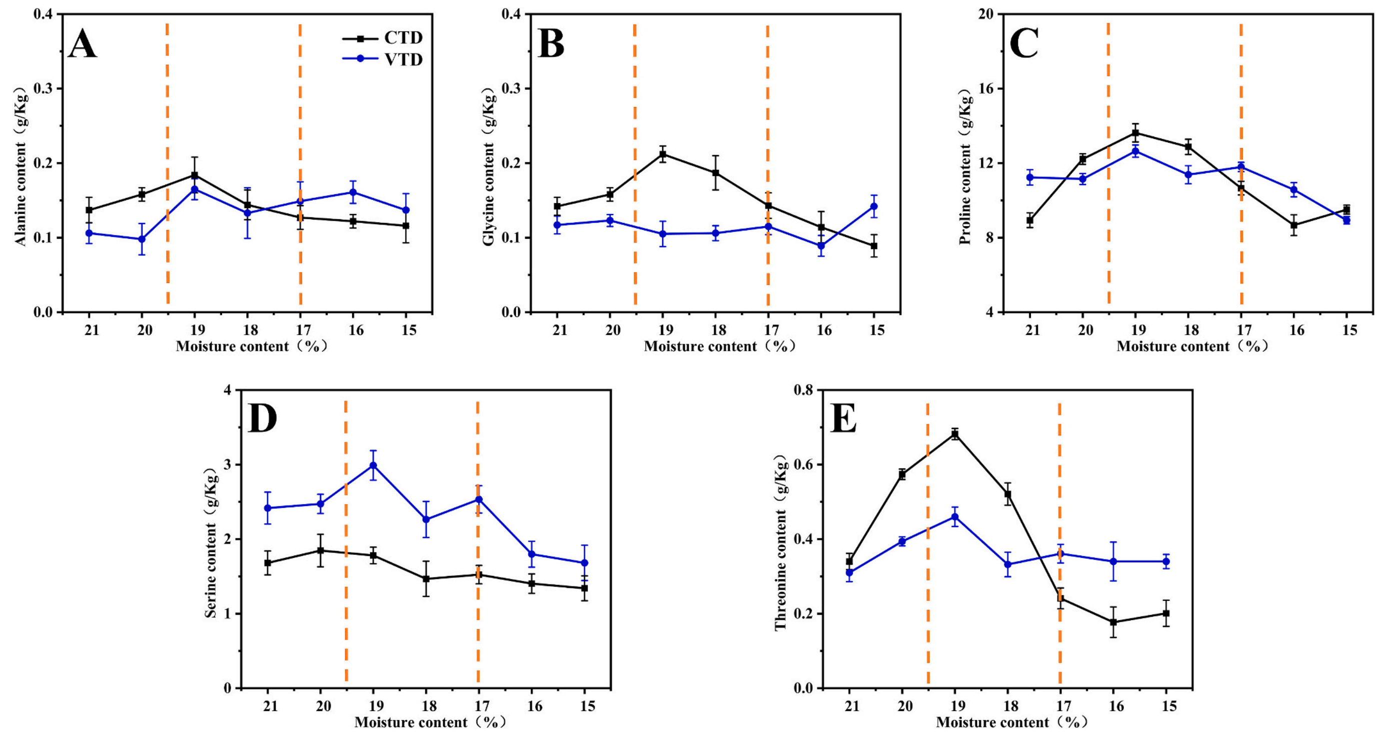

Fig. 6. Effects of variable temperature drying (VTD)and constant temperature drying (CTD) the alanine (A), glycine (B), proline (C), serine (D) and threonine (E) of jujube samples.

significantly during the drying process. Therefore, the next experiment can study and discuss the organic acid content and related enzymes of organic acid metabolism in the drying process of jujube, and further provide a more comprehensive theoretical basis for improving the quality of dried jujube.

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CRediT authorship contribution statement

Yaxuan Liao: Writing – original draft, Methodology, Investigation, Conceptualization. **Yuxing Liu:** Methodology, Investigation. **Weida Zhang:** Software, Data curation. **Hao Dong:** Formal analysis, Data curation. **Liqing Yang:** Validation, Formal analysis. **Jiajun Zhang:** Validation, Formal analysis. **Yunuo Wang:** Validation, Formal analysis. **Shaobo Cheng:** Project administration, Conceptualization, 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

The authors do not have permission to share data.

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References

Chen, A., Achkar, G. E. L., Liu, B., & Bennacer, R. (2021). Experimental study on moisture kinetics and microstructure evolution in apples during high power microwave drying process. *Journal of Food Engineering, 292*. https://doi.org/10.1016/j.jfoodeng.2020.110362

Ho, J. C., Chou, S. K., Chua, K. J., Mujumdar, A. S., & Hawlader, M. N. A. (2002). Analytical study of cyclic temperature drying: Effect on drying kinetics and product quality. *Journal of Food Engineering, 51*(1), 65–75. https://doi.org/10.1016/s0260-8774(01)00038-3

Hong, S. J., Boo, C. G., Lee, J., Hur, S. W., Jo, S. M., Jeong, H., … Shin, E.-C. (2021). Chemosensory approach supported-analysis of wintering radishes produced in Jeju island by different processing methods. *Food Science and Biotechnology, 30*(8), 1033–1049. https://doi.org/10.1007/s10068-021-00948-2

Hu, S., Feng, X., Huang, W., Ibrahim, S. A., & Liu, Y. (2020). Effects of drying methods on non-volatile taste components of Stropharia rugoso-annulata mushrooms. *Lwt-Food Science and Technology, 127*, 7. https://doi.org/10.1016/j.lwt.2020.109428

Kamal, T., Song, Y. K., Tan, Z., Zhu, B. W., & Tan, M. (2019). Effect of hot-air oven dehydration process on water dynamics and microstructure of apple (Fuji) cultivar slices assessed by LF-NMR and MRI. *Drying Technology, 37*(15), 1974–1987. https://doi.org/10.1080/07373937.2018.1547312

Karabacak, A. O. (2019). Effects of different drying methods on drying characteristics, colour and in-vitro bioaccessibility of phenolics and antioxidant capacity of blackthorn pestil (leather). *Heat and Mass Transfer, 55*(10), 2739–2750. https://doi.org/10.1007/s00231-019-02611-3

Kowalski, S. J., & Szadzinska, J. (2014). Kinetics and quality aspects of beetroots dried in non-stationary conditions. *Drying Technology, 32*(11), 1310–1318. https://doi.org/10.1080/07373937.2014.915555

Kumar, C., Karim, M. A., & Joardder, M. U. H. (2014). Intermittent drying of food products: A critical review. *Journal of Food Engineering, 121*, 48–57. https://doi.org/10.1016/j.jfoodeng.2013.08.014

Li, J. W., Fan, L. P., Ding, S. D., & Ding, X. L. (2007). Nutritional composition of five cultivars of chinese jujube. *Food Chemistry, 103*(2), 454–460. https://doi.org/10.1016/j.foodchem.2006.08.016

Liu, F. X., Cao, X. M., Wang, H. Y., & Liao, X. J. (2010). Changes of tomato powder qualities during storage. *Powder Technology, 204*(1), 159–166. https://doi.org/10.1016/j.powtec.2010.08.002

Liu, Y. X., Liao, Y. X., Guo, M. R., Zhang, W. D., Sang, Y. Y., Wang, H., … Chen, G. G. (2022). Comparative elucidation of bioactive and volatile components in dry mature jujube fruit (*Ziziphus jujuba* mill.) subjected to different drying methods. *Food Chemistry-X, 14*, 10. https://doi.org/10.1016/j.fochx.2022.100311

Liu, Y. X., Sang, Y. Y., Guo, J. Y., Zhang, W. D., Zhang, T. Y., Wang, H., … Chen, G. G. (2021). Analysis of volatility characteristics of five jujube varieties in Xinjiang Province, China, by HS-SPME-GC/MS and E-nose. *Food Science & Nutrition, 9*(12), 6617–6626. https://doi.org/10.1002/fsn3.2607

Liu, Y. X., Zhang, W. D., Yang, W. T., Sang, Y. Y., Guo, M. R., Liu, D., … Chen, G. G. (2021). The effects of drying temperature on the kinetics, color, structure, and pectin composition of *Zizyphus jujuba* mill. cv. Junzao. *Journal of Food Processing and Preservation, 45*(11), 12. https://doi.org/10.1111/jfpp.15942

Locas, C. P., & Yaylayan, V. A. (2008). Isotope labeling studies on the formation of 5-(hydroxymethyl)-2-furaldehyde (HMF) from sucrose by pyrolysis-GC/MS. *Journal of Agricultural and Food Chemistry, 56*(15), 6717–6723. https://doi.org/10.1021/jf8010245

Lopez-Ortiz, A., Rodriguez-Ramirez, J., Mendez-Lagunas, L. L., Martynenko, A., & Pilatowsky-Figueroa, I. (2018). Non-isothermal drying of garlic slices (*Allium sativum*, L.): Wave period and initial temperature of the heating/cooling effect. *Food and Bioproducts Processing, 111*, 83–92. https://doi.org/10.1016/j.fbp.2018.06.005

Oikonomopoulou, V., Krokida, M., & Karathanos, V. (2013). Influence of structure on saltiness and sweetness of dehydrated food products. *Drying Technology, 31*(7), 837–847. https://doi.org/10.1080/07373937.2013.768265

Pei, F., Shi, Y., Gao, X. Y., Wu, F. N., Mariga, A. M., Yang, W. J., … Hu, Q. H. (2014). Changes in non-volatile taste components of button mushroom (*Agaricus bisporus*) during different stages of freeze drying and freeze drying combined with microwave vacuum drying. *Food Chemistry, 165*, 547–554. https://doi.org/10.1016/j.foodchem.2014.05.130

Phat, C., Moon, B., & Lee, C. (2016). Evaluation of umami taste in mushroom extracts by chemical analysis, sensory evaluation, and an electronic tongue system. *Food Chemistry, 192*, 1068–1077. https://doi.org/10.1016/j.foodchem.2015.07.113

Pu, Y. F., Ding, T., Wang, W. J., Xiang, Y. J., Ye, X. Q., Li, M., & Liu, D. H. (2018). Effect of harvest, drying and storage on the bitterness, moisture, sugars, free amino acids and phenolic compounds of jujube fruit (*Zizyphus jujuba*.cv. Junzao). *Journal of the Science of Food and Agriculture, 98*(2), 628–634. https://doi.org/10.1002/jsfa.8507

Sahoo, M., Titikshya, S., Aradwad, P., Kumar, V., & Naik, S. N. (2022). Study of the drying behaviour and color kinetics of convective drying of yam (*Dioscorea hispida*) slices. *Industrial Crops and Products, 176*, 13. https://doi.org/10.1016/j.indcrop.2021.114258

Shi, Q. Q., Zhang, Z., Su, J. J., Zhou, J., & Li, X. G. (2018). Comparative analysis of pigments, Phenolics, and antioxidant activity of Chinese jujube (*Ziziphus jujuba* mill.) during fruit development. *Molecules, 23*(8), 14. https://doi.org/10.3390/molecules23081917

Song, J. X., Bi, J. F., Chen, Q. Q., Wu, X. Y., Lyu, Y., & Meng, X. J. (2019). Assessment of sugar content, fatty acids, free amino acids, and volatile profiles in jujube fruits at different ripening stages. *Food Chemistry, 270*, 344–352. https://doi.org/10.1016/j.foodchem.2018.07.102

Song, J. X., Chen, Q. Q., Bi, J. F., Meng, X. J., Wu, X. Y., Qiao, Y. N., & Lyu, Y. (2020). GC/MS coupled with MOS e-nose and flash GC e-nose for volatile characterization of Chinese jujubes as affected by different drying methods. *Food Chemistry, 331*, 8. https://doi.org/10.1016/j.foodchem.2020.127201

Veloso, A. C. A., Dias, L. G., Rodrigues, N., Pereira, J. A., & Peres, A. M. (2016). Sensory intensity assessment of olive oils using an electronic tongue. *Talanta, 146*, 585–593. https://doi.org/10.1016/j.talanta.2015.08.071

Wang, C., Zhang, L., Qiao, Y., Liao, L., Shi, D., Wang, J., & Shi, L. (2022). Effects of ultrasound and ultra-high pressure pretreatments on volatile and taste compounds of vacuum-freeze dried strawberry slice. *Lwt, 160*. https://doi.org/10.1016/j.lwt.2022.113232

Wojdylo, A., Figiel, A., Legua, P., Lech, K., Carbonell-Barrachina, A. A., & Hernandez, F. (2016). Chemical composition, antioxidant capacity, and sensory quality of dried jujube fruits as affected by cultivar and drying method. *Food Chemistry, 207*, 170–179. https://doi.org/10.1016/j.foodchem.2016.03.099

Yang, W. T., Kang, J. W., Liu, Y. X., Guo, M. R., & Chen, G. G. (2022). Effect of salicylic acid treatment on antioxidant capacity and endogenous hormones in winter jujube during shelf life. *Food Chemistry, 397*, 11. https://doi.org/10.1016/j.foodchem.2022.133788

Yao, X. D., Zang, Y. Z., Gu, J. W., Ding, H. Y., Niu, Y. B., Zheng, X., … Wang, Q. (2022). Microstructure analysis and quality evaluation of jujube slices dried by hot air combined with radio frequency heat treatment at different drying stages. *Foods, 11*(19), 13. https://doi.org/10.3390/foods11193086

Zhang, C. M., Bian, Y., Hou, S. H., & Li, X. G. (2018). Sugar transport played a more important role than sugar biosynthesis in fruit sugar accumulation during Chinese jujube domestication. *Planta, 248*(5), 1187–1199. https://doi.org/10.1007/s00425-018-2971-1

Zhang, H.-X., Wang, Z.-Z., & Du, Z.-Z. (2022). Sensory-guided isolation and identification of new sweet-tasting dammarane-type saponins from Jiaogulan (*Gynostemma pentaphyllum*) herbal tea. *Food Chemistry, 388*. https://doi.org/10.1016/j.foodchem.2022.132981

Zhang, J., Feng, W., Xiong, Z., Dong, S., Sheng, C., Wu, Y., … Ning, J. (2024). Investigation of the effect of over-fired drying on the taste and aroma of Lu'an Guapian tea using metabolomics and sensory histology techniques. *Food Chemistry, 437*. https://doi.org/10.1016/j.foodchem.2023.137851

Zhang, L., Yu, X., Arun, S., & Zhou, C. (2022). Effect of freeze-thaw pretreatment combined with variable temperature on infrared and convection drying of lotus root. *Lwt, 154*. https://doi.org/10.1016/j.lwt.2021.112804

Zia, M. P., & Alibas, I. (2021). The effect of different drying techniques on color parameters, ascorbic acid content, anthocyanin and antioxidant capacities of cornelian cherry. *Food Chemistry, 364*, 11. https://doi.org/10.1016/j.foodchem.2021.130358