



TITLE:

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CITATION:

Nakagawa, Kyuya ...[et al]. Influence of processing conditions of atmospheric freeze-drying/low-temperature drying on the drying kinetics of sliced fruits and their vitamin C retention. *Journal of Agriculture and Food Research* 2021, 6: 100231.

ISSUE DATE:

2021-12

URL:

<http://hdl.handle.net/2433/279270>

RIGHT:

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Contents lists available at ScienceDirect

Journal of Agriculture and Food Research

journal homepage: www.sciencedirect.com/journal/journal-of-agriculture-and-food-research



Influence of processing conditions of atmospheric freeze-drying/low-temperature drying on the drying kinetics of sliced fruits and their vitamin C retention

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ARTICLE INFO

Keywords:

Atmospheric freeze-drying
Drying kinetics
Shrinkage
Vitamin C
Apple
Kiwi

ABSTRACT

Sliced fruits (apple and kiwi) were dried under atmospheric freeze-drying (AFD) and/or low-temperature drying (LTD) conditions, and the drying kinetics, resultant degree of shrinkage, and vitamin C retention were evaluated. The air temperature programs were set in the range of -20 to 10 °C, and the impact of the sub-zero temperature setting was investigated. As expected, shortening the sub-zero temperature time and increasing the air temperature led to a shorter total drying time. Notably, the application of freezing had an effect to reduce the drying time. The application of the LTD condition without freezing significantly increased the total drying time compared with that of the drying condition with freezing. When the temperature was increased stepwise from -20 to 10 °C, the total drying time was effectively reduced when the sub-zero temperature was maintained for a relatively long time. This was the most practical drying program for maintaining product quality. It was found that drying at sub-zero temperatures has advantages in reducing product shrinkage; however, the impact was not remarkable. The application of sub-zero temperature conditions was advantageous for vitamin C retention for both apple and kiwi drying. However, if the negative effect of freezing on quality is significant, simple LTD conditions may be a good compromise for balancing total drying time and product quality.

1. Introduction

It is widely recognized that the quality of a dried product is strongly influenced by processing conditions, where changes in physicochemical, chemical, and biological properties can occur depending on various parameters. It is not surprising that the application of a high drying temperature reduces the drying time, leading to serious product damage [1]. Sensitivity to heat is dependent on the type of food, ingredients, and how these ingredients are stabilized in a food structure. Water removal is also a cause of product damage. The capillary force of the water meniscus in the porous structures of the product leads to shrinkage or structural collapse during water vaporization. This stress can be minimized by freeze-drying. The vacuum freeze-drying (VFD) process is widely used for a variety of food, drugs, and products. Atmospheric freeze-drying (AFD) is an alternative freeze-drying technique that achieves ice sublimation at atmospheric pressure, where the water vapor

pressure difference between the frozen zone and the ambient gas is the driving force [2–4].

The dehydration mechanism of freeze-drying is mainly sublimation from the ice crystal phase (primary drying), which does not result in damage due to the capillary force. Dehydration from the freeze-concentrated phase occurs by evaporation above the glass transition temperature of the maximally freeze-concentrated matter (T_g). The T_g of agricultural products is generally low; for example, that of strawberry ranges from -33 to -41 °C, that of peach is approximately -36 °C [5], apple at approximately -58 °C [6], persimmon at approximately -55 °C [7], pineapple at approximately -52 °C [8], and tomato at approximately -59 °C [9]. Hence, when a product is dried under typical AFD conditions (i.e., approximately -20 °C or higher), the drying products are under the influence of capillary forces. Product deformations such as shrinkage, melt-back, and/or structural collapse occur as a consequence of glass-rubber-liquid transitions [1,10–12]. Although the AFD process

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<https://doi.org/10.1016/j.jafr.2021.100231>

Received 30 August 2021; Received in revised form 22 October 2021; Accepted 1 November 2021

Available online 4 November 2021

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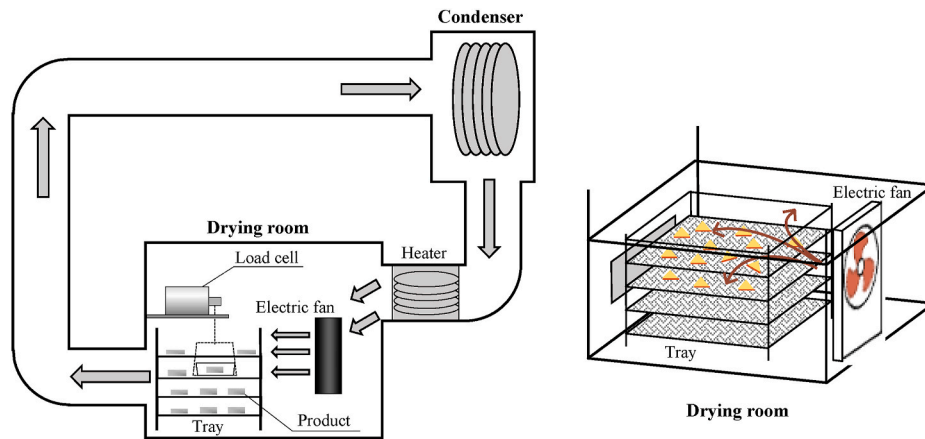


Fig. 1. Schematic illustration of the dryer component.

Table 1
Settings of the applied air temperature programs.

Program ID	Freezing step temperature (holding time)	Setting temperature in drying step	Actual temperature in drying step
P1	-22.5 °C (1000 min)	-10.0 °C (7500 min)	-10.0/-10.7 °C
P2	-22.5 °C (1000 min)	0 °C (5000 min)	-0.9/-1.8 °C
P3	-22.5 °C (1000 min)	10.0 °C (4500 min)	8.5/7.1 °C
P4	-22.5 °C (1000 min)	-10.0 °C (720 min)	-11.9 °C
		-0.0 °C (720 min)	-1.5 °C
		5.0 °C (720 min)	7.6 °C
P5	N/A	10.0 °C (1000 min)	10.3 °C
		10.0 °C (4500 min)	11.3/8.4 °C
HAD ^a	N/A	70 °C (420 min)	N/R

^a Hot air drying (HAD) was performed using a different dryer for reference data acquisition.

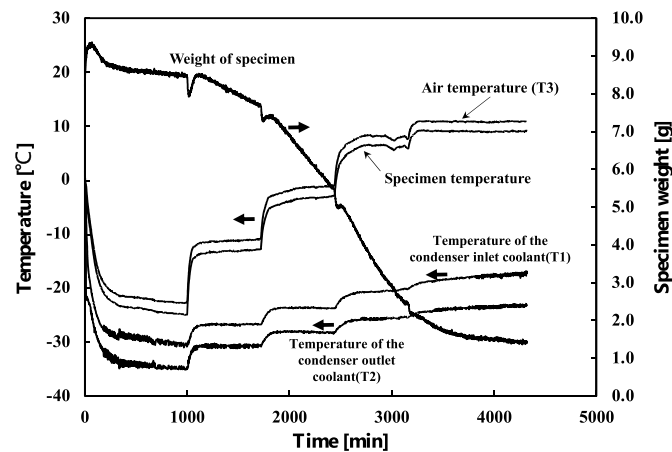


Fig. 2. An example of drying run (temperature program P4 was applied to dry an apple slice).

is considered to be advantageous for quality assurance owing to the use of low temperature and ice sublimation, the negative impact of quality loss due to structural collapse should be considered. However, there have been few detailed systematic studies on this issue.

To perform drying at sub-zero temperature, the air humidity must be kept at an appropriately low level. A heat-pump system is commonly used to create low-humidity air [13]. The condenser temperature of the heat-pump unit is necessarily set lower than the product temperature during drying, and the dehumidified air must be properly heated to

realize an appropriate driving force for mass and heat transfer. Several studies on the AFD process have sought to optimize this heat-pump drying system in terms of energy consumption and drying rate [14–17]. Mass and heat transfer properties can be intensified by drying in a fluidized system and/or hybridizing drying components with external heating systems, such as microwave irradiation systems and ultrasound systems [17–23]. The AFD process merits easy operation with simplified drying components. Performing the process above freezing temperatures, recognized as low-temperature drying (LTD), can also be carried out with the same drying device [24]. Considering the long drying time required for AFD, it is practical to combine AFD and LTD conditions. The motivation to apply AFD is to reduce the loss of product quality; therefore, it is of interest to determine the impact of AFD and LTD conditions on product quality.

In this study, apple and kiwi slices were dried under AFD and LTD conditions. The drying programs were divided into cooling, freezing, and drying stages, and experiments were conducted with different air temperature settings and sample feeding timing. The degree of shrinkage and retention of vitamin C were evaluated as quality factors. We aimed to identify the advantages of the AFD process and the optimal drying conditions by systematically investigating the effects of drying temperature programs on quality. Using the same drying equipment for these verifications, we expect to eliminate the changes that depend on equipment characteristics such as thermal and air flow characteristics. In particular, we believe this approach is important in order to study the effects of non-isothermal conditions with a wide temperature range on the drying kinetics and product quality factors.

2. Materials and methods

2.1. Materials

Fresh apple (Sun-Fuji, Aomori, Japan) and kiwi (green kiwi imported from Chili) were purchased from a domestic market. The skin of the kiwi was first removed and then the kiwi was cut into slices 5 mm thick (horizontal to the direction of the core). The wet basis moisture contents of the apple and kiwi were approximately 0.85–0.88 and 0.86–0.88, respectively. Apples with their skin were also cut horizontally to the direction of the core into slices 5 mm thick, and cut into four quarters with the core parts removed. The sliced apple was immediately soaked in 5 wt% NaCl solution for 1 min, and the surface solution was removed using a paper towel. Metallic acid, ascorbic acid, dehydroascorbic acid, and acetic acid were purchased from Wako Pure Chemical Industries (Osaka, Japan). All reagents were of analytical grade.

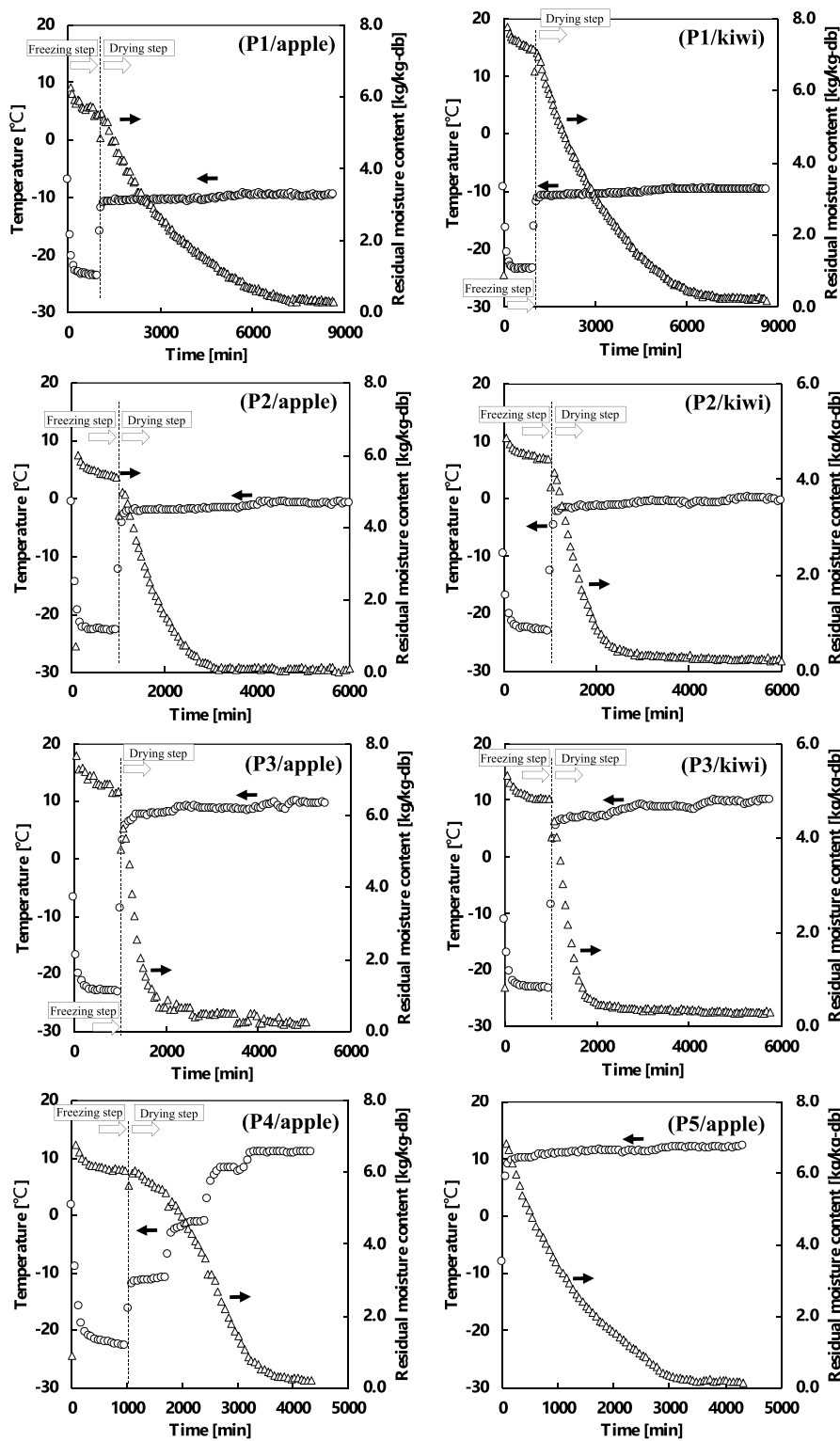


Fig. 3. Drying kinetics of apple and kiwi slices dried using different temperature programs.

2.2. AFD protocol

The atmospheric freeze-dryer composed of a condenser and heating device was set up as schematized in Fig. 1. The condenser temperature was maintained at approximately $-30\text{ }^{\circ}\text{C}$ by the coolant circulating in the heat exchanger. The temperature of the out-gas from the condenser was controlled by the heating device in the range of -20 to $10\text{ }^{\circ}\text{C}$

according to the program details shown in Table 1. The freeze-drying program is divided into cooling, freezing, and drying stages, and the programs P1–P5 described below differ in their air temperature setting and sample feeding timing. In programs P1, P2, P3, and P4, the specimens were frozen in the drying room at the first cooling stage and then dried in the subsequent heating stage. The air temperature was increased stepwise in program P4, so it contained both AFD and LTD

Table 2
Results of drying experiments.

Drying program and raw material		DT50* [min]	DT30* [min]	Final residual moisture content* (wet basis) [%]	RVP [%]
P1	Apple	4328	6002	18.6	61
P1	Kiwi	4158	5331	17.5	55
P2	Apple	1294	1683	18.4	60
P2	Kiwi	918	1528	19.4	53
P3	Apple	737	1369	17.9	57
P3	Kiwi	610	1251	21.7	50
P4	Apple	2043	2350	18.3	52
P4	Kiwi	N/R	N/R	18.5	49
P5	Apple	2258	2757	17.7	61
P5	Kiwi	N/R	N/R	13.9	46
HAD	Apple	N/R	N/R	16.5	37
HAD	Kiwi	N/R	N/R	19.6	37

*DT50: drying time to reach 50% residual moisture content (wet basis).

*DT30: drying time to reach 30% residual moisture content (wet basis).

*Residual moisture content measured using a moisture meter.

conditions. The air temperature applied in program P3 was a LTD condition where the air temperature was applied above the freezing point throughout the drying period, and the samples were frozen in a separate freezer beforehand. However, program P5 did not include a freezing stage. The airflow rate in the drying room was maintained by an electric fan at 0.43 m/s. Slices of fruit (i.e., apple and kiwi) were placed on a drying shelf made of steel mesh. A cage of aluminum mesh was suspended in the dryer with a connection to a load cell (U2X1-0.5-A, A&D Co., Ltd., Japan). A slice of fruit was placed in the cage to monitor weight change during drying. In order to compare and verify the drying kinetics, the parameters DT30 and DT50 were introduced. The drying time until the residual moisture content reaches 30% (50%) was defined as DT30 (DT50). These values were obtained by finding the corresponding residual moisture content and time on the obtained weight change curve measured by the load cell. The dried samples were immediately placed in nylon packs with silica gel and stored at 4 °C.

2.3. Moisture content and relative vapor pressure measurement

The moisture content of the dried samples was measured using an infrared heater moisture meter (MS-70, A&D Co., Ltd., Japan). The relative vapor pressure (RVP) of the samples was measured using a wireless humidity and temperature sensor (MESH-100TH, Sony Co., Ltd., Japan). Dried samples were placed in a nylon pack (Ziploc®, Asahi Kasei Home Products Corporation, Japan) with the sensor and equilibrated at 4 °C for 3 h. The resultant RVP was recorded without opening the pack.

2.4. Vitamin C content measurement

Ascorbic acid and dehydroascorbic acid were extracted from fresh and dried samples using a metaphosphoric acid solution. Fresh kiwi (1.5 g) was ground in a ceramic mortar with 3 mL of 6 wt% metaphosphoric acid solution. Fresh apple (1.5 g) was ground in the same manner with 1.5 mL of 6 wt% metaphosphoric acid solution. Similarly, 0.3 g of dried sample (apple and kiwi) were ground with 2.7 mL of the metaphosphoric acid solution. The mixture was filtered through a polyethylene mesh (144 µm). The filtered solution was collected in a 2 mL microtube and centrifuged for 2 min at 10,000 rpm. The supernatant was again filtered using a membrane filter with a mesh size of 0.2 µm. This solution was subjected to HPLC with a pump and UV/Vis spectrophotometer units (SPD-10AV and LC-20AT, Shimadzu, Kyoto, Japan). A reverse-phase column (5C18-PAQ water, 3.0 × 150 mm, YMC, Kyoto, Japan) was employed for the analyses with 1.0 wt% acetic acid solution for the mobile phase at a flow rate of 0.4 mL/min. The absorbance at 245 nm was monitored to evaluate the concentrations of ascorbic acid and

dehydroascorbic acid in the extract. The vitamin C content in the present study corresponded to the sum of ascorbic acid and dehydroascorbic acid contents. The measurements were performed at least three times for each condition. The data obtained from the experiments were analyzed using a one-way analysis of variance (ANOVA).

2.5. Product appearance observation and shrinkage measurement

Images of the samples were captured using a digital camera under a fixed light setting. In order to calibrate the size of the sample, a scale image was also taken at the same time. The ratio of shrinkage was evaluated by taking the ratio of the visible cross-sectional area of the dried sample to that of the original sample. The cross-sectional area was estimated using digital images taken from the samples using ImageJ 1.80 (NIH, USA). At least three measurements were performed for three different samples under each condition. The data obtained from the experiments were analyzed using a one-way ANOVA.

3. Results and discussions

3.1. Drying runs and product appearance

The results of the drying run are shown in Fig. 2, where the air temperature and specimen weight are plotted as a function of time. The beginning of the 1000-min period corresponds to the freezing step. During this step, ice sublimation progresses, as confirmed by the temperature difference between the air and the specimen. However, the total amount of dehydration during this step was quite small. As the air temperature increased, the drying rate increased, as confirmed by the weight loss of the specimen. The specimen temperature was approximately 2.0 °C lower for all the applied air temperatures in the range of -20 to 10 °C. The difference between the inlet and outlet coolant temperatures of the condenser is linked to the energy load required for dehumidification, suggesting that the drying is being conducted properly [24].

The drying kinetics of specimens dried at different temperatures are shown in Fig. 3, where the dry basis moisture content is plotted as a function of time. The residual moisture content (wet basis) and RVP values of the collected specimens are listed in Table 2. As confirmed from the figures for both apple and kiwi drying, the curved moisture content profiles observed when the air temperatures were kept constant suggested that drying occurred mainly during the falling rate drying period. This is common in freeze-drying and food drying, where the drying rate is always limited by the mass transfer resistance of the product. The trends of the drying kinetics of apple and kiwi slices and the final residual moisture contents were not significantly different. The RVP values of the dried kiwi slices (approximately 55–60%) were slightly higher than those of the apple slices (approximately 50–55%). As expected, the total drying time could be reduced by applying a higher air temperature. The drying rates at the early stage were compared with the values of DT50 (Table 2). Here, as explained above, DT50 and DT30 are defined as the drying time to reach 50% and 30% residual moisture content (wet basis), respectively. Approximately 4200 min was required to reach a moisture content of 50% (wet basis) when the air temperature was -10 °C (i.e., program P1). It could be reduced to 1100 min with an air temperature of 0 °C (P2), and to 700 min at 10 °C (i.e., P3). The subsequent drying stage (from 50% to 30% moisture content) was also significantly influenced by the air temperature; approximately 1500 min was required for P1, 500 min for P2, and 600 min for P3. It is not surprising, as confirmed from these results, that the air temperature has a critical impact on the drying rate.

For P4, the temperature was increased step-by-step from -20 °C to 10 °C. This approach effectively reduced the total drying time while maintaining the products at sub-zero temperatures for a relatively long time. When it is necessary to keep the product in a frozen state for as long as possible to maintain product quality, this may be the most

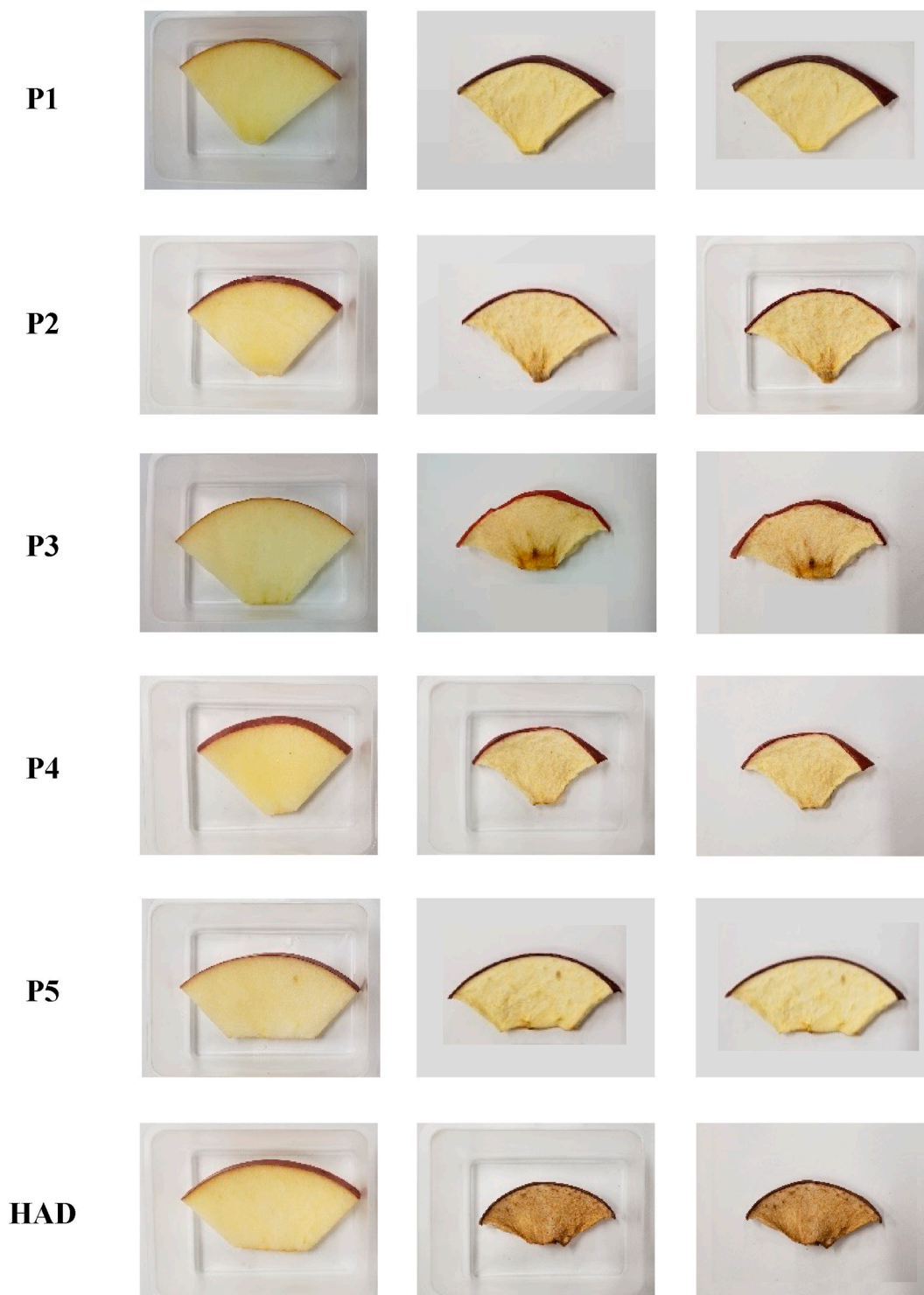


Fig. 4. Appearance of dried apple slices.

practical drying program. As confirmed, DT50 and DT30 were approximately 2000 and 2300 min, respectively. One can see from the drying curve that the drying rate was accelerated by the stepwise temperature program.

P5, alternatively, is a simple LTD condition in which the products were dried without being frozen at a set point of 10 °C. This condition did not achieve shorter drying times than the other conditions that implemented freezing, such as P2, P3, and P4. Even though the same set point was applied in P3 and P5, DT50 and DT30 in the P5 condition resulted in approximately twice as much as those for P3. It is interesting

to note that freezing had a positive impact on reducing the total drying time, whereas the impact was not obvious in the final residual moisture content and RVP. The reduction of drying time could be caused by the destruction of cellular structures by the formation of ice crystals during freezing [25,26]. Further verification is needed to clarify the relationship between the frozen structure and the drying kinetics under AFD conditions. It is also important to determine the impact of the drying program on the quality of the product, which is the main concern of the following section.

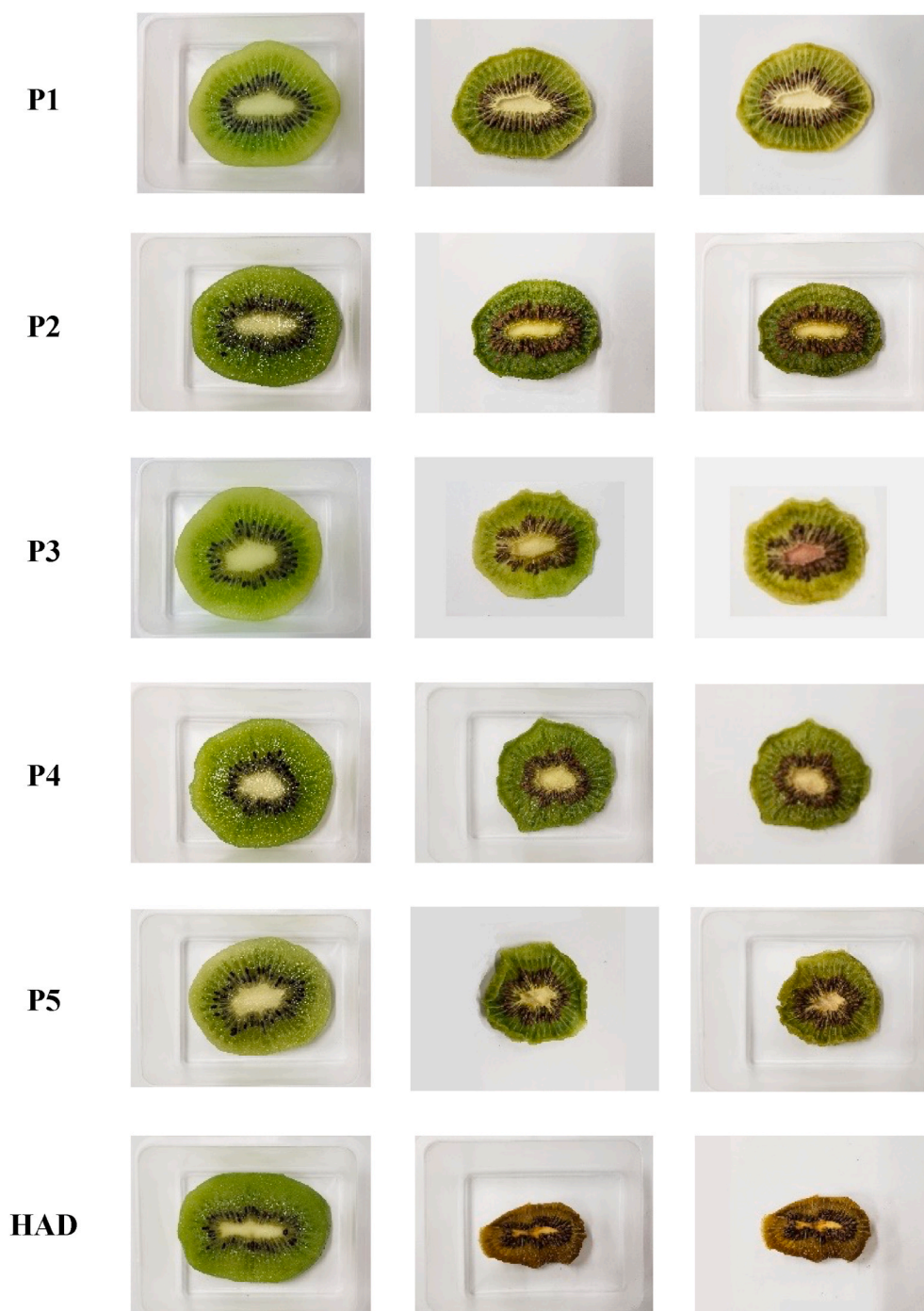


Fig. 5. Appearance of dried kiwi slices.

3.2. Appearance and shrinkage

The appearances of the samples prepared under different drying conditions are compared in Figs. 4 and 5. It can be seen that the colors of the original samples are well maintained in all the applied drying operations, which is a significant difference from the case where hot air drying at 70 °C was applied. The difference in color after applying the drying conditions P1–P5 was not substantial, and it is reasonable to assume that any difference is due to the differences between the original colors. No significant browning was observed, even after one month of storage, for both the dried apple and kiwi specimens. Notably, the color fading was slight, so the retention of nutritional ingredients could be expected. Because the difference between the original colors of each

specimen is evident, and to conduct quantitative color evaluation using the color difference under each drying condition, a sophisticated experimental design to control for the original colors is required, thus no quantitative color evaluation was conducted in this study. Instead, the change in shrinkage before and after drying was quantitatively evaluated, as shown in Fig. 6. The degree of shrinkage due to drying of both the apple and kiwi specimens was approximately 60% under all operative conditions. The difference between the specimens prepared via the P1–P5 conditions was not statistically significant. However, when compared with the HAD conditions, the shrinkage was significantly reduced for kiwi specimens dried via P1, P2, and P4 and apple via P1, P2, P4, and P5. This suggests that the application of sub-zero drying temperatures hinders product shrinkage.

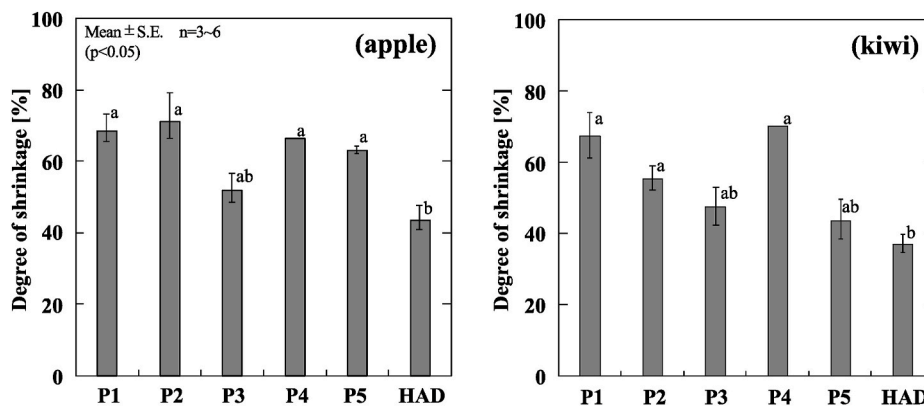


Fig. 6. Degree of shrinkage as a cause of drying and storage.

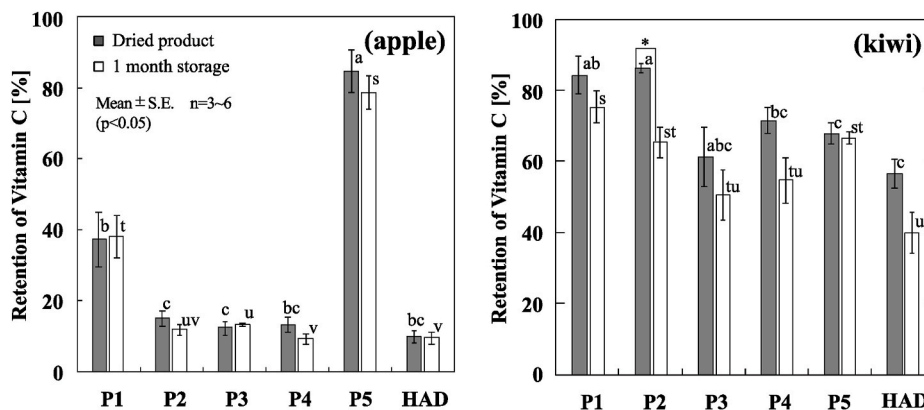


Fig. 7. Retention of vitamin C in dried specimens.

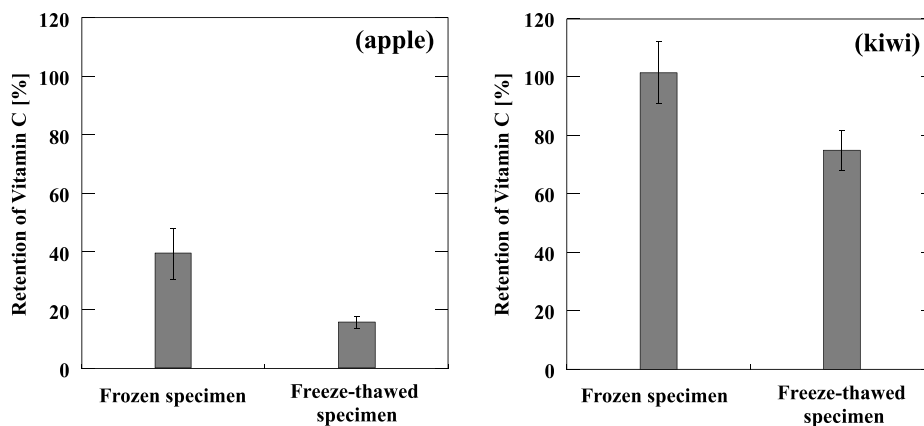


Fig. 8. Retention of vitamin C in frozen and freeze-thawed specimens.

3.3. Vitamin C retention

The amount of residual vitamin C was measured as the influence of the present drying conditions on the retention of nutritional value. Vitamin C (L-ascorbic acid) is a water-soluble compound that can be lost from food through oxidation and/or direct leaching during processing and storage. The oxidation mechanism and kinetics of food systems have already been investigated, such as oxidation by dissolved oxygen in the liquid phase and auto-oxidation catalyzed by metal ingredients [27–30].

The retention of vitamin C is summarized in Fig. 7. When kiwi slices were dried under P1 and P2 conditions, the retention values were significantly higher than those under the other conditions. These

conditions were significantly superior to P4 and P5 conditions in terms of vitamin C retention immediately after drying. After the specimens were stored for one month, the residual vitamin C was reduced in all cases. Specimens dried via P1, P2, and P5 had higher levels of vitamin C than those dried via P3 and P4. The retention after storage was almost equivalent for samples dried under P1, P2, and P5 conditions. This suggests that the application of sub-zero air temperature conditions could merit the retention of vitamin C, whereas this drying condition does not always improve storage stability. The P5 condition, which is a simple low-temperature air-drying condition, would be a good compromise in terms of total drying time and product quality. When apple slices were dried, it could be observed that condition P5 was

significantly advantageous for the retention of vitamin C compared with the other conditions. Among the AFD conditions (i.e., P1–P4 conditions), P1 was the best condition; however, the retention was approximately half that of the specimen prepared via P5. It is clear that the freezing step affects vitamin C retention.

The vitamin C content was measured for frozen specimens and freeze-thawed specimens. Frozen specimens were immediately dissolved in the test solution (i.e., metaphosphoric acid solution) and used for vitamin C content measurement. The frozen specimens were thawed in a refrigerator at 4 °C for several hours and then measured. As shown in Fig. 8, freezing did not have a significant impact on the vitamin C retention in kiwi, whereas it significantly reduced the vitamin C in apple. Freeze-thawing reduced the retention in both apple and kiwi, which could be due to leaching. These differences between kiwi and apple may be due to differences in their original cellular microstructures, and whether or not the processing route has a positive effect depends on the microstructure. From the results obtained in this study, there is no doubt that AFD conditions have positive effects on drying kinetics and product quality, and that maintaining a sub-zero temperature for a long period is beneficial. However, the advantage is not always apparent, especially when freezing has a negative effect on the resultant quality.

4. Conclusion

In this study, apple and kiwi slices were dried under AFD and LTD conditions. The drying kinetics, retention of vitamin C, and degree of shrinkage were investigated, and the following conclusions were obtained: The influence of the air temperature programs on these quality factors was investigated to clarify the merits and demerits of the AFD process. The air temperature programs clearly affected the drying kinetics of the apple and kiwi slices; as expected, shortening the sub-zero temperature time led to a shorter total drying time. When the temperature was increased stepwise from sub-zero to 10 °C, the total drying time was effectively reduced when the sub-zero temperature was maintained for a relatively long time. This was the most practical drying program when the product had to remain frozen for as long as possible to maintain product quality. It was notable that the application of freezing had a positive effect on reducing the drying time, as the application of LTD conditions without freezing resulted in a significant increase in the total drying time. It was found that drying at sub-zero temperatures had an advantage in reducing product shrinkage; however, the impact was not remarkable. For both apple and kiwi drying, it was confirmed that applying sub-zero temperature conditions was advantageous to vitamin C retention. However, it was not necessarily advantageous to improving storage stability, and it was found that sufficient storage stability could be achieved under LTD conditions. Based on the results obtained in this study, AFD conditions have a positive impact on product quality and maintaining sub-zero temperatures for extended periods of time is beneficial. However, it is notable that when freezing results in a negative impact on quality, as was observed during apple drying, a simple low-temperature drying condition could also serve as a good compromise in terms of total drying time and product quality.

Conflict of 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.

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