

Effects of moisture content and storage method on the physical properties of dried persimmon during frozen storage

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Abstract

This study investigated the effects of moisture content and the proposed storage method on the physical properties of dried persimmon during frozen storage. The samples with 35.21% moisture content exhibited more rapid changes in surface color, thickness, and hardness of the secondary surface than those with 41.11% and 32.09%. The samples with 35.21% moisture content also exhibited faster sugar crystal formation than those with 41.11% and 32.09% moisture content during frozen storage. Moreover, the three samples' proportions of freezable and bound water differed. The results indicate that controlling the dried persimmon's moisture content between 32.09% and 35.21% is optimal for frozen storage. According to the surface sugar and sensory evaluations, the proposed storage method (samples receive kneading treatment without brushing during the drying process, have an initial moisture content of 33.25%, and are stored in a thermal insulation box) in the present study could maintain the quality of dried persimmon better compared with the common storage method (samples are subjected to kneading and brushing treatment during the drying process, have an initial moisture content of 36.02%, and are stored directly in a freezer).

Keywords: dried persimmon; frozen storage; moisture content; physical properties; sensory evaluation

Introduction

Dried persimmon is a unique dried fruit commonly consumed in Asia, especially in China, Japan, and Korea (Kursun and Karaca, 2018). Traditionally, persimmons are dried to obtain a product with good sensory attributes and storage stability (Nicoletti *et al.*, 2005). Although the drying process extends the shelf life of dried persimmon, the quality of the fruit deteriorates at various stages, such as processing, storage, transportation, wholesale, and distribution (Hyun *et al.*, 2019). Generally, dried persimmons have a higher moisture content than most dried fruits; thus, they are unsuitable for long-term storage. The

shelf life of dried persimmon is up to 6 months based on several factors, such as its initial properties, packaging methods, and storage temperature and time. Therefore, managing these factors is essential for maintaining quality and extending shelf life (Hayashi, 1990; Jia *et al.*, 2019b).

During an investigation of the microbiological, physicochemical, and visual properties of dried persimmons during storage at various temperatures, Hyun *et al.* (2019) found the visual appearance and color index were useful indicators for determining the product's shelf life. The current packaging method is limited by the presence of an oxygen scavenger, the high moisture content of the

dried persimmon, and poor packaging material, leading to quality deterioration during storage. (Hayashi, 1990). For example, Park *et al.* (1989) mentioned that dried persimmon packed with polyethylene bags could be stored for 1 month at room temperature, and those packed with nylon materials could be stored for 1.5–2 months. For instance, Choi *et al.* (2017) It has been mentioned that semi-dried persimmons stored under freezing conditions might expire within six months when packaged in normal paper boxes. However, their shelf life increases to one year when stored in modified atmosphere packaging. Few studies have investigated the quality changes that occur in dried persimmons during storage and transportation.

In Japan, the “Dojo-Hachiya” dried persimmon is considered a high-quality variety. Its surface has a lower initial amount of sugar crystals and is typically stored in the freezer until consumption. During frozen storage and distribution, dried persimmons tend to develop polygonal sugar crystals, which appear as a white powder. The sugar crystals are characteristic of the product. (Hayashi, 1990; Ishii and Yamanishi, 1982; Jia *et al.*, 2020). An excessive amount of sugar crystals on the surface of dried persimmons during storage is considered undesirable because it reduces consumer acceptance. (Jia *et al.*, 2019a). In addition, the color and texture of the flesh are also primary quality attributes that can be influenced by the formation of sugar crystals on the surface. (Hayashi, 1989; Jia *et al.*, 2020). Excessive sugar crystals and changes in quality greatly affect the commercial value of dried persimmons. Therefore, it is essential to understand the mechanisms behind these changes during frozen storage and distribution. In our previous work, we investigated the effects of kneading and brushing treatments on the initial properties of dried persimmon. (Jia *et al.*, 2020). We also clarified how temperature fluctuations affect these quality changes during frozen storage. (Jia *et al.*, 2022). Water plays a complex and vital role in the frozen product; thus, revealing the status and movement of water provides a more comprehensive understanding of the frozen product. The influence of moisture content on the quality changes of dried persimmon during frozen storage has not been thoroughly investigated, especially for long-term frozen storage.

Therefore, the present study aimed to clarify the influence of moisture content on surface sugar crystal formation and the physical and thermal properties of dried persimmon during frozen storage. In addition, a long-term frozen storage method proposed by our previous studies was verified. (Jia *et al.*, 2020, 2022). The quantitative analysis presented herein contributes to our understanding of the effects of moisture content on the shelf life of dried persimmon and supports the use of a new long-term frozen storage method.

Materials and Methods

Sample preparation

Dried persimmons with different moisture contents

This study used a “Dojo Hachiya” variety of dried persimmon. Dried persimmons with different moisture content (41.11%, 35.21%, 32.09%) were obtained from the Japanese Agricultural Co-operative Association (Minokamo City, Japan) and carefully transported to the laboratory at the end of December. Samples were prepared using traditional sun-drying methods and conditions, which were previously described in detail by Jia *et al.* (2020). All the samples were obtained from the same producer and prepared from the same lot. Three batches of dried persimmons were packaged in sealed polyethylene bags and stored in a thermostatic bath at a constant temperature (−20°C). Three pieces of each batch were taken out for analysis every 15 days.

Common storage method and proposed storage method

Two types of samples were prepared for the common storage method and the proposed storage method. Type 1 samples were subjected to kneading and brushing treatment during the drying stage, and the moisture content was 36.02% (wet basis). Type 2 samples received kneading treatment (without brushing) during the drying stage, and their moisture content was 33.25% (wet basis). The kneading and brushing treatments used in this study were previously described in detail by Jia *et al.* (2020) Type 1 was stored in a freezer directly (a common storage method), and Type 2 was first stored in a thermal insulation box (thickness of 3 cm) and then stored in the same freezer (a proposed storage method). The physical properties and sensory qualities were evaluated at day 0 and after 240 days.

Surface sugar analysis

Surface sugar crystal separation

A portion of the surface layer (10×10 mm²) was removed at two positions from the equatorial region of the dried persimmon using forceps and stainless-steel scissors. The forceps then carefully removed flesh from the surface. Subsequently, the surface layer was soaked in 100 mL of distilled water and stirred at a constant speed for 5 min to dissolve the sugar crystals. The free sugars were expressed in mg/cm².

Sugar analysis

The dissolved sugar crystal solutions were filtered through 0.45 µm filters. The white powder was analyzed by a high-performance liquid chromatograph (Model D-2000 Elite; Hitachi, Japan) with a fluorescence detector (Model L-2485; Hitachi, Japan) at a 330–470 nm

wavelength. The column (Asahipak *et al.*, N1620109, Japan) was heated at 40°C. Mobile phase A and mobile phase B consisted of acetonitrile, water, and phosphoric acid in volumetric ratios of 900:95:5 and 750:245:5, respectively. The reaction phase consisted of phosphoric acid, acetic acid, and phenylhydrazine in a volumetric ratio of 542:443:15. The flow rate of the mobile phase was 1 mL/min, and the flow rate of the reaction phase was 0.4 mL/min. The concentrations were calculated from the corresponding sugar standard solutions. Glucose, fructose, and sucrose were obtained from Nacalai Tesque, Inc. (Japan) and used as standard reference sugars for identification.

Surface color measurement

The L^* (lightness and darkness), a^* (redness and greenness), and b^* (yellowness and blueness) coordinates (CIE-Lab) were measured using a color reader (CR-20; Konica Minolta, Inc., Japan). The illuminant was D65, and the observer was a 10° standard observer. The $L^*a^*b^*$ color system is preferred for the quantification of foods with curved surfaces (Garcia-Martinez, 2013). The surface color of the samples was scanned at four different locations in the equatorial region of the dried persimmons. Three pieces were sampled per batch; thus, 12 measurements were taken. Before measurement, the meter was calibrated using a white standard background provided by the manufacturer. The values of the L^* , a^* , and b^* coordinates were recorded, and the whiteness index (WI) value was calculated using Eq.1 to reflect surface color changes.

$$\Delta E = 100 - \sqrt{(100 - L^*)^2 + (a^*)^2 + (b^*)^2} \quad (1)$$

Thickness of the secondary surface

The secondary surface was removed from two different positions of each sample using small scissors and forceps, and the thickness was measured using a digital caliper (Model 19975, Shinwa Rules Co., Ltd., Japan) with a 0.01–150 mm range and expressed as mm. The thickness was measured for six replicates using three pieces for each type.

Texture properties

A universal texture analyzer (TA-XT plus, Stable Micro Systems Ltd., UK) was employed for the texture analysis, using a P/2N (Batch No.11278, Stable Micro Systems Ltd., UK) needle probe with a diameter of 2 mm. The pre-test speed was 2 mm/s, the test speed was 2 mm/s, the post-test speed was 2 mm/s, and the distance was

10 mm. Three different samples were analyzed for their textural properties, and the measurements were repeated four times for each sample.

Thermal properties of persimmon flesh

The thermal properties of the persimmon flesh were analyzed with differential scanning calorimetry (DSC) 6200, equipped with an internal cooling system and Thermal Analysis System software to control experimental runs. The instrument was also equipped with an external liquid nitrogen cooling accessory. The secondary surface layer was removed from a small piece of dried persimmon (approximately 5 mg) and placed in an aluminum pan. The pan was then placed in the DSC head, with an empty pan for reference. The sample was cooled to -40°C and held at a constant temperature for 10.0 min. The sample was then heated to 25°C at a rate of 5°C/min. Experiments were repeated at least three times to ensure the reproducibility of the data. The enthalpy (ΔH) was recorded, and the freezable water content (FW%) was calculated according to the following equation (2):

$$\text{Freezing water} = \frac{\text{Sample}(\Delta H)}{\text{Distill water}(\Delta H) \times \text{Moisture content}} \times 100\% \quad (2)$$

Sensory evaluation

The sensory qualities of dried persimmons that were stored under long-term frozen storage were also evaluated, using a 7-point Hedonic scale (strongly dislike = 1 and strongly like = 7). Then, 20 panelists (10 females, 10 males, aged 20–28 years) were recruited from the food process laboratory (Gifu University, Gifu, Japan). Most had previous sensory evaluation experience and were selected based on their availability and willingness to participate in the evaluation of dried persimmons. Each group was assessed according to appearance, surface color, flesh color, flavor, texture, and overall acceptance attributes. The overall flavor intensity was also evaluated when the package was opened. Water was provided to panelists to rinse their mouths between samples during the sensory evaluation.

Statistical analysis

The data were analyzed by Analysis of Variance (ANOVA), and multiple comparisons between mean values were made using Tukey's test in Origin software (version 2016; OriginLab Corp., Northampton, MA, USA).

The level of significance was $P < 0.05$. All the data are expressed as the means and standard deviation (SD).

Results and Discussion

Sugar crystals on the surface of dried persimmon

Our previous research revealed that the free sugars in “Dojo-Hachiya” dried persimmon and the surface sugar crystals are fructose and glucose. (Jia *et al.*, 2022). Figure 1 shows the effect of moisture content on the formation of surface sugar crystals during 105 days of frozen storage. For all samples, the number of sugar crystals on the surface of the dried persimmon increased during frozen storage, and there was a significant difference between the samples with different moisture content ($P < 0.05$). Samples with 32.09% and 35.21% moisture content formed more sugars than those with 41.11% moisture content. In addition, the sugar crystals formed faster on the samples with 35.21% moisture content than those

with 32.09%. After 105 days of frozen storage, the final ratios of glucose to fructose were 2.41, 3.44, and 3.21 when the moisture contents were 41.11%, 35.21%, and 32.09%, respectively. Overall, glucose crystals seemed to form more quickly than fructose crystals.

For samples with 41.11%, 35.21%, and 32.09% moisture content, the number of sugar crystals increased to 7.33, 19.38, and 10.89 mg/cm^2 during frozen storage (Table 1). The data suggest a relationship between the fruit’s moisture content and the sugar crystal formation rate during frozen storage. Thus, it is necessary to control the moisture content of the final product during the drying process.

Surface color

Color is a significant quality attribute in dried food products (Francis, 1995). Here, the WI was used to evaluate the overall whiteness of the dried persimmons during frozen

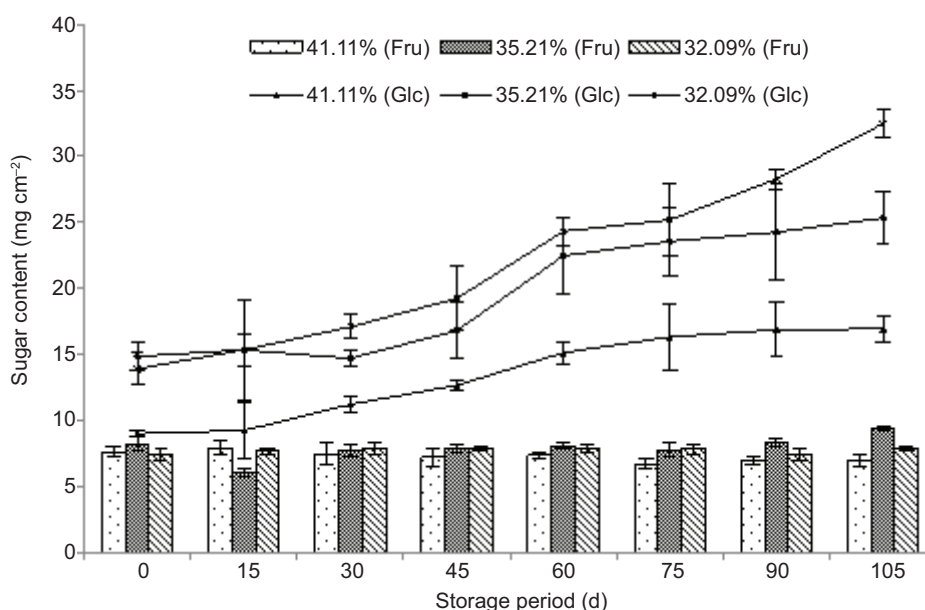


Figure 1. Effect of moisture content on the formation of sugar crystals on the surface of dried persimmons during frozen storage. The data points and error bars represent the means and standard deviations of the measured values ($n = 6$) at each time point.

Table 1. Sugar crystal composition, expressed as individual fructose and glucose (mg/cm^2), after 105 days of frozen storage at -20°C .

Moisture content	Fructose (mg/cm^2)			Glucose (mg/cm^2)		
	0 day	105 days	Increased amount	0 day	105 days	Increased amount
41.11%	7.64 ± 0.41^a	7.04 ± 0.46^a	-0.60	9.02 ± 0.26^c	16.95 ± 1.02^d	7.93
35.21%	8.25 ± 0.54^a	9.42 ± 0.29^b	1.17	14.27 ± 1.20^c	32.48 ± 1.03^d	18.21
32.09%	7.46 ± 0.51^a	7.89 ± 0.11^a	0.43	14.87 ± 1.02^c	25.33 ± 2.01^d	10.46

Different superscript letters in the same row indicate a significant difference between groups ($P < 0.05$); the data are expressed as mean values \pm SD.

storage as an indicator of discoloration on the surface. A higher WI value indicates a much whiter surface (Pathare *et al.*, 2013). Figure 2 shows the changes in the whiteness of dried persimmon with different moisture content during frozen storage. The moisture content affected the WI values of dried persimmon. The initial WI values of the samples with 35.21% and 32.09% moisture content were 53.68 and 53.12, respectively, significantly higher than those with 41.11% moisture content (40.37, $p < 0.05$). In addition, the moisture content affected the change rate of WI values. The WI values of the dried persimmons with 41.11% moisture content showed little variation and were consistently lower than the other two groups. At higher moisture content (41.11%), the sugars did not readily crystallize at the surface. Moreover, the WI values of the dried persimmon with 35.21% moisture content changed more rapidly than the other two types. During the storage or distribution of dried persimmons, color may change due to several chemical and biochemical reactions, such as internal physicochemical changes or ascorbic acid browning (Carel *et al.*, 2010). With the extension of frozen storage to 105 d, the WI value increased due to the formation of sugar crystals and physicochemical changes or ascorbic acid browning on the surface.

Changes in thickness of the secondary surface

For dried persimmon, a secondary surface forms during the drying process because of the presence of pectin,

which affects the quality of products (Manabe *et al.*, 1980). The secondary surface is formed by surface moisture evaporation and tissue shrinkage caused by applied heat during drying. It is also susceptible to quality changes. (Hayashi, 1989), and these properties will continue to change during frozen storage (Jia *et al.*, 2022).

Figure 3 shows the effects of moisture content on the thickness of the secondary surface during frozen storage. As shown in Figure 3, the initial average thicknesses of the samples were different. The thickness of dried persimmon, with 35.21% (3.08 mm) and 41.11% (3.02 mm) moisture content, was more significant than the dried persimmon, with 32.11% (2.77 mm) moisture content. The thickness of dried persimmon increased in the first 30 days of frozen storage and then decreased. The thickness of the dried persimmon with 35.21% moisture content changed faster than the dried persimmon with 32.11% and 41.11% moisture content. An increase in the thickness of the secondary surface was expected due to moisture release in the vicinity of the surface and the generation of a new secondary surface layer. The decrease in thickness may be a result of tissue shrinkage due to moisture loss of the outer layer.

Texture of secondary surface

The texture of dried fruit will influence consumer sensory perception and commercial value. (Fernandes *et al.*, 2011).

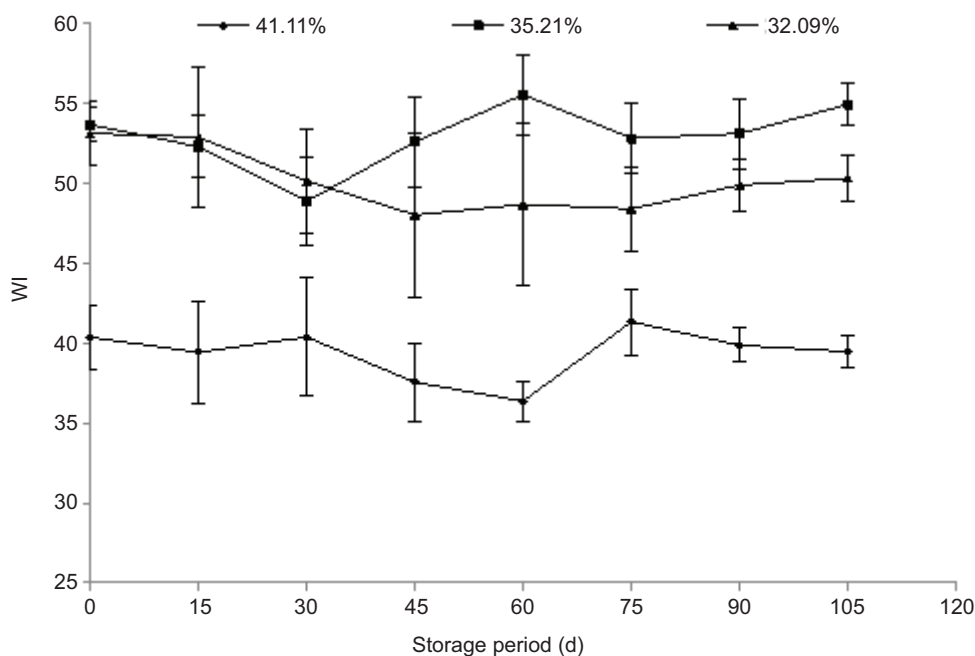


Figure 2. Effect of moisture content on the WI values of dried persimmons during frozen storage. The data points and error bars represent the means and standard deviations of the measured values from three pieces ($n = 12$) at each time point.

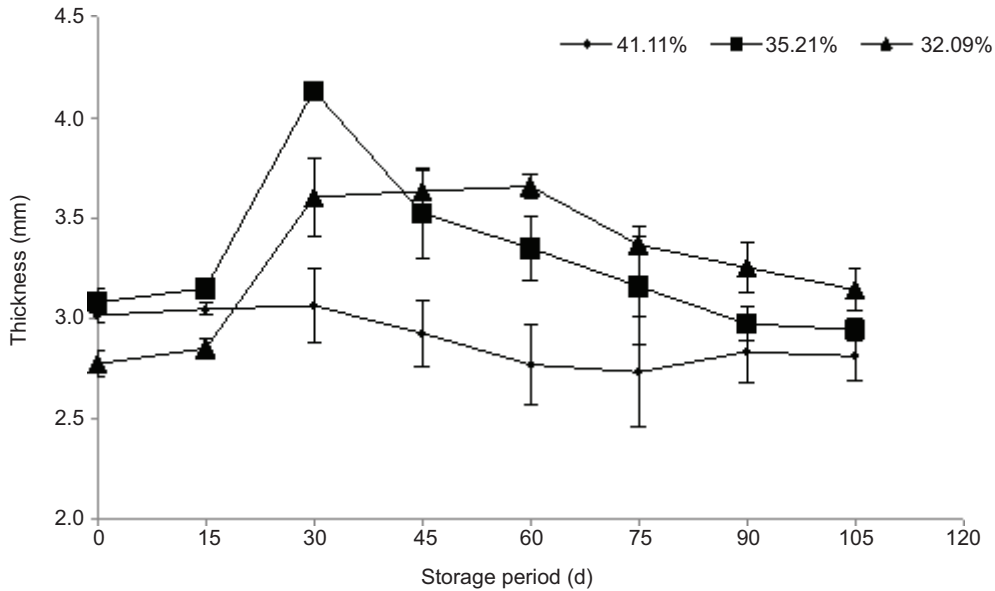


Figure 3. Effects of moisture content on the thickness of the secondary surface of dried persimmon during frozen storage. The data points and error bars represent the means and standard deviations of measured values from three pieces (n = 12) at each time point.

The maximum puncture forces of dried persimmons with different moisture contents are given in Table 2. The initial average hardness of dried persimmon with 35.21% (156.89g) and 32.09% (201.46g) moisture content was higher than the dried persimmon with 41.11% (96.85g) moisture content. This is because higher moisture content leads to soft cellular tissue. In addition, the hardness of the secondary surface changed over 105 days of frozen storage. The hardness of samples with 35.21% moisture content decreased to 27.29g, which was more significant than the samples with 32.09% (17.82g) moisture content after 105 days of frozen storage. The hardness of samples with 41.11% moisture content remained relatively stable during storage. The hardness is related to the maximum force needed for the first bite during eating. (Guine and Barroca, 2012), and the hardness of the secondary surface may affect the mouthfeel. The results suggest the secondary surface of dried persimmon became softer after frozen storage, and the moisture content influenced the change in hardness during frozen storage.

Ice crystals that form during frozen storage become larger as the storage period increases. (Ullah *et al.*, 2014) The freezing, thawing, and recrystallization of foods increase crystal size and damage the internal structure. (James *et al.*, 2015). The results imply that the microstructure of the secondary surface of the dried persimmon changed after frozen storage. The sugars crystallized on the surface may influence the proportions of the components, which may also affect the structural properties of the secondary surface. (Jia *et al.*, 2020).

Table 2. Effect of moisture content on the textural properties of the secondary surface of dried persimmons during frozen storage.

Moisture content	Hardness (g)		
	0 day	105 days	Average decreased value
41.1%	98.65±14.12	104.31±13.27	-5.66
35.2%	156.89±20.36	129.60±25.13	27.29
32.1%	201.46±12.13	183.84±21.23	17.82

The data are expressed as mean values±standard deviation (SD).

Thermal properties of dried persimmon flesh

The formation of ice crystals during freezing and frozen storage causes physical changes to the structure of foods. (James *et al.*, 2015). The content of freezable water plays a critical role in the amount, size, and distribution of ice crystals during frozen storage, so it is necessary to determine the freezable water content. (Kontogiorgos *et al.*, 2008; Tran *et al.*, 2008) DSC determined the samples' thermal characteristics, which have been used to confirm the presence of freezable water in dried persimmon with different moisture content. In the DSC measurement, the enthalpy of ice crystals derived from the heat flow curve was recorded and converted to the percentage of freezable water content via Eq. 2.

Figure 4 and Table 3 show the enthalpy of ice formation and the percentage of freezable water inside the flesh

of dried persimmon with different moisture contents during frozen storage. The enthalpy decreased significantly with decreasing moisture content in the product ($p < 0.05$) (Figure 4). The enthalpy decreased to 1.03 mJ/mg and 0 mJ/mg when the moisture content was 35.21% and 31.21%, respectively. Moreover, the proportion of freezable water inside the dried persimmon also decreased with decreasing moisture content (Table 3), and the freezable water in the flesh was almost 0% when the moisture content was lower than 35.21%. This could be due to water immobilization by the pectin-sugar system in the flesh, and the high hydrophilic characteristic of pectin could reduce the water mobility of the flesh. (Einhorn-Stoll, 2018). During frozen storage, the samples with greater amounts of freezable water formed more ice crystals in the flesh and exhibited more quality changes. Xuan *et al.* (2017) It was also reported that the proportion of freezable water might damage the product more, generating a loose and broken structure.

In the present study, the proportion of freezable water decreased, which may be caused by the proportion of water molecules bound to pectin during drying. To minimize the negative impacts of frozen storage on the

products, the moisture content of dried persimmon should be controlled to a suitable range.

Comparison of the common storage method and proposed storage method

Surface sugar changes during frozen storage

The sugars on the surface of dried persimmons were fructose and glucose (Jia *et al.*, 2020). Figure 5 compares the change in sugar crystal formation on the surface of dried persimmon after 240 days of frozen storage with the common and proposed storage methods. The surface sugar of all samples significantly increased after frozen storage ($P < 0.05$); the samples under the common storage method crystallized more than in the proposed storage method ($P < 0.05$). The moisture content of the dried persimmons decreased after long-term frozen storage, and the moisture content of the samples with the standard storage method decreased by 1.75%, which was more than those stored with the proposed method (decreased by 0.65%) (Table 4). Thus, the reduction in moisture content may induce the formation of sugar crystals (Jia *et al.*, 2022).

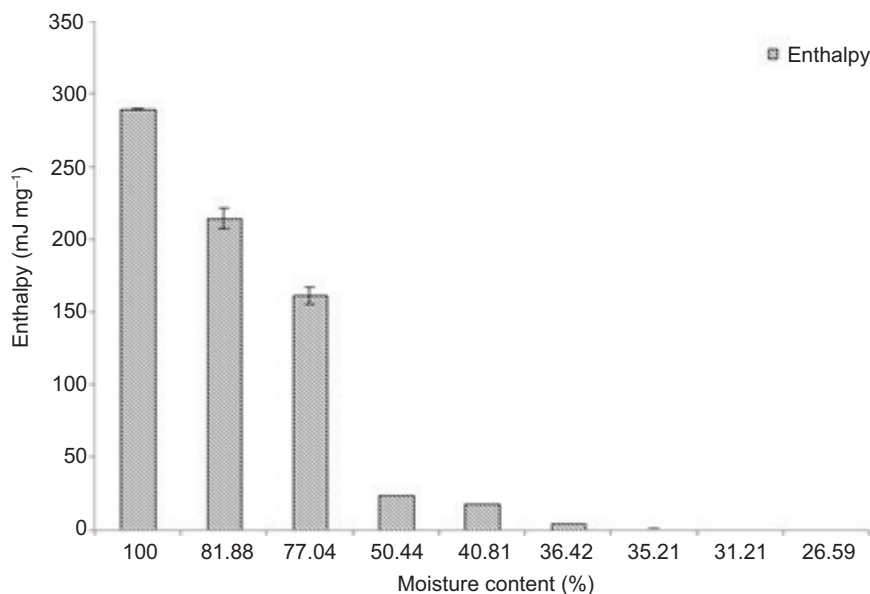


Figure 4. Effect of moisture content on the enthalpy of the dried persimmon samples. The data points and error bars represent the means and standard deviations of measured values from three pieces at each time point.

Table 3. The proportion of freezable water and non-freezable water in the dried persimmon samples.

Moisture content (%)	100	81.88	77.04	50.44	40.81	36.42	35.21	31.21	26.59
Freezable water	100	74.06	55.65	8.12	6.10	1.50	0.35	0	0
Non-freezable water	0	7.82	21.39	42.32	34.71	34.92	34.86	31.21	26.59

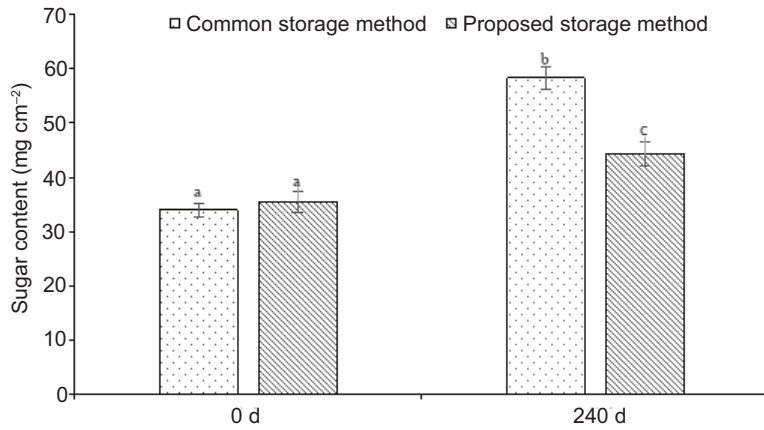


Figure 5. Influence of different storage methods on the surface sugar content of dried persimmon. The results are expressed as the mean of six trials. a, b, and c indicate significant differences ($P < 0.05$).

Table 4. The changes in moisture content under different storage methods after 240 days of frozen storage.

Storage conditions	Common storage method	Proposed storage method
Decreased moisture content (%)	1.75	0.65

Sensory evaluation

Sensory analysis involves inspecting a product using the senses, such as appearance, flavor, color, texture, and overall acceptance. (Singh-Ackbarali and Maharaj, 2014). Figure 6 compares the initial sensory evaluation of the dried persimmon samples and the sensory qualities after 240 days of frozen storage. The sensory evaluation results were similar between the two groups before storage (Figure 6a). After 240 days of frozen storage, the differences between the two groups were apparent (Figure 6b). Appearance is the first characteristic the human senses perceive, including surface color, shape, and size. The

shape and size of dried persimmon did not change during frozen storage; however, there was a significant difference in the appearance of the two types of samples. The surface color change of dried persimmon may cause this. During storage, the number of sugar crystals on the surface increased, influencing the surface color. The results suggest the proposed storage method could maintain the appearance accepted by consumers; however, there was no significant difference in flesh color between the two storage methods.

Dried persimmons possess a unique flavor, and the change in flavor was not significant. Texture is a combination of touch, mouthfeel, sight, and sound. It is one of the most imperative features of food. (Barrett *et al.*, 2010). After 240 days of frozen storage, the texture of the samples under the different storage methods was significantly different. The texture of dried persimmons under common storage conditions decreased more than the samples under the proposed storage method. Therefore, the storage method proposed in this study greatly enhanced the stability of dried persimmons during frozen storage.

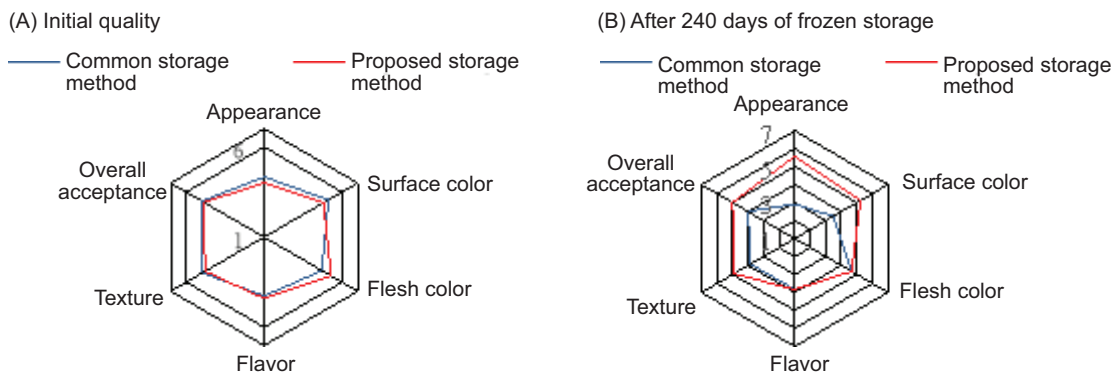


Figure 6. Sensory evaluation of the dried persimmons under different storage methods.

Conclusion

This study found that the initial moisture content of dried persimmon affected the change in the quality of the fruit during frozen storage; for the samples with 35.21% moisture content, the color, thickness, and hardness of the secondary surface changed more rapidly compared to those with 41.11% and 32.09% moisture content during frozen storage. In addition, surface sugar crystals formed earlier than the samples, with 41.11% and 32.09% moisture content. According to the DSC data, the proportions of freezable and bound water differed in the different types of samples. Thus, controlling the moisture content of the dried persimmon between 32.09% and 35.21% was optimal for frozen storage. The present study also showed that the proposed storage method was advantageous compared to the common one. The results implied that the type of treatment used during drying, appropriate moisture content, and temperature control were important to maintain the quality of dried persimmon during frozen storage. Using this information, it was possible to manage the quality of dried persimmon during frozen storage and enhance consumer acceptance.

Conflict of Interest

This manuscript has not been published elsewhere and is not under consideration by another journal. We have approved it and agree to submit it to the Italian Journal of Food Science. There are no conflicts of interest to declare.

Availability of Data and Material

The original data used to support this study's findings are available from the first author and corresponding author upon request.

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