Comparison of freezing and convective dehydrofreezing of vegetables for reducing cell damage

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ABSTRACT

Freezing is a standard method to preserve perishable agricultural products such as fruits and vegetables, which increases off-season availability. Nevertheless, freezing of plant tissue with high water content causes cellular damage by the formation of ice crystals. This damage leads to drip loss and decreased firmness, which then reduces the quality of the thawed product. To maximize cell survival for industrial freezing processes, a promising freezing method, namely convective dehydrofreezing, was benchmarked against conventional freezing methods for different fruit and vegetables. We analyzed the final quality of thawed carrot, bell pepper, and cucumber cuts by quantifying drip loss and tissue firmness. The tissue microstructure was investigated by X-ray computed tomography after slow and fast freezing. We found that convective dehydrofreezing of bell pepper leads on average to a 52% firmer product in comparison with conventional freezing at –20 °C. For dehydrofrozen carrot, the firmness was similarly increased by 35%. Together with the significantly reduced drip loss for all tested species, these results are indicative of lower cell damage in dehydrofrozen samples. We found that dehydrofreezing of bell pepper, using different pre-drying times with resulting moisture content between 818% and 1303% dry basis, did not lead to a significant difference in drip loss or product firmness. Additionally, it was shown that freezing at an ultra-low temperature of –196 °C reduced product quality as the cucumber firmness decreased by 34% compared to conventional freezing. Freezing at low temperatures by convective freezing at –80 °C improved quality for bell pepper by producing 67% firmer products than conventional freezing.

1. Introduction

Most fruits and vegetables are highly perishable products with a short shelf life. Freezing is one of the popular techniques to preserve these crops and to increase their off-season availability. However, during the freezing process in plant cells, the volume of cellular water increases through ice formation. After thawing, the product quality in fruits and vegetables with a very high water content deteriorates by cellular damage and texture loss (Jha et al., 2019). Several factors are influencing the degree of freeze injury in fruits and vegetables. According to Wang (2016), a crucial factor leading to freeze damage is the combination of the freezing temperature and time, thus the freezing rate. Freezing methods are categorized based on their freezing rates: a) Very slow freezing (<0.1 cm h⁻¹), slow freezing (0.1–0.5 cm h⁻¹), b) normal freezing (0.5–1 cm h⁻¹), c) rapid freezing (1–10 cm h⁻¹), and d) ultra-rapid freezing (>10 cm h⁻¹) (Bogh-Sorensen, 2006; Silva et al., 2008). a) Slow and very slow freezing occurs when products are frozen by airblast freezers, or where the product is in cartons or other large packed units; b) airblast freezers are used for normal freezing; c) rapid freezing technologies are typically fluidized bed or plate freezers; d) ultra-rapid freezing as cryogenic freezing is conducted by the use of liquefied gases of nitrogen or carbon dioxide.

The freezing rate affects the change of the water state in the cellular structure. In plant cells, water is present as vacuole, cytosolic, or extracellular water. The vacuole water is essential to maintain the cellular turgor pressure and thereby, the firmness and crispiness of the product. The water in the cytoplasm interacts with proteins and solutes in the cytoskeleton and maintains, in this way, the membrane integrity (Li et al., 2018). Slow freezing typically starts in the extracellular space most likely because of the higher freezing point as fewer solutes and
water-bound molecules are present than inside the cell (Fig. 1). The ice formation reduces the amount of liquid water and increases the extracellular solute concentration, which consequently leads by osmosis to water transport outside the cell. This process leads to large extracellular ice crystals, cell dehydration, and deformation (Parreno and Torres, 2011; Zaritzy, 2011). Furthermore, cell shrinkage can cause membrane integrity loss, and finally, cell collapse. On the other hand, smaller and more uniformly distributed intra- and extracellular ice crystals are formed during fast freezing. Faster heat removal results in less cell shrinkage and deformation. However, very fast freezing at ultra-low temperatures can cause freeze cracking or cell membrane puncturing by the rapid water expansion. The former is due to radial tensile stress, as, at the freezing start, the sample surface freezes first and consequently pulls the sample center by expanding. The subsequent freezing of the inner part can then result in compressive stress and crack initiation. Nevertheless, fast freezing methods are often preferred as they generally lead to a better-preserved microstructure (Bonat Celli et al., 2016; Chassagne-Berces et al., 2009; George, 1993; Hung, 1997; Li et al., 2018; Shi et al., 1998; Silva et al., 2008; Wu et al., 2017; Zaritzy, 2011).

Additional factors influencing the freezing injury are the plant tissue’s physical properties, such as porosity of the intercellular space and the cell size (Castillo et al., 2004; Hung, 1997). Cell size is often linked to tissue porosity, whereas small cells usually are tighter packed than big cells, which are embedded in large intercellular spaces (Jha et al., 2019). Large void spaces can provoke internal stress as they affect the heat and mass transfer during freezing (Li et al., 2018). Furthermore, these air gaps can favor extracellular ice formation. Besides, the cell wall composition and rigidity, which varies among cell types, affect the freezing characteristics in different commodities (Rajashekar and Burke, 1996).

Today several new pre-treatments and freezing methods have been developed to reduce cellular damage during freezing. As a result, one can obtain a tissue that is as close as possible to the fresh product after thawing. Examples are pressure-assisted freezing or ultrasound-assisted freezing, which can enhance freezing rates and thereby reduce the freezing injuries (Cheng et al., 2017; James et al., 2015). These new technologies, however, are often accompanied by a higher energy cost, so environmental impact, or by additional infrastructure investments. In contrast to such novel and complex technologies, dehydrofreezing is a technique that has already been developed in the 1940s for selected fruits and vegetables (Howard and Campbell, 1946). Whereas recently, a convective dehydrofreezing technique was defined and used (Ben Haj Said et al., 2015a). For dehydrofreezing, the products are pretreated by mild dehydration, which is done either through air-drying (herein referred to as convective dehydrofreezing) or by the addition of an osmotic-active solute (Osmo-dehydrofreezing). The reason for this drying step is to reduce the high amount of cellular water, which can be critical in vegetable tissue. Due to the partial removal of water, the plant cells are under less turgor pressure and thus more relaxed. Therefore, the cells can withstand the volume increase during freezing, and the damage of cell walls can be decreased, especially during the fast freezing of partially dried cells. Furthermore, drying before slow freezing can reduce the volume of extracellular ice.

In comparison to convective dehydrofreezing, osmo-dehydrofreezing is less energy-intensive. Nevertheless, this method has the disadvantage that it can lead to changes in nutritional and sensory characteristics of the products, usually due to the use of sugar or salt solutions. Several studies about osmotic dehydrofreezing of various fruits and vegetables are present (James et al., 2014; Li and Sun, 2002; Ramya and Jain, 2017). On the contrary, for convective dehydrofreezing (and/or osmo-convective dehydrofreezing), only a few studies are present on hot air pre-drying of fruits such as strawberry, pineapple, melon, kiwi, apple, and quince (Ben Haj Said et al., 2015a, 2015b, 2016; Hajji et al, 2019, 2020; Maestrelli et al., 2001; Moraga et al., 2006; Ramallo and

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**Fig. 1.** Schematic overview of possible freeze processes in plant cells during the tested freezing methods.
However, thermal drying at high temperatures can be disruptive to the cell membrane (Ando et al., 2016) or alter aroma compounds (Maestrelli et al., 2001). By Ben Haj Said et al., 2016; Hajji et al., 2019, it was confirmed that partial pre-drying at medium temperatures (40–45 °C) together with instant controlled pressure drop treatment allows reducing the freezing time and improving the final product texture. Today, only less is known about the impact of convective dehydrofreezing by cold air (Anmella, 2010). Furthermore, a detailed analysis of the product quality (e.g., texture and microstructure) for freezing of very freeze susceptible vegetable are scarce.

In the current study, we aim to benchmark convective dehydrofreezing against conventional freezing methods for three different species, namely carrot, sweet bell pepper, and cucumber. To this end, we analyzed the microstructure and food quality of the thawed samples, including by X-ray micro-computer tomography (micro-CT). We tested if the quality of the thawed samples can be improved by convective dehydrofreezing, compared to conventional freezing. For bell pepper, this hypothesis was further investigated by a sensory panel test.

2. Materials and methods

2.1. Sample preparation

Carrot (cv. not known), sweet red bell pepper (cv. California Wonder), and cucumber (cv. not known) were purchased at a local supermarket. All vegetables were stored at 4 °C and were washed before the experiments. In Table 1, the sample sizes are listed. For each commodity and freezing method, 30 replicates with uniform cylindrical shape (diameter = 13 mm) were prepared. Therefore, the carrots were peeled, and 7 mm thick pieces were made by a kitchen slicer. Carrot samples, including both cortex and vascular tissue, were cut out using a cork borer. For cucumber, the endocarp tissue and seeds were removed, and samples were cut out from the remaining meso- and exocarp tissue (total thickness = 10 mm). Bell pepper seeds and fruit placenta were entirely removed, and only the pericarp tissue was cut into cylindrical samples. For bell pepper and cucumber, samples of five fruits each were randomized and tested by all six freezing treatments. Since fewer cuts could be produced from a single carrot, samples from three to four carrots were randomized over two to three freezing methods.

2.2. Pre-treatments and freezing methods

In Fig. 2, the general workflow for all tested freezing methods is illustrated. Three different freezing temperatures (−20 °C, −80 °C, −196 °C) were tested. Additionally, it was investigated whether pre-treatments such as precooling prior to freezing close to the freezing point or drying (convective dehydrofreezing) give rise to similar or even better quality as by conventional freezing at −20 °C. Therefore, two climate test chambers were initially preconditioned to 0 °C and 70% RH before they were frozen and stored at −20 °C (RUMED 3401, 350l; RUMED 3401, 530 l). Additionally, a chest freezer was preconditioned for convective freezing at 80 °C (Brower, Skadi DF9020GL). After sample cutting, the fresh-cut weight (w_fresh, [g]) was determined. To avoid moisture loss, we wrapped the samples into a paraffin film (Parafilm M, Bemis, Neenah, United States). After the freezing process, all samples were stored overnight at −20 °C until the measurements were performed (except for min80, see below).

The following six different freezing methods were employed:

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Table 1

| Test sample dimensions. |
|-------------------------|
| thickness [mm] | surface area [mm²] | volume [mm³] |
| Carrot | 7 ± 1 | 551 ± 41 | 929 ± 133 |
| Bell pepper | 6 ± 1 | 511 ± 41 | 796 ± 133 |
| Cucumber | 10 ± 1 | 674 ± 41 | 1327 ± 133 |
2.3. Estimation of the freezing rates

Freezing rates were recorded for each commodity during conventional freezing with and without the precooling step at 0 °C. For bell pepper, the freezing rates were additionally measured during dehydrofreezing. Convective drying was performed for 3 h at 0 °C with 5% loss of fresh weight. For each method, one sample was tested using cuts from the same vegetable. In Fig. 3, the measurement setup is illustrated. Therefore, one NTC probe of an Ecolog temperature data logger was inserted in the geometric sample center. A second probe was installed to record the air temperature (Ecolog TN2 with 2 NTC probes, Elpro-Buchs AG; Temp. range: −50 °C to −140 °C, ±0.2 °C). Simultaneously, a second Ecolog temperature data logger was installed to measure the sample surface and air temperature of the same sample. For a second biological replicate, which was wrapped with paraffin, the temperature-time profile was recorded to test the effect of the paraffin foil on the freezing rate. These freezing curves and freezing rates are attached in the Supplementary Material. The temperature data were analyzed using the elproLOG ANALYZE software (V23.64.01). The temporal \( r_t \) [°C min\(^{-1}\)], Eq. (1), and spatial \( r_s \) [cm h\(^{-1}\)], Eq. (2) freezing rates were calculated as described by Bult et al. (2018). The time elapsed from the initial temperature (ambient or precooling temperature) to the set freezing air temperature is herein referred to as the total freezing time \( t_{t} \) [min]). The spatial freezing rate was determined as the minimal distance between the surface and thermal center \( d \) [cm] over the time elapsed between a surface reaching a temperature of 0 °C and the thermal center reaching a temperature of −15 °C \( (t_f \) [min]).

\[
t_{\text{temporal freezing rate}} = \frac{T_{\text{min}} - T_{\text{freez}}} {t_f} \quad (1)
\]

\[
t_{\text{spatial freezing rate}} = \frac{d} {t_f} \quad (2)
\]

2.4. Drip loss and moisture content

For each commodity, moisture content (%), wet (wb) (Eq. (3)), and dry basis (db) (Eq. (4)) just before freezing were evaluated for 30 replicates. For convective dehydrofrozen samples, the water loss (%) after the pre-drying was additionally evaluated (Eq. (5)). Drip loss (%) (Eq. (6)) after freezing was determined relative to the initial moisture content of the fresh or dehydrated sample.

\[
\text{Moisture content wb} = \frac{(W_{\text{fresh or dehydrated}} - W_{\text{dried}})}{W_{\text{fresh or dehydrated}}} \times 100 \quad (3)
\]

\[
\text{Moisture content db} = \frac{(W_{\text{fresh or dehydrated}} - W_{\text{dried}})}{W_{\text{dried}}} \times 100 \quad (4)
\]

\[
\text{Water loss after drying} = \frac{(W_{\text{fresh or dehydrated}} - W_{\text{dried}})}{W_{\text{fresh or dehydrated}}} \times 100 \quad (5)
\]

\[
\text{Drip loss} = \frac{W_{\text{fresh or dehydrated}} - W_{\text{dried}}}{W_{\text{fresh or dehydrated}}} \times 100 \quad (6)
\]

\[
\text{Dry matter content} = 1 - \frac{W_{\text{dried}}}{W_{\text{fresh or dehydrated}}} \times 100 \quad (7)
\]

After fresh weight \( (w_{\text{fresh}} [g]) \) was determined, the samples were either precooled and/or convective dried and frozen or were directly frozen by one of the different freezing methods (Fig. 2). For convective dehydrofreezing, sample weight was determined after the drying process \( (W_{\text{dehyd}} [g]) \). To measure the sample drip loss, we weighted frozen cuts \( (W_{\text{frozen}} [g]) \) and thawed them for about 2 h at room temperature until the center reached 20 °C. The weights of the fully thawed samples \( (W_{\text{thawed}} [g]) \) were determined after paraffin, and the excessive free water was soaked up by a tissue paper. The dry weight \( (w_{\text{dried}} [g]) \) was measured after the samples were oven-dried overnight for about 15 h at 80 °C to determine the dry matter content (Eq. (7)).

2.5. Firmness

The tissue firmness of fully thawed samples was evaluated using a ‘TA-XT2i Texture Analyzer’ (Stable Micro Systems ‘TA-XT2i, UK). A puncture test was performed using a 3 mm stainless steel cylinder probe (P/3) with the following parameters: 1 mm s\(^{-1}\) speed, 2 mm penetration depth, and a trigger force of 10 g. The firmness of the cucumber mesocarp and the bell pepper pericarp was determined by the mean of four measurements at different positions of the same sample. For carrot samples, the mean firmness of the phloem tissue was evaluated from triplicates.

2.6. X-ray micro-CT and image analysis

X-ray micro-CT was performed to capture the microstructural changes after the thawing of tested species. Therefore, cylindrical samples with a 2 mm diameter and a height of 10 mm were prepared for each vegetable (carrot cortex/phloem, cucumber mesocarp, bell pepper pericarp). The samples were wrapped in paraffin to avoid moisture loss. Six fresh samples had been scanned and subsequently were divided into two sets. Each set contained one sample of each vegetable. The first set of samples underwent the conventional freezing process, where the samples were directly frozen to the final temperature of −20 °C. The second set of samples were frozen using liquid nitrogen. After freezing, all samples were stored for 2 h at −20 °C, and subsequently, they were fully thawed at 20 °C and then again scanned. Additional X-ray tomographs were taken from freeze-dried carrot samples that were frozen either by conventional freezing or by cryogenic freezing and afterward dried. For this, the frozen samples were directly placed in a freeze dryer (Christ Alpha 1–4 LCS plus). This process was performed under constant low pressure of 63 Pa for 18 h with a condenser temperature of −85 °C and a chamber temperature of 20 °C.

The X-ray imaging experiments were done in the Center for X-ray Analytics of Empa (Switzerland). The X-ray system being used was the EasyTom-XL system from Rx Solutions (Rx Solutions SAS, Chavanod, France) with a power source of 20 W at 70 kV and 285 μA. The sample was rotated over 360° by angular steps of 0.5°. As such, 721 images were generated for one tomogram dataset. Each image has 1800 \( \times \) 1800 pixels with a spatial resolution of approximately 2.3 μm. One
tomographic scan lasted for 10 min, and the tomograms were reconstructed with a phase-contrast algorithm implemented in the XAct software by RX Solutions. The Avizo software (Thermo Fisher ScientificTM) was utilized to perform image processing. A segmentation algorithm was used to separate the cell and pore domains in the tissue (Prawiranto et al., 2019). From the volume of cell and pore domains, the porosity of the samples was calculated. For the freeze-dried carrot scans, the volumes and equivalent diameter of the plant tissue’s remaining voids were calculated for conventionally and cryogenically frozen samples to approximate the space previously occupied by ice crystals. Therefore, the segmentation algorithm was used to separate and specify the volume of single void structures (Prawiranto et al., 2019). Extra- and intracellular structures were thereby not differentiated. As the physical size of the cryogenically frozen sample was higher than the conventional frozen sample, we divided the former into three fractions that have a similar size as the conventional frozen sample. For the comparison of the distribution of the pore size between those samples, we compared each fraction of the cryogenically frozen sample to the conventionally frozen one. As the results of each comparison led to a similar result, only one comparison is shown below.

2.7. Sensory analysis

We investigated if a noticeable difference in the sensory level exists between samples frozen with conventional freezing and convective dehydrofreezing. To this end, a triangle discrimination test was performed for bell pepper samples, which had been subjected to the drying time of 3 h associated with 5% weight loss. A sensory panel of 16 panelists was used. Each panelist received three fully thawed bell pepper samples, which were prepared as described in section 2.2. Two of these samples were frozen using the same freezing method. We evaluated if the odd sample, which was frozen by the second freezing method, could be successfully discriminated by the panelist. The sample order was randomized over all the panelists.

2.8. Statistical analysis

Statistical analyses were performed using the software “R and RStudio” (version 1.1.463) (R Core Team, 2018), and graphical plots were created with ggplot2 (Wickham, 2016). The variance of drip loss and initial moisture content of different freezing methods were analyzed by one-way ANOVA with a confidence interval of 95%. The variance between the mean firmness values for different freezing techniques was tested by the non-parametric Kruskal Wallis test. The significance between the mean moisture content, drip loss, and firmness of each treatment was evaluated by post-hoc analysis Dunnett’s test to compare with the reference method (conventional freezing). By multiple comparisons of the mean using the Tukey’s-test, difference for drip loss values among all freezing techniques were analyzed; these results are attached in the Supplementary Material. Additionally, significances of the mean firmness values among all groups determined by post-hoc Dunn’s-test with adjusted p-values by the Benjamini-Hochberg correction that is included in the Supplementary Material. To evaluate the possible relationship between moisture content db and drip loss, we calculated Kendall’s rank correlation coefficient for each species. Correct answers resulting from the sensory triangle test were tested by a one-tailed binomial test with a significance level of α = 0.05.

3. Results

3.1. Freezing rates

In order to assess the freezing process at −20 °C, freezing rates were measured for conventional freezing with and without precooling. Additionally, the freezing curve was obtained for dehydrofrozen bell pepper after convective drying with 5% weight loss. The moisture content of the single sample was not determined; for comparison, the mean moisture contents measured for each vegetable from subsequent tests are listed below. In Fig. 4, the freezing curves are shown for each of the tested commodities. The pre-freezing stage, where the heat is being removed until the freezing point is reached, varies between the different species due to differences in moisture content and composition (Silva et al., 2008). As cucumber has a higher moisture content (96% wb), the pre-freezing stage was longer than for carrot and bell pepper (88% and 90% wb) (Fig. 4c). The pre-freezing step is not present for precooling and dehydrofreezing methods, as, during the pre-treatments, the temperature was not recorded. A typical plateau is visible in each freezing curve representing the phase change from liquid water to ice. Compared to cucumber, the plateau’s slope, which depends on the amount of water and solute concentration (Zaritzky, 2011), was steeper in carrot and bell pepper. Therefore, we assume that the ice crystal formation occurred faster in carrot and bell pepper than in cucumber. After the crystallization process, the sample temperature decreased to the final temperature of −20 °C. For all vegetables, the spatial freezing rates were in the same range of 1–1.5 cm h⁻¹. In contrast, small variations in freezing rates are related to different moisture content and dimension of the vegetable cuts (Table 1). We assume that the amount of water to be frozen in each sample was similar for freezing with and without precooling, which could explain why the corresponding freezing rates are in the same range. After convective drying with a weight reduction of 5%, we did not observe a higher freezing rate when comparing with conventional freezing. However, it should be noted that only one sample for each freezing method was evaluated and that sample biological variability (e.g., for moisture content) could affect the freezing rate. In conclusion, the tested methods are comparable to freezing protocols for “normal” freezing rates (0.5–1 cm h⁻¹) such as air blast or plate freezers (Parreño and Torres, 2011; Silva et al., 2008).

3.2. Change in the microstructure

As the freezing process affects the sample porosity by cellular damage and drip loss, we compared the relative volume of intercellular airspaces (i.e., porosity) of fresh and thawed samples after conventional and cryogenic freezing. Fig. 5 shows the cross-section of each sample before freezing and after thawing obtained by X-ray micro-CT. It can be seen that after thawing, the samples deformed, and most of the pores disappeared or were filled with liquid. Ring artifacts of the x-ray scans, which altered the grey value of the cell tissue only, can be observed. However, this did not affect the segmentation to separate cells and airspaces since a high contrast was maintained between these two domains. The changes in the porosity of each sample are tabulated in Table 2. In cucumber and bell pepper, almost all pores vanished with a porosity reduction of 96–99%, for both conventional and cryogenic freezing methods. In contrast, the reduction of porosity was less in the carrot samples, namely in the range of 64–69%. We assumed that most of the pores were filled by meltwater after thawing, deriving from extracellular ice after slow freezing or intracellular water through cell membrane puncturing when fast freezing (Fig. 1). Carrot seems to have more resistance to freeze damage compared to cucumber and bell pepper. The lower moisture content in carrots (88% wb) compared with cucumber (96% wb) and bell pepper (90% wb) could explain this observation. Furthermore, the carrot root tissue has a different composition of cell types, namely cortical, endodermal, and vascular cells. The latter two often present thickened cell walls (Geldner, 2013), making them more rigid and possibly more resistant against freezing injury by deformation (Ye, 2002). The cryogenic method gave lower porosity changes in all species. However, the differences were relatively small, namely in the range of 1–5%. We assume that slow freezing, usually leading to large extracellular ice crystals, causes more melting water and pore reduction than cryogenic freezing in all tested species.

The carrot samples were further investigated by looking deeper into the impact of the freezing methods on the microstructure. The reason is
that the most significant change in terms of the porosity between the two freezing techniques was observed in those samples (Table 2). Freeze-dried carrot cylinders were analyzed, and the void volume of the remaining cellular structures was evaluated for conventional and cryogenic freezing methods (Fig. 6). As both carrot samples differed in size, a 3D image fraction of the cryogenically frozen sample with the same size as the conventionally frozen one was compared. Through freeze-drying, ice crystals sublimated and left behind a fibrous cellular structure. The resulting void spaces in the freeze-dried samples correspond to the initial space previously occupied by ice. We quantified the void volumes to approximate the ice crystal size before the samples were freeze-dried, whereas we did not distinguish between intra- and extracellular structures. Nevertheless, it has to be noted that the freeze-drying process can also alter the microstructure by shrinkage or deformation (Voda et al.,

Fig. 4. Freezing curves recorded at the sample center and the sample surface for carrot (a, b), cucumber (c, d), and bell pepper (e, f, g) during conventional freezing (-20 °C) with (b, d, f) and without (a, c, e) precooling at 0 °C. For bell pepper, the temperature-time profile was additionally measured during freezing after convective drying at 0 °C (g).

Fig. 5. Cross-sections of the scanned carrot (a), cucumber (b), and bell pepper (c) samples before conventionally (min20) and cryogenically (LN) freezing and after thawing.
The porosity of different vegetables before freezing (fresh) and after thawing.

| Method | Fresh (%) | Thawed (%) | Change (%) |
|--------|-----------|------------|------------|
| Carrot | min20     | 3.11       | 0.97       | -68.78     |
|        | LN        | 1.97       | 0.79       | -64.21     |
| Cucumber| min20    | 1.85       | 0.03       | -98.53     |
|        | LN        | 1.46       | 0.05       | -96.39     |
| Bell pepper | Min20 | 0.95       | 0.00       | -99.81     |
|        | LN        | 1.08       | 0.01       | -99.06     |

### 3.3. Quality

#### 3.3.1. Carrot

In Fig. 7, the measurement of the quality parameters such as moisture content before freezing, as well as total moisture loss, drip loss, and firmness after thawing, are summarized. After conventional freezing of carrot samples, the drip loss was, on average, 18.6% (Fig. 7g). Compared to conventional freezing, all freezing techniques, except for convective dehydrofreezing, showed a reduction in drip loss in the same range of 2–4%. Carrot samples frozen by convective dehydrofreezing resulted in a high total moisture loss of 33.9%, namely 26.5% during pre-drying but only 7.4% during the freezing step (drip loss) (Fig. 7d). Thus, the average drip loss that is assumed to occur due to tissue damage was significantly reduced in dehydrofrozen carrot samples compared with conventional frozen ones (18.6%, p < 0.001). These results indicate that pre-drying with a final moisture content of 51% db leads to reduced cell damage, either by less shrinkage or less membrane leakage. Freezing with versus without precocing prior to conventional freezing led to a small but significant reduction of the mean drip loss (16.1%, p < 0.05). It implies that precocing can slightly improve the freezing process and, thus, the cell survival rate, but not by much. After freezing at -80 °C, the mean drip loss was significantly reduced (14.7%, p < 0.001) compared with the average drip loss after conventional freezing. However, it has to be noted that the mean moisture content of these samples (708% db) was also significantly lower compared with conventional frozen samples (883% db, p < 0.001) (Fig. 7a). In comparison to conventional freezing, cryogenic freezing showed a similar reduction in drip loss (16.6%, p > 0.05). These results are in contrast to our previous findings that fast freeze-drying method utilized here could also be a reason for this change in the microstructure. Thereby the small sizes of ice clusters that had formed during cryogenic freezing might have collapsed together during lyophilization. Thus, these ruptures subsequently could have led to an overestimation of the approximate ice crystal size in the cryogenically frozen sample. However, another explanation for smaller ice crystals in slowly-frozen carrot could be the formation of extracellular ice induced by osmosis and water transport from the cytosol to the extracellular space. This process usually leads to cell shrinkage and, therefore, could lead to less intracellular ice. In the fast-frozen samples, the nucleation sites are typically more uniformly distributed outside and inside the cell, which prevents a higher degree of cell shrinkage. Hence, further chemical analyses to elucidate the origin of the thaw exudate water (intra-/extracellular) of different frozen samples would be beneficial.

**Fig. 6.** XY-cross section of the freeze-dried carrots frozen by conventional (a) and cryogenic (c) freezing. Red arrows indicate freeze-cracks and cell collapse. X-Z-cross section, void labeling, and 3D rendering of the voids are shown for conventional (b) and cryogenic (d) freezing. The distribution of the void volume, representing the ice crystal size, is displayed for conventional (red) and cryogenic (yellow) freezing for two size sections (e,f). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)
freezing leads to pronounced microstructural alterations, which are presumably associated with enhanced drip loss (Fig. 6). Nevertheless, it has to be considered that the initial moisture content before freezing was significantly lower in cryogenically (800% db) frozen samples compared to conventional frozen ones (883% db, \( p < 0.05 \)). Precooling plus cryogenic freezing lead to a small but statistically significant reduction in drip loss (16.2%, \( p < 0.05 \)). In conclusion, in each tested method, the mean drip loss was lower compared to conventional freezing. The best results could be obtained for dehydrofreezing and convective freezing at 80°C, relative to conventional freezing by a reduction of 60% and 21%, respectively. Furthermore, we observed that apart from the freezing method, the initial moisture content before freezing can be linked to the drip loss of thawed carrot samples.

Compared with the firmness of fresh carrot cuts, the mean firmness of thawed carrots sharply decreased with all freezing methods, namely, on average, by 91% (Fig. 7j). However, in contrast to conventional freezing, sample firmness retention was significantly better for all freezing techniques \( (p < 0.001) \). On average, those samples’ mean firmness was 0.4–1.2 N higher than for carrots obtained from conventional freezing (1.1 N). The highest firmness was recorded for samples frozen for precooling before freezing, ‘d_min20’ convective dehydrofreezing, ‘min80’ convective freezing at 80°C, ‘LN’ cryogenic freezing. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

**Fig. 7.** The average (±SD) sample moisture content directly before freezing of carrot (a), cucumber (b), and bell pepper (c) \( (n = 30) \). The average percentage of moisture loss after thawing of carrot (d), cucumber (e), and bell pepper (f) \( (n = 30) \). The moisture loss after freezing (drip loss) is indicated in dark-blue, whereas the moisture loss after the pre-drying step is shown in pale-blue. Boxplots of the percentage of drip loss after thawing of carrot (g), cucumber (h), and bell pepper (i). Boxplots of measured firmness for carrot (j), cucumber (k), and bell pepper (l) presented for all tested freezing methods \( (n = 30) \). Significant differences were tested against the control method as conventional freezing (min20) and are indicated. Abbr.: *** \( p < 0.001 \), ** \( p < 0.01 \), * \( p < 0.05 \), ‘min20’ conventional freezing, ‘p_’ precooling before freezing, ‘d_min20’ convective dehydrofreezing, ‘min80’ convective freezing at 80°C, ‘LN’ cryogenic freezing.
moisture content db of the tested carrot samples was 779%. The drip loss of carrot samples was significantly correlated with the sample moisture content ($\tau = 0.55$, $p < 0.001$) (Table 3). These findings are in line with previous studies that have shown this relationship for apple or quince (Ben Haj Said et al., 2016; Hajji et al., 2020). Hence, we conclude that the product moisture content could be a potential factor to estimate the degree of drip loss, independent of the freezing method used. This result described here for carrot is done similarly for cucumber and bell pepper below.

3.3.2. Cucumber

Conventional freezing of cucumber resulted on average in 14.0% drip loss, whereas previous precooling induced a slight but non-significant reduction of 1.5% (Fig. 7h). In comparison to conventional freezing, cryogenic freezing induced no reduction in the drip loss. Freezing by convective dehydrationfreezing, lead to a mean total moisture loss of 23.5% (Fig. 7e). It has to be considered that after drying, about 14.3% of the moisture was evaporated. Consequently, the average drip loss of those samples (9.2%) was significantly lower compared with the mean drip loss of conventional frozen cucumber cuts (14.0%, $p > 0.001$). Thus, we presume that by decreasing the total water content in dehydrated cucumber, cellular damage and leakage could be reduced. On the other hand, convective freezing at $-80^\circ$C induced a significant increase in the mean drip loss (17.8%, $p < 0.001$) in comparison with conventional freezing. Therefore, it seems that freezing at higher temperatures is more beneficial for cucumber. We assume that particularly in cucumber with very high water content, freezing at ultra-low temperature is likely to cause freeze-cracks at cellular and macroscopic levels leading to enhanced drip loss.

The firmness of thawed cucumber samples was, on average, 68% lower than the mean firmness of fresh samples (Fig. 7k). Interestingly, in contrast to conventional freezing, all tested methods lead to samples with comparable firmness or even softer samples. Compared with conventional freezing (2.4 N), precooling plus conventional freezing (1.9 N, $p < 0.01$), and both cryogenic freezing methods (1.6 N, $p < 0.001$), lead to significantly lower firmness. In regard to the initial moisture content, only in dehydrated sample (2000% db) a significant difference was measured compared with conventional freezing (2403% db, $p < 0.001$) (Fig. 7b). However, even though the measured drip loss was reduced, we did not observe a significant change in product firmness in those samples. To this end, similar analyses on the texture of thawed cucumber cuts have not been described yet. Nevertheless, previous studies on apple had shown that the dry matter content is positively - so water content is negatively correlated with the sample firmness (Ben Haj Said et al., 2015a, 2015b, 2016). However, for samples with high moisture content (>2000% db), the freezing process sharply reduced samples firmness (Ben Haj Said et al., 2015a). Together with our observation, these findings could indicate that for cucumber with very high moisture content (>2000% db), the freezing method rather than the pre-freezing water content could be an important factor influencing product texture. We conclude that cucumber is very susceptible to freezing by any of the tested methods; whereas, we found that freezing at ultra-low temperatures (-196 °C) had the most prejudicial effects on product firmness.

Cucumber samples had, on average, a moisture content of 2306 db %, whereas the correlation coefficient between water content and drip loss resulted in $\tau = 0.37$ ($p < 0.001$) (Fig. 8, Table 3). Even though the average moisture content in cucumber is rather high, we can still observe this relationship. Our observation supports the hypothesis that samples with a high water content, and therefore low dry matter content, likely are more prone to exhibit an increased drip loss.

3.3.3. Bell pepper

In bell pepper samples, a mean drip loss of 22.4% was measured after conventional freezing (Fig. 7i). All other methods, except dehydrofreezing, lead to an equivalent drip loss in the range of 22–24%. The total moisture loss after convective dehydrofreezing was, on average, much higher (34.3%) due to the amount of water evaporated during the drying step (15.7%) (Fig. 7f). Consequently, the mean drip loss after the freezing step was significantly lower (18.6%, $p < 0.001$) in comparison with the mean drip loss after conventional freezing (22.4%). Thus, only convective dehydrofreezing leads to a markedly lower drip loss among the tested freezing methods.

Although there were no strong discrepancies between all freezing methods for bell pepper drip loss, clear trends among the groups we observed for bell pepper firmness (Fig. 7f). The mean firmness reduction after freezing was in the range of 52%. In contrast with the mean firmness of conventional frozen samples (2.9 N), convective dehydrofreezing (4.5 N, $p < 0.001$) as well as freezing at $-80^\circ$C (4.9 N, $p < 0.001$) revealed on average significantly higher firmness values. Similar to carrot, these results could indicate a reduction in cell damaged for convective freezing at $-80^\circ$C and dehydrofreezing. Although in carrots, the drip loss reduction in $-80^\circ$C frozen samples possibly was induced by the lower initial moisture content rather than the freezing method. Precooling before conventional freezing had a positive but non-significant effect on the mean sample firmness (3.4 N, $p > 0.05$). Interestingly, next to conventional freezing, both cryogenic freezing methods, with (3.0 N) and without precooling (2.9 N), produced, on average, the softest samples. Similar to cucumber, we found that freezing at very high freezing rates ($-196^\circ$C) is destructive to bell pepper cells. The high initial moisture content (>900% db) could explain these observations, as, during fast freezing, very rapid volume expansion causes tensile stress, possibly leading to cell damage and freeze-cracks. In conclusion, convective dehydrofreezing as well as freezing at $-80^\circ$C retained the bell pepper firmness the best.

The moisture content of tested bell pepper samples was, on average, 956% db. Similar to the other tested vegetables, there was a significant correlation between the moisture content and drip loss ($\tau = 0.37$, $p < 0.001$) (Fig. 8, Table 3).

3.4. Convective dehydrofreezing of bell pepper for different drying periods

We further investigated convective dehydrofreezing on bell pepper samples, where different lengths of mild drying periods were tested before freezing (3, 4, 8 h). After this pre-drying step and before freezing, samples were sealed from the environment to avoid further dehydration and kept at a low temperature for 12 h. This relaxation period aimed to

Table 3

|         | $\tau$ | Probability   |
|---------|--------|--------------|
| Carrot  | 0.55   | <2.2E-16     | ***        |
| Cucumber| 0.37   | 2.99E-13     | ***        |
| Bell pepper | 0.37 | 3.47E-13 | *** |

***p < 0.001.
redistribute the water within the sample in order to even out gradients in moisture content. The results are presented in Fig. 9, where the measurements on convective dehydrofrozen bell pepper from the first set of experiments (15 h, fresh, see 3.3.3) are also included. After pre-drying periods of 3, 4, and 8 h, the mean moisture content db was in the same range of 1292%, 1303%, and 1259%, respectively (p > 0.05) (Fig. 9a). Even though, when compared with 15 h of dehydration, a considerable difference in water content can be observed (818% db). Fig. 9b displays that the average total moisture loss (including dehydration and drip loss) tended to increase with the drying time. The average moisture loss during dehydration amounted to 5.4%, 5.6%, 8.5%, and 15.7% for 3, 4, 8, and 15 h of convective drying, respectively. The percentage of the mean drip loss for each group followed a similar trend with 15.1%, 17.1%, 14.8%, and 18.6% for 3, 4, 8, and 15 h, respectively (Fig. 9c). It can be seen that a slight increase in initial moisture content for samples dehydrated for 4 h can be linked with a slight increase in drip loss. However, when considering 15 h of pre-drying, we do not observe that a lower initial moisture content leads to less drip loss. Regarding the bell pepper texture, no significant differences in the firmness were detected among samples with different dehydration time (p > 0.1) (Fig. 9d). Hence, we assume that a drying time longer than 3 h does not reduce bell pepper drip loss further or retain firmness noticeably better.

In a subsequent triangle discrimination test, we investigated whether consumers can notice a difference between thawed bell pepper samples, which were either conventionally frozen or by convective dehydro-freezing. The results revealed no significant differences between the two products (p > 0.05). Note that a relatively small group of panelists was available. However, this outcome indicates that although a significant difference in firmness and drip loss can be measured, sensory aspects as texture and taste are not noticeably affected by convective dehydro-freezing. As such, this technology needs to be optimized further also to achieve these sensory benefits.

4. Discussion

4.1. Precooling before conventional freezing

By precooling the sample, smaller temperature gradients are present within the product, and consequently, less heat needs to be removed during freezing. The freezing rates of precooled samples were in the same range as those of conventional frozen samples as the amount of water to be frozen possibly did not differ considerably. However, it must be noted that only one replicate was measured for each method. We observed that precooling before conventional freezing always led to a lower drip loss for the three tested species compared to conventional freezing. This lower drip loss might imply that precooling improves the freezing process and, therefore, reduces freezing injuries. However, only in carrots, the mean drip loss after precooling was statistically significantly reduced compared to conventional freezing, namely by 13%. The measured firmness of those samples was 2.1-fold higher than the firmness of conventional frozen carrots. In contrast, conventional freezing with precooling had no significant effect on the firmness of bell pepper. The firmness of cucumber was even significantly lower than with the sample without precooling. It seems that in precooled carrot, the ice crystal formation caused less damage on the cell wall and cell membrane than in precooled cucumbers and bell pepper, indicated by better firmness retention. The lower moisture content and the variation of cell types present in the carrot tissue compared to cucumber and bell pepper could explain these results.

4.2. Freezing at ultra-low temperatures

Convective freezing at −80 °C led to different outcomes, depending on the tested commodities. For carrot, the quality was enhanced, on average, by 21% less drip loss and 56% higher firmness than in conventional frozen samples. Whereas the initial water content was also found to be a key factor influencing the product quality of those samples. For bell pepper, convective freezing at −80 °C enhanced the quality, as those samples had, on average, 67% higher firmness values than conventional frozen ones. However, for cucumber, product quality decreased, as indicated by 7% lower firmness and 28% higher drip loss compared to conventional freezing.

Rapid freezing of carrots by liquid nitrogen did show a positive impact on firmness retention. Nevertheless, for bell pepper and cucumber, which have a high water content (≥900% db), cryogenic freezing mostly reduced sample quality. It was shown before that freezing with liquid nitrogen can critically affect the cellular structure by freeze-cracks and reduced product firmness, and that such effects occurred less when freezing at −80 °C (Chassagne-Bercès et al, 2009, 2010). Particularly when freezing products with high water content at ultra-low temperatures, adverse forces like tensile stress become critical (Shi et al., 1998). The X-ray micro-CT results revealed that the porosity was reduced to a lesser extent in cryogenically frozen samples compared to conventionally frozen ones. However, this difference in the porosity of thawed samples of 1–5% was rather small. We measured the void volume in freeze-dried carrots to approximate the cellular (intra- and extracelluar) ice. In contrast to previous studies, we found that the estimated ice crystals of freeze-dried cryogenic frozen carrots were, on average, larger than in the conventional frozen ones (Vicent et al., 2019). Nevertheless, the large void spaces could be provoked by the lyophilization step or the rapid volume expansion during the freezing process. An explanation of the observation of smaller ice crystals in conventional frozen carrot could be the occurrence of cell shrinkage, which is induced by osmosis during slow freezing.

Precooling before cryogenic freezing had no significant effect on product quality in comparison to cryogenic freezing. When precooling, the temperature reduction to 0 °C is relatively small compared to the final freezing temperature of −196 °C. Hence, we assume that only a small decrease in mechanical stresses will be induced.
We conclude that fast freezing of cucumber and bell pepper at −80 °C is preferred over cryogenic freezing, as tissue firmness was better retained. However, we assume that rapid freezing at ultra-low temperatures leads to fast water-ice conversion with large temperature gradients. Due to the little or no expansion possibilities in this short time frame, severe internal stresses can occur, which could induce macroscopic freeze cracks and microscopic cell damage. Especially, cucumber with high water content seems to be very susceptible to such damage.

4.3. Convective dehydrofreezing

When testing convective dehydrofreezing, a significant amount of moisture had been lost during the pre-drying step in all tested samples. Nevertheless, by this dehydration, the drip loss of thawed samples could thereby be significantly decreased, which was also shown in previous studies for selected fruits (Ben Haj Said et al., 2016; Hajji et al., 2020; Maestrelli et al., 2001; Ramallo and Mascheroni, 2010). As drip loss can be linked to the amount of cellular damage, we assume that in total more cells survived in dehydrofrozen samples. A further explanation for the reduced drip loss could be the occurrence of less extracellular ice, which during thawing usually leaks out of the tissue rather than flowing back into the cells.

Concerning firmness, the quality was improved in dehydrofrozen carrot and bell pepper compared to conventional freezing as it increased by 35% and 52% after dehydration with moisture loss of 27% and 16%, respectively. Thus, we conclude that drying before freezing can positively affect the product’s texture even though its turgidity gets reduced. However, for dehydrofrozen cucumber, with high overall moisture content, we measured 14% of moisture loss but no pronounced firmness retention. In line with what was stated by James et al., 2014, we conclude that the optimal pre-freezing moisture content and related quality enhancement differ among species due to the variation of their freeze resistance.

We further investigated the impact of different drying timeframes (3, 4, and 8 h) on sample quality for bell pepper samples. It was found that the resulting moisture content and related quality parameters, such as drip loss and firmness, did not significantly differ for different drying durations. However, compared with 15 h of drying, our results indicated that shorter drying periods did not reduce product quality, despite the higher total water amount. Therefore, we suggest using similar or even smaller drying periods to shorten the production time. In subsequent studies, the appropriate drying duration, as well as the corresponding operating temperatures, should be investigated and optimized. The aim here would be to optimally maintain firmness, decrease drip loss, and reduce processing time. However, from this pilot study, we found that dehydrofreezing is a promising freezing method to enhance bell pepper quality by inducing less drip loss and a higher firmness in comparison to conventional freezing.

5. Conclusion

In the present study, we found that convective dehydrofreezing is a promising alternative to the conventional freezing method. Compared to conventional freezing, dehydrofreezing led to a significantly higher product firmness in carrot and bell pepper, namely by 35% and 52%, respectively. Furthermore, we found that the drip loss was significantly reduced by convective dehydrofreezing for all three species. Additional precooking prior to conventional freezing was proven to increase product quality only in carrots, namely by a 13% reduction in drip loss and a 2.1-fold higher firmness. Cryogenic freezing versus conventional freezing led in carrots to product improvement in terms of drip loss reduction and higher firmness retention. However, freezing at ultra-low temperatures favored an increase in drip loss likely due to freeze-cracks and cell membrane damage, particularly in species with very high water content, such as cucumber and bell pepper. For those species, dehydrofreezing or fast freezing at -80 °C is preferred over cryogenic freezing as the measured quality parameters were improved. For precooking before fast freezing, we did not observe a beneficial impact on product quality. In conclusion, when freezing vegetables, convective dehydrofreezing is a promising alternative compared to current technologies. By using mild drying as pre-treatment, quality can be enhanced since cell damage and collapse are being reduced. Its key advantages are that no additional equipment investments are required and that the process does not use considerably more energy compared to conventional freezing. In order to go to industrial implementation, the process operating conditions need to be optimized still to increase the sensory quality more.

Author contributions

T.D. conceptualized the study and wrote the project outline, T.D did project administration and was the Principal Investigator (PI) in the project; S.S., K.P., and T.D. developed the methodology; S.S. conducted the quality measurements; K.P. and S.S. performed the X-ray experiments and image analysis. S.S. and K.P. performed the interpretation and visualization of the results; T.D. supervised K.P and S.S.; S.S. and K. P. wrote the original draft with key inputs from T.D.; T.D. critically reviewed and edited the manuscript; S.S revised the manuscript based on these suggestions.

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.

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Appendix A. Supplementary data

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