Effect of pulsed electric field pretreatment on shrinkage, rehydration capacity and texture of freeze-dried plant materials

T. Fauster, M. Giancaterino, P. Pittia, H. Jaeger

PII: S0023-6438(19)31279-4

DOI: https://doi.org/10.1016/j.lwt.2019.108937

Reference: YFSTL 108937

To appear in: LWT - Food Science and Technology

Received Date: 13 August 2019

Revised Date: 3 December 2019

Accepted Date: 8 December 2019

Please cite this article as: Fauster, T., Giancaterino, M., Pittia, P., Jaeger, H., Effect of pulsed electric field pretreatment on shrinkage, rehydration capacity and texture of freeze-dried plant materials, *LWT* - *Food Science and Technology* (2020), doi: https://doi.org/10.1016/j.lwt.2019.108937.

This is a PDF file of an article that has undergone enhancements after acceptance, such as the addition of a cover page and metadata, and formatting for readability, but it is not yet the definitive version of record. This version will undergo additional copyediting, typesetting and review before it is published in its final form, but we are providing this version to give early visibility of the article. Please note that, during the production process, errors may be discovered which could affect the content, and all legal disclaimers that apply to the journal pertain.

© 2019 Published by Elsevier Ltd.



- 1 Effect of pulsed electric field pretreatment on shrinkage, rehydration capacity and texture of
- 2 freeze-dried plant materials
- 3 Fauster, T.^{1,2,#,*}, Giancaterino, M.^{1,3,#}, Pittia, P.³, Jaeger, H.¹
- 4 ¹Institute of Food Technology, University of Natural Resources and Life Sciences (BOKU), Muthgasse
- 5 *18, 1190 Vienna, Austria.*
- 6 ²*FFoQSI GmbH* Austrian Competence Centre for Feed and Food Quality, Safety and Innovation,
- 7 Technopark 1C, 3430 Tulln, Austria
- 8 ³Faculty of Bioscience and Technologies for Food, Agriculture and Environment, University of
- 9 Teramo, Via R. Balzarini 1, 64100 Teramo, Italy
- [#]Both authors contributed equally to this manuscript.
- 11 * Corresponding author: Thomas Fauster, University of Natural Resources and Life Sciences (BOKU),
- 12 Muthgasse 18, 1190 Vienna, Austria, e-mail: thomas.fauster@boku.ac.at
- 13 Keywords: Pulsed electric field, freeze-drying, shrinkage, rehydration, texture
- 14

15 Abstract

For this study, strawberries and red bell pepper were pre-treated with pulsed electric fields 16 (PEF) to reduce the negative effects on physical properties of the products after freeze-drying. 17 PEF treatment was carried out at constant electric field strength of E=1.0 kV/cm and specific 18 energy input was varied between 0.3–6.0 kJ/kg (treatment time 2.0–28.6 ms). Additionally, 19 the impact of different pre-freezing temperatures (-4 and -40 °C) on the final product quality 20 was described. Investigations showed that due to PEF treatment a significant reduction of the 21 shrinkage phenomena for both bell peppers and strawberries was detected compared to 22 untreated samples with 30 % and 50 % lower volume losses, respectively. The rehydration 23 capacity of PEF pre-treated freeze-dried samples increased for both matrices up to 50 %. 24 25 Furthermore, the mechanical properties of the final product were improved for both matrices with a significant firmness reduction up to 60 %. The results of this study suggest that PEF 26 27 can be an effective pretreatment with low energy requirements to improve the quality of freeze-dried fruits and vegetables. 28

29 1. Introduction

Freeze-dried products get more and more attention and are used directly as snack products or 30 in a broad range of foods, such as dried soups, chocolate or breakfast cereals and cereal bars. 31 The freeze-drying process better maintains the natural shape of the raw material and the 32 nutritional and sensory properties compared to hot air drying since the product is stabilised by 33 freezing and removing water based on sublimation (Fellows, 2009). Freeze-drying is 34 performed under vacuum and consists of three main steps: freezing, primary drying 35 (sublimation) and secondary drying (desorption). In particular, freezing temperature and ice 36 37 morphology can significantly affect the freeze-drying process by modulating the mass transfer and thus the rate of primary and secondary drying (Kochs, Körber, Nunner, & Heschel, 1991; 38 Searles, Carpenter, & Randolph, 2001). 39

To obtain high quality dried products various authors recommended the pretreatment of fruits 40 and vegetables with pulsed electric fields (PEF) to affect economic aspects and quality 41 42 parameters of different drying processes. The permeabilization of the cell membrane by PEF leads to reduced drying time through the acceleration of water transport from the biological 43 44 tissue (Wu & Zhang, 2014). PEF pre-treated carrots (E=1-1.5 kV/cm; n=20 pulses) subjected to hot air drying showed an increased drying rate and improved final product quality in 45 comparison to an untreated control (Gachovska, Simpson, Ngadi, & Raghavan, 2009). Similar 46 findings were stated by Lamanauskas, Šatkauskas, Bobinaitė, and Viškelis (2015), who 47 reported a reduction of the drying time of kiwifruits (T= 50 $^{\circ}$ C) by the application of PEF (E= 48 5 kV/cm) without affecting the colour and the ascorbic acid content of the product. Janositz, 49 Noack, and Knorr (2011) investigated the drying of potato slices and detected an enhanced 50 release of cell liquid during hot air drying for the PEF pre-treated samples. In general, the 51 drying efficiency was found to improve in the range of 40–70 % by PEF (Toepfl, 2006). 52 53 Furthermore, it is already known that PEF affects the freezing process, leading to smaller ice crystals and better product quality after thawing (Jalté, Lanoisellé, Lebovka, & Vorobiev, 54 2009; Shayanfar, Chauhan, Toepfl, & Heinz, 2014; Wiktor, Schulz, Voigt, Witrowa-Rajchert, 55 & Knorr, 2015). As reported by Jalté et al. (2009), Ben Ammar, Lanoisellé, Lebovka, Van 56 Hecke, and Vorobiev (2010), Parniakov, Bals, Mykhailyk, Lebovka, and Vorobiev (2016) and 57 Lammerskitten et al. (2019), the application of PEF as pretreatment for freeze-drying leads to 58 a significant reduction of undesirable shrinkage of apples and potatoes. Moreover, the 59 reduction of shrinkage and collapse phenomena by PEF affects the rehydration capacity of the 60 61 final dried product (Jalté et al., 2009; Parniakov, Bals, Lebovka, & Vorobiev, 2016), which is

especially relevant for the use in instant soups and cereal products. However, in the previous studies, the volume reduction was detected only by visual evaluation, and the mechanisms related to PEF and decreased volume reduction during freeze-drying are still under discussion. Parniakov, Bals, Lebovka, et al. (2016) observed that during the freeze-drying process the temperature in the core of PEF pre-treated samples remains at lower temperatures for a longer time in comparison to untreated samples. Thereby, the frozen water inside the sample protects the original shape.

This work aims to contribute to the further clarification of underlying mechanisms by exact quantification of the volume reduction using 3D-scanning and by measuring the rehydration capacity of freeze-dried strawberries and bell peppers. Furthermore, different freezing temperatures (-40 °C and -4 °C) are investigated in order to take into consideration different states regarding ice crystal size and distribution. Moreover, the impact on texture was investigated of freeze-dried samples over a storage period of 30 days.

75

77

76 2. Material and methods

2.1. Raw material

Commercial strawberries (Fragaria x ananassa var. Elsanta) from Germany and commercial 78 red bell peppers (Capsicum annuum L. var. Roberta) from a local Austrian producer were 79 80 used. Raw materials were selected of good and uniform quality, rotten and unripe fruits were removed. Ripe strawberries and bell peppers were immediately used or stored under 81 controlled conditions (4 °C) and used within 7 days. The strawberries were used in a whole, 82 only a manual washing with water was performed, and the green sepals were removed. The 83 84 red bell peppers were washed and cylinders with a diameter of 3.0 ± 0.1 cm and a thickness of 0.6 ± 0.2 cm were obtained using a cylindrical cutter. 85

86

87 2.2. PEF treatments

The PEF treatments were performed using a PEF batch system (Cellcrack 1, DIL, Germany) consisting of a high voltage power supply, a capacitor with 0.5 μ F and a spark gap used as switch. A treatment chamber consisting of parallel plate electrodes with a distance of 13.5 cm and a total volume of 4.0 L was used. For each treatment, 500 ± 10 g of strawberries (n= 40) or 100 ± 10 g of bell peppers (n=20) were placed in the treatment chamber and filled up with tap water (T= 25 ± 1 °C, conductivity 320–350 μ S/cm), to a final weight of 3.0 and 1.5 kg,

respectively. Exponential decay pulses with a pulse width of 100 µs for the strawberries and 94 143 µs for the bell peppers, and a frequency of 1 Hz were applied. The pulse width was 95 determined at 37 % of the peak voltage detected with a high voltage probe (P6015A, 96 Tektronix, UK) and an oscilloscope (TBS 1102B-EDU, Tektronix, UK). The field strength 97 was set to 1.0 kV/cm, and the number of pulses was varied between 20 and 200. Thus, the 98 treatment time varied between 2.0–28.6 ms. The applied pulses delivered a pulse energy of 99 45.5 J and resulted in a total specific energy input for strawberry and bell pepper between 100 0.3–1.5 and 0.7–6.0 kJ/kg, respectively. Strawberries and bell peppers samples were prepared 101 as control reference (untreated) by immersion in tap water for a duration equal to the duration 102 of the PEF treatment and at the same sample/water ratio. At least three repetitions per 103 treatment condition were conducted for both raw materials. 104

105

106 2.3. Cell disintegration index (CDI)

The degree of cell permeabilization after PEF treatment was determined by calculating the 107 cell disintegration index (CDI). The electrical conductivity was measured with an impedance 108 measurement device (Impedance Analyser, Sigma Check, Germany) in the frequency range of 109 1.4 kHz to 11.2 MHz using a cylindrical sample (diameter 1.0 cm, length 1.0 cm). Impedance 110 curves were obtained for both, the intact and the PEF pre-treated samples. Related CDI was 111 calculated using the equation described by Knorr and Angersbach (1998) considering 112 impedance values at a frequency of 5.5 and 1400 kHz. The value of CDI varies between 0 113 representing intact cells and 1 representing totally disintegrated cells. A reference sample with 114 maximum CDI was obtained by applying three freezing and thawing cycles. 115

116

117 2.4. Freeze-drying process

PEF treated and control samples were placed on stainless steel plates in a single layer, not in 118 contact with each other, and frozen in a blast freezer (Blast Freezer IF101L, Sagi, Italy) at 119 120 temperatures of -4 ± 2 and -40 ± 2 °C for a minimum duration of 2 h. Prior to freeze-drying, samples were tempered at -40 °C for 2 h. The frozen samples were placed onto shelves in a 121 122 lab scale freeze-drier (FreeZone 6, LABCONCO, USA) without additional shelf heating. The condenser was pre-cooled to -45 ± 2 °C before the samples were placed in the freeze drier. 123 The freeze-drying process was performed for 72 h, and the chamber pressure was maintained 124 at 0.5 ± 0.02 mbar throughout the drying process. During the freeze-drying, the temperature 125

of the untreated and PEF treated (0.7 kJ/kg) strawberries frozen at -40°C was recorded with two thermocouples placed at the core and at the external layer of the samples by a temperature monitoring system (E-VAL flex, Ellab, Denmark). After the freeze-drying process, part of the samples was immediately used for physical analyses, and a representative sample of both raw materials was stored for 30 days in tightly sealed glass jars containing 4 g silica gel as water absorber (Carl Roth GmbH, Germany) at T=20 ± 1 °C in the dark.

132

133 2.5. Determination of volume reduction

134 The volume reduction measurement was performed to evaluate the extent of the shrinkage phenomenon that can occur during the freeze-drying process. The volume of the same 135 strawberry was measured before the treatment and after the conducted freeze drying process 136 using a 3D laser scanner (BVM 6600, Perten Instruments, Sweden). The surface area of the 137 bell peppers was determined using an image capture system (DigiEye, version 2.8, VeriVide, 138 UK). The pictures of each sample were analysed by using image J (National Institute of 139 Health, USA) to calculate the difference of the diameter after the freeze-drying. The thickness 140 of the freeze-dried bell peppers was manually determined with a vernier caliper. Measurement 141 was performed with at least five samples per treatment condition. 142

143

144 2.6. Rehydration capacity

The rehydration capacity was determined according to Agnieszka and Andrzej (2010), 145 146 including some modifications. After the freeze-drying treatment, whole strawberries and bell pepper cylinders were submerged in 100 mL of deionised water (20 ± 1 °C), and the weight 147 148 was recorded before and after different immersion times (1, 5, 15, 60 min). To keep the samples submerged during the rehydration measurement a device consisting of a thin iron 149 150 wire and a plastic net was used. The dry matter was determined at the end of the rehydration procedure in an oven at 105 ± 2 °C until a constant weight was achieved (AOAC,1995). The 151 152 results were expressed as rehydration percentage according to Meda and Ratti (2005). Measurement was performed with five samples per treatment condition. 153

154

155 2.7. Mechanical properties

The mechanical properties of the freeze-dried samples were determined by texture analyser (TA-XT Plus, Stable Micro System, UK), equipped with a 5.0 kg load cell. The penetration test was applied on whole strawberries and bell pepper cylinders frozen at -40°C with a cylindrical probe (2.0 mm), at a constant speed of 4.0 mm/s. The mechanical properties were defined by the maximum positive force [N] which represents the hardness of the dried samples (Wang, Zhang, & Mujumdar, 2010). Measurement was performed with ten samples per treatment condition.

163

164 2.8. Statistics

165 The statistical analysis was done with STATISTICA 10 software (Statsoft, USA). The 166 significance level for the one-way ANOVA was set to p < 0.05 and the Tukey test was applied 167 to assess significant differences between investigated parameters. The plotted results are 168 reported as mean value \pm standard deviation.

169

170 **3. Results and Discussion**

171 3.1. Cell disintegration index

The cell disintegration index (CDI), obtained by the impedance analysis is commonly used to 172 evaluate the effects of different PEF parameters applied regarding the permeabilization effect 173 in plant products (Angersbach, Heinz, & Knorr, 1999). The impact of PEF pretreatment at 174 fixed electric field strength (1 kV/cm) and varied specific total energy input on strawberry and 175 bell pepper is shown in Figure 1. It is evident that samples showed a statistically different cell 176 disintegration depending on the applied energy input, with increasing energy input leading to 177 an increase in the CDI. Increasing the energy input from 0.3 to 1.5 kJ/kg for strawberry and 178 0.7 to 6 kJ/kg for bell pepper leads to increased cell disruption of 25 and 30 %, respectively. 179 180 The results obtained for the peppers are in accordance with those reported in previous studies (Ade-Omowaye, Rastogi, Angersbach, & Knorr, 2002, 2003; Jalté et al., 2009). In accordance 181 to Angersbach, Heinz, and Knorr (2000), the investigations additionally demonstrate that 182 depending on the selected raw material, applied energy inputs reached significant differences 183 regarding CDI. For a CDI of 0.6 for strawberries, the application of 0.3 kJ/kg is sufficient. In 184 comparison, 1.5 kJ/kg is required for reaching a similar CDI for bell peppers. The most 185 186 intensive PEF treatment resulted in a CDI of 0.77 for strawberries also showing increased softness and leakage of intracellular content, which could compromise the mechanical 187

processing in industrial scale. However, for bell pepper, these effects and especially the aforementioned leakage were not detectable for the applied energy inputs. The different response to the PEF treatment can be explained by the different intrinsic characteristics of the investigated plant products, such as cell shape and size, electric conductivity, pH or ionic strength (Puértolas, Luengo, Álvarez, & Raso, 2012; Toepfl, Siemer, Saldaña-Navarro, & Heinz, 2014).

194

195 <u>Figure 1</u>

- 196
- 197 3.2. Freeze-drying kinetics

Since the characteristics of a freeze-dried product depend on the material temperature during 198 the process (Roos, 1997), the temperature of the outer layer (Out) and the inner layer (In) of 199 PEF treated (0.7 kJ/kg) and untreated strawberries were monitored during the freeze-drying 200 process (Figure 2). The outer layer of the untreated and treated sample (Out_Untreated and 201 Out_PEF) show the same trend and it was not possible to highlight significant differences 202 during the course of freeze-drying. Just after the beginning of the freeze-drying process, the 203 outer layers showed an increase in temperature, indicating the rapid removal of water from the 204 external layer. The temperature for both inner layers (In_Untreated and In_PEF) was constant 205 206 or slightly decreased in the first phase of the process and then increased up to the shelf 207 temperature. During the freeze-drying process, the rapid increase of the temperature in the core of the product can be considered as the end of primary drying (sublimation) and the 208 209 beginning of secondary drying (desorption) (Kramer et al., 2009). The untreated sample recorded a constant temperature for 9 h, afterwards the temperature slightly increased until it 210 211 reached the shelf temperature. The inner section of the PEF treated sample showed a lower temperature, with a minimum value of -17.36 °C, within 19.5 h from the start of freeze-212 213 drying, followed by a rapid temperature increase. The untreated and PEF treated samples reached the shelf temperature after 39.3 and 38.6 h, respectively. These results illustrate that 214 215 PEF has no significant effect on the duration of the freeze-drying process. However the increased pore formation leads to a prolonged sublimation step and thus stabilizes the shape 216 of the final product. Similar concepts were reported by Parniakov, Bals, Lebovka, et al. 217 (2016). In their study, PEF treated apples, depending on the degree of cell disintegration, 218 show lower temperatures during freeze-drying and a delayed temperature rise. During fast 219 freezing, small ice crystals occur that result in the formation of small pores after their 220

sublimation. This can result in a higher resistance to mass transfer and thus representing a 221 limit for sublimation (Patapoff & Overcashier, 2002). The resistance to the passage of water 222 vapour can increase the temperature at the sublimation interface resulting in partial melting of 223 the ice, which leads to the plasticization of solids and structural collapse (Harnkarnsujarit, 224 Charoenrein, & Roos, 2012). The effect of PEF pretreatment can be compared to slow 225 freezing, with the formation of large ice crystals in the extracellular space. This promotes low 226 dried product resistance and low product temperature with reduced consequences on the 227 volume changes of freeze-dried products (Assegehegn, Brito-de la Fuente, Franco, & 228 Gallegos, 2018; Harnkarnsujarit et al., 2012). However, the formation of small crystals can 229 favour desorption for which a larger specific area is required (Hottot, Vessot, & Andrieu, 230 2004). Jalté et al. (2009) also stated that the application of PEF favours the mass transfer 231 during freezing and the formation of larger ice crystals especially in the extracellular space, 232 233 which promotes sublimation and keeps the samples at lower temperatures for a longer time. Moreover, Rambhatla, Obert, Luthra, Bhugra, and Pikal (2005) suggested that the shrinkage 234 235 occurs during early secondary drying when the moisture of the product is still high and the temperature is close to the glass transition temperature. 236

- 237
- 238 *Figure 2*
- 239

240 3.3. Volume reduction

241 During different drying processes, the water removal causes changes on the physical structure of food and phenomena such as shrinkage can occur determining loss of the structural 242 properties and thus the quality of the product and consumer acceptance (Agudelo-Laverde, 243 Schebor, & del Pilar Buera, 2014). Collapsed or shrunk products show poor rehydration 244 characteristics and uneven and extended drying (Tsourouflis, Flink, & Karel, 1976). Drying at 245 sufficiently low temperatures permits to limit the effect on the physical structure of the 246 samples thus freeze-drying allows better results compared to other dehydration methods 247 (Krokida, Karathanos, & Maroulis, 1998). As mentioned before, to avoid the increase of 248 temperature and thus the shrinkage, an increased mass transfer is required and can be 249 achieved through pretreatments such as PEF or slow freezing. The effects of PEF 250 pretreatment regarding volume reduction after the freeze-drying of whole strawberries and 251 bell pepper discs are shown in Figure 3. The untreated strawberries frozen at -40 °C show a 252

volume reduction of approximately 48 ± 16 %. In comparison, the PEF treated strawberries 253 show significantly lower volume reduction. The lowest energy input (0.3 kJ/kg) results in the 254 highest reduction with only 19 ± 8 % of volume loss and an energy input of 1.5 kJ/kg leads to 255 a volume reduction of 15 ± 6 %. The PEF treated samples show not only a lower volume 256 257 reduction but also a lower variability of the data, which reflects a higher homogeneity of the product indicating that cell disintegration due to PEF reduces mass transfer barriers in the 258 frozen product and results in a more uniform tissue structure. Additionally, the beneficial 259 shrinkage behaviour of the PEF pre-treated freeze-dried matrices is of main importance for 260 marketing purposes. The observed volume reduction for strawberries was higher than reported 261 by Shishehgarha, Makhlouf, and Ratti (2002). This might be attributed to different raw 262 material properties and slightly different process conditions. For the bell peppers frozen at -263 40 °C (Figure 3b) similar findings were obtained, thereby the PEF pretreatment leads to a 264 volume reduction of approximately 30 % independent from the applied energy input. The 265 untreated samples showed a volume change higher than 80%. The volume reduction of 266 strawberries frozen at -4 °C was 15.1 % for the untreated and 7.1–9.0 % for the PEF treated 267 samples. For the bell peppers frozen at -4 °C, the untreated samples showed a volume change 268 269 of 62.9 % and the PEF pretreatment leads to a volume reduction of 33.7-46.0 %. For the untreated samples frozen at -4 °C a significant reduction of shrinkage phenomenon occurs 270 271 compared with the fast freezing. As discussed earlier, this can be attributed to the formation of larger ice crystals, which favour a more rapid removal of the water vapour during 272 sublimation. However, for PEF treated samples, the benefit of the higher freezing temperature 273 is less pronounced or, in other words, when freezing at higher temperatures, only a small 274 additional benefit can be derived from PEF pretreatment. The PEF treated strawberries were 275 only slightly improved by the higher freezing temperature and no significant difference 276 between the different freezing temperatures could be observed in the case of PEF treated bell 277 peppers. The differences between the two matrices can be due to their different composition 278 which affects the collapse temperature (Bhandari & Howes, 1999). These results confirm that 279 the main aspect that limits the shrinkage phenomenon is the improvement of the mass transfer 280 that allows keeping the temperature of the samples lower than their glass transition 281 temperatures (T_g) for a longer time. In accordance with Levi and Karel (1995), the collapse 282 phenomenon is a dynamic process occurring when the process temperature is above the T_{g} . 283 Lammerskitten et al. (2019) further describe that the application of PEF treatment results due 284 to the facilitated movement of components in a more homogeneous distribution of water and 285 sugar inside of the investigated raw material and thereby sugars are less able to seals natural 286

pores. The fact that the improvement resulting from a PEF treatment was most pronounced for 287 the low freezing temperature at -40 °C represents a major benefit with industrial relevance. 288 The current freezing processes are performed at low temperatures in order to allow short 289 processing times and provide the appropriate freezing conditions especially for the 290 manufacturing of individually quick frozen (IQF) product. Moreover, the low heat transfer 291 coefficient of slow freezing leads beside long holding times to significant quality loss (Singh 292 & Wang, 1977). In that case, the PEF treatment can significantly contribute to an improved 293 quality without losing the benefits of the lower freezing temperature. 294

295

296 *Figure 3*

297

298 3.4. Rehydration

Rehydration of food is a complex phenomenon affected by numerous factors like pre-drying 299 treatments, drying technology and conditions, food structure, composition and medium 300 viscosity (Alejandro Marabi & Saguy, 2004). A study conducted by A. Marabi, Livings, 301 Jacobson, and Saguy (2003) showed that for freeze-dried samples the most crucial mechanism 302 is capillary diffusion. In Table 1 the results of the rehydration capacity and behaviour of 303 strawberries and bell peppers immediately after the freeze-drying process are shown. 304 Regardless of the freezing temperature, significant advantages in terms of rehydration ability 305 were determined for both matrices by the application of PEF. Freeze-dried strawberries frozen 306 at -40 °C had better rehydration capacity for energy inputs higher than 0.7 kJ/kg. After just 307 one minute of immersion, the PEF treated strawberries indicate a 49.5-70.7 % higher 308 rehydration rate than the untreated samples. After 15 min of immersion, it was possible to 309 detect differences depending on the applied PEF treatment intensity. Strawberries frozen at -310 4 °C showed a decrease of the rehydration rate compared to the sample subjected to rapid 311 freezing. Also for the products frozen at -4 °C, the PEF treated samples showed a better 312 rehydration compared to the untreated samples. In particular, the sample treated with the 313 highest PEF intensity (1.5 kJ/kg) had a statistically higher rehydration at all immersion times. 314 As for the samples frozen at -40 °C, after just one minute of immersion, the PEF treated 315 samples show a rehydration being 52.5–68.6 % higher than the untreated sample. It can be 316 assumed that the freezing process at -4 °C has a significant influence on the final structure of 317 the freeze-dried strawberries as the rehydration capacity can be considered as a valid measure 318 319 to assess the state of injury caused by pretreatments and drying process (Krokida et al., 1998).

The behaviour of freeze-dried peppers frozen at -40 °C was similar to the strawberries. 320 However, up to 15 min of immersion only the sample treated with the highest PEF intensity 321 (6.0 kJ/kg) showed a statistically higher rehydration. After 60 min of immersion, the 322 rehydration of PEF treated samples was 22.1-54.0 % higher compared with the untreated 323 control. For freeze-dried bell peppers frozen at -4 °C the freezing temperature didn't affect the 324 rehydration capacity and the PEF treated samples showed again a higher rehydration already 325 after the first minute of immersion compared to the untreated samples. Generally, the 326 increased number of pores in the membrane induced by PEF favours the capillary diffusion 327 328 process and may cause the increased rehydration capacity of the PEF treated samples. Jalté et al. (2009) and Parniakov, Bals, Mykhailyk, et al. (2016) stated similar results for potatoes and 329 apples, relating the better rehydration for PEF treated samples to a higher porosity of the 330 vegetable matrix after the freeze-drying process. Furthermore, the different rehydration 331 ability of the investigated matrices could be described by their specific cellular and 332 microstructural properties, considering that the rehydration capacity is closely dependent by 333 the used plant material (Witrowa-Rajchert & Lewicki, 2006). In particular, Lee, Farid, and 334 Nguang (2006) reported that different plant materials can have different rehydration rates 335 336 according to their microscopic structure even when the drying conditions are the same.

337

338 <u>*Table 1*</u>

339

Texture is one of the most important physical parameter for dehydrated products, which can 341 drastically influence the consumer's acceptability (Fan, Zhang, & Mujumdar, 2018). The 342 mechanical properties of dried food products are affected by moisture content, food 343 344 composition, pH, and the ripening stage of the raw material (Sagar & Kumar, 2010). For the determination of the mechanical properties of the freeze-dried strawberries and bell peppers 345 texture measurement was conducted. The results of the penetration test measured after freeze-346 drying and 30 days of storage are plotted in Figure 4. The maximum force decreases 347 significantly from 8.6 to 4.2–4.5 N for strawberry and from 8.4 to 2.4–3.3 N for bell pepper 348 due to the applied PEF pretreatment independent of the treatment intensity. It can be stated 349 that the PEF decreased the hardness of freeze-dried strawberries and bell peppers by 47.4-350 50.7 % and 61.1–71.1 % respectively, when compared to the untreated samples. Ben Ammar 351 et al. (2010) and Lammerskitten et al. (2019) also reported a noticeable softening for freeze-352

^{340 3.5.} Texture

dried PEF pre-treated potatoes and apples. The softer structure of the pre-treated samples can
be explained by the increased pore formation due to PEF, which leads to reduced shrinkage
and higher porosity of the samples (Chauhan, Shayanfar, & Toepfl, 2019).

356 The product stability was evaluated by measuring the textural properties after a storage period of 30 days. No significant changes of firmness were found for both untreated products after 357 30 days of storage. Also, the firmness of the PEF pre-treated products was found to remain 358 unchanged over the storage period. It has to be mentioned that the product stability especially 359 360 the crispiness of freeze-dried products is particularly affected by the humidity during storage, therefore the selection of suitable packaging material and storage conditions is crucial for the 361 362 transfer of results. In the current study, appropriate storage avoided changes in the moisture content of the samples during storage which could result in changes of the textural properties 363 364 of the products.

rent

365

366 *<u>Figure 4</u>*

367

368 4. Conclusion

The pore formation due to the applied PEF treatment prior to freeze-drying reduces the 369 shrinkage phenomenon and increases the rehydration capacity of freeze-dried strawberries 370 and bell peppers. The present results indicate that the improved mass transfer resulting from 371 the application of PEF promotes the primary drying by keeping the product temperature at 372 lower values for a longer time. This affects the product structure in a way that shrinkage and 373 374 collapse of the tissue are reduced. The beneficial effects on volume reduction have already been observed at the lowest applied energy input and CDI. Besides the cell disintegration 375 achieved by PEF treatment, the volume reduction can be further reduced by increasing the cell 376 disintegration and mass transfer due to slow freezing and related formation of larger ice 377 crystals. Moreover, regardless of freezing temperature, PEF treated samples show higher 378 rehydration capacity compared to untreated samples. Further basic research is needed in order 379 380 to clarify the underlying mechanisms of this process-structure-function interaction affecting the freezing and freeze-drying behaviour. However, based on the obtained results, the 381 application of PEF treatment in industrial scale especially at low freezing temperatures seems 382 383 to be a promising approach to preserve the initial shape and improve the rehydration of freeze-dried fruits and vegetables. 384

385 5. Conflict of interest

386 The author have no conflict of interest

387

Journal Prevention

388 6. Acknowledgement

This work was created within a research project of the Austrian Competence Centre for Feed and Food Quality, Safety and Innovation (FFoQSI). The COMET-K1 competence centre FFoQSI is funded by the Austrian ministries BMVIT, BMDW and the Austrian provinces Niederoesterreich, Upper Austria and Vienna within the scope of COMET - Competence Centers for Excellent Technologies. The programme COMET is handled by the Austrian Research Promotion Agency FFG. Part of the equipment used in this study was financed by EQ-BOKU VIBT GmbH and belongs to the Center for Preservation and Aseptic Processing.

396

397 7. References

- Ade-Omowaye, B., Rastogi, N., Angersbach, A., & Knorr, D. (2002). Osmotic dehydration of bell
 peppers: influence of high intensity electric field pulses and elevated temperature treatment.
 Journal of food Engineering, 54(1), 35-43.
- Ade-Omowaye, B., Rastogi, N., Angersbach, A., & Knorr, D. (2003). Combined effects of pulsed
 electric field pre-treatment and partial osmotic dehydration on air drying behaviour of red
 bell pepper. *Journal of Food Engineering*, 60(1), 89-98.
- 404 Agnieszka, C., & Andrzej, L. (2010). Rehydration and sorption properties of osmotically pretreated
 405 freeze-dried strawberries. *Journal of Food Engineering*, *97*(2), 267-274.
- Agudelo-Laverde, L. M., Schebor, C., & del Pilar Buera, M. (2014). Evaluation of structural shrinkage
 on freeze-dried fruits by image analysis: Effect of relative humidity and heat treatment. *Food and Bioprocess Technology*, 7(9), 2618-2626.
- Angersbach, A., Heinz, V., & Knorr, D. (1999). Electrophysiological model of intact and processed
 plant tissues: cell disintegration criteria. *Biotechnology Progress*, *15*(4), 753-762.
- Angersbach, A., Heinz, V., & Knorr, D. (2000). Effects of pulsed electric fields on cell membranes in
 real food systems. *Innovative Food Science & Emerging Technologies*, 1(2), 135-149. doi:
 https://doi.org/10.1016/S1466-8564(00)00010-2
- Assegehegn, G., Brito-de la Fuente, E., Franco, J. M., & Gallegos, C. (2018). The Importance of
 Understanding the Freezing Step and its Impact on Freeze Drying Process Performance. *Journal of Pharmaceutical Sciences, 108*(4), 1378-1395. doi:
- 417 https://doi.org/10.1016/j.xphs.2018.11.039.
 418 Ben Ammar, J., Lanoisellé, J.-L., Lebovka, N. I., Van Hecke, E., & Vorobiev, E. (2010). Effect of a Pulsed
 419 Electric Field and Osmotic Treatment on Freezing of Potato Tissue. *Food Biophysics, 5*(3),
- 420 247-254. doi: 10.1007/s11483-010-9167-y
- Bhandari, B., & Howes, T. (1999). Implication of glass transition for the drying and stability of dried
 foods. *Journal of Food Engineering*, 40(1-2), 71-79.
- Chauhan, O. P., Shayanfar, S., & Toepfl, S. (2019). Cell Permeabilisation, Microstructure and Quality
 of Dehydrated Apple Slices Treated with Pulsed Electric Field During Blanching. *Defence Life Science Journal*, 4(1), 38-44.
- Fan, K., Zhang, M., & Mujumdar, A. S. (2018). Recent developments in high efficient freeze-drying of
 fruits and vegetables assisted by microwave: A review. *Critical Reviews in Food Science and Nutrition*, 1-10.
- Fellows, P. J. (2009). *Food processing technology: principles and practice* (3th ed.). Cambridge:
 Woodhead Publishing in Food Science, Technology and Nutrition.

431	Gachovska, T. K., Simpson, M. V., Ngadi, M. O., & Raghavan, G. (2009). Pulsed electric field treatment
432	of carrots before drying and rehydration. Journal of the Science of Food and Agriculture,
433	<i>89</i> (14), 2372-2376.
434	Harnkarnsujarit, N., Charoenrein, S., & Roos, Y. H. (2012). Microstructure formation of maltodextrin
435	and sugar matrices in freeze-dried systems. Carbohydrate Polymers, 88(2), 734-742.
436	Hottot, A., Vessot, S., & Andrieu, J. (2004). A direct characterization method of the ice morphology.
437	Relationship between mean crystals size and primary drying times of freeze-drying
438	processes Drying Technology 22(8) 2009-2021
439	lalté M. Lanoisellé II. Leboyka N. L. & Vorobiev, F. (2009). Freezing of notato tissue pre-treated
433	by pulsed electric fields [doi: DOI: 10 1016/i lwt 2008 09 007] / WT - Food Science and
441	Technology, 42(2), 576-580.
442	Janositz, A., Noack, A. K., & Knorr, D. (2011). Pulsed electric fields and their impact on the diffusion
443	characteristics of potato slices. LWT - Food Science and Technology, 44(9), 1939-1945. doi:
444	https://doi.org/10.1016/i.lwt 2011.04.006
445	Knorr D & Angershach A (1998) Impact of high-intensity electric field nulses on plant membrane
116	permeabilization Trends in Food Science & Technology (25) 185-191 doi:
440	https://doi.org/10.1016/S0924-2244/98)00040-5
447 110	Kochs M. Körber C. Number P. & Heschel J. (1001) The influence of the freezing process on
440	Nochs, M., Kolber, C., Numer, B., & Hescher, I. (1991). The innuence of the neezing process on
449	and Mass Transfer, 34(9), 2395-2408.
451	Kramer, T., Kremer, D., Pikal, M., Petre, W., Shalaev, E., & Gatlin, L. (2009). A procedure to optimize
452	scale-up for the primary drying phase of lyophilization. <i>Journal of Pharmaceutical Sciences</i> .
453	98(1), 307-318.
454	Krokida, M. K., Karathanos, V. T., & Maroulis, Z. B. (1998). Effect of freeze-drying conditions on
455	shrinkage and porosity of dehydrated agricultural products. <i>Journal of Food Engineering</i>
456	<i>35</i> (4), 369-380. doi: https://doi.org/10.1016/S0260-8774(98)00031-4
457	Lamanauskas, N., Šatkauskas, S., Bobinaitė, R., & Viškelis, P. (2015). Pulsed Electric Field (PEF) Impact
458	on A ctinidia kolomikta Drying Efficiency. Journal of Food Process Engineering, 38(3), 243-
459	249.
460	Lammerskitten, A., Wiktor, A., Siemer, C., Toepfl, S., Mykhailyk, V., Gondek, E., Rybak, K., Witrowa-
461	Rajchert, D., & Parniakov, O. (2019). The effects of pulsed electric fields on the quality
462	parameters of freeze-dried apples. Journal of Food Engineering, 252, 36-43. doi:
463	https://doi.org/10.1016/i.ifoodeng.2019.02.006
464	Lee, K. T., Farid, M., & Nguang, S. K. (2006). The mathematical modelling of the rehydration
465	characteristics of fruits. Journal of Food Engineering, 72(1), 16-23. doi:
466	https://doi.org/10.1016/i.ifoodeng.2004.11.014
467	Levi, G., & Karel, M. (1995). Volumetric shrinkage (collapse) in freeze-dried carbohydrates above their
468	glass transition temperature. Food Research International, 28(2), 145-151.
469	Marahi A Livings S Jacobson M & Saguy J S (2003) Normalized Weibull distribution for
470	modeling rehydration of food particulates. European Food Research and Technology 217(4)
470 //71	311-318 doi: 10.1007/s00217-003-0719-v
471	Marabi A & Saguy I S (2004) Effect of porosity on rehydration of dry food particulates <i>Journal of</i>
472	the Science of Food and Agriculture 84(10) 1105-1110 doi: 10.1002/isfa.1703
475	Mode L & Batti C (200E) Bobydration of frage dried strawbarries at varying temperatures
4/4	lowing of Food Drocoss Engineering, 29(2), 222-246
475	Journal of Food Process Engineering, 20(5), 255-240.
470	Parillakov, O., Bals, O., Lebovka, N., & Voroblev, E. (2016). Pulsed electric field assisted vacuum
477	Treeze-drying of apple tissue. Innovative Food Science & Emerging Technologies, 35, 52-57.
478 470	ramiakov, O., Bais, O., iviyknaliyk, V., Lebovka, N., & Voroblev, E. (2016). Untreezable water in apple
479	reated by pulsed electric fields: impact of osmotic impregnation in giveerol solutions. Food
40U	unu Bioprocess rechnology, $9(2)$, 243-251.
481	Patapoli, I. W., & Overcashier, D. E. (2002). The importance of freezing on lyophilization cycle
482	aevelopment. <i>Biopharm, 15</i> (3), 16-21.

483	Puértolas, F., Luengo, F., Álvarez, L. & Raso, J. (2012). Improving Mass Transfer to Soften Tissues by
484	Pulsed Electric Fields: Eundamentals and Applications. Annual Review of Food Science and
485	Technology, 3(1), 263-282, doi: 10.1146/annurev-food-022811-101208
486	Rambhatla S. Obert, L. Luthra, S. Bhugra, C. & Pikal, M. I. (2005). Cake shrinkage during freeze
487	drying: A combined experimental and theoretical study. <i>Pharmaceutical Development and</i>
488	Technology 10(1) 33-40
489	Roos Y (1997) Frozen state transitions in relation to freeze drying <i>Journal of Thermal Analysis</i>
490	48(3), 535-544.
491	Sagar, V., & Kumar, P. S. (2010). Recent advances in drying and dehydration of fruits and vegetables:
492	a review. Journal of Food Science and Technology, 47(1), 15-26.
493	Searles, J. A., Carpenter, J. F., & Randolph, T. W. (2001). The ice nucleation temperature determines
494	the primary drying rate of lyophilization for samples frozen on a temperature-controlled
495	shelf. Journal of Pharmaceutical Sciences, 90(7), 860-871.
496	Shayanfar, S., Chauhan, O., Toepfl, S., & Heinz, V. (2014). Pulsed electric field treatment prior to
497	freezing carrot discs significantly maintains their initial quality parameters after thawing.
498	International Journal of Food Science & Technology, 49(4), 1224-1230.
499	Shishehgarha, F., Makhlouf, J., & Ratti, C. (2002). Freeze-drying characteristics of strawberries. Drying
500	Technology, 20(1), 131-145.
501	Singh, R. P., & Wang, C. (1977). Quality of frozen foods—a review. Journal of Food Process
502	Engineering, 1(2), 97-127.
503	Toepfl, S. (2006). Pulsed Electric Fields (PEF) for Permeabilization of Cell Membranes in Food-and
504	Bioprocessing–Applications, Process and Equipment Design and Cost Analysis. PhD Thesis,
505	Berlin University of Technology, Berlin.
506	Toepfl, S., Siemer, C., Saldaña-Navarro, G., & Heinz, V. (2014). Overview of pulsed electric fields
507	processing for food. In D. W. Sun (Ed.), Emerging Technologies for Food Processing (2nd ed.,
508	pp. 93-114): Academic Press.
509	Tsourouflis, S., Flink, J. M., & Karel, M. (1976). Loss of structure in freeze-dried carbohydrates
510	solutions: effect of temperature, moisture content and composition. Journal of the Science of
511	Food and Agriculture, 27(6), 509-519.
512	Wang, R., Zhang, M., & Mujumdar, A. S. (2010). Effect of osmotic dehydration on microwave freeze-
513	drying characteristics and quality of potato chips. Drying Technology, 28(6), 798-806.
514	Wiktor, A., Schulz, M., Voigt, E., Witrowa-Rajchert, D., & Knorr, D. (2015). The effect of pulsed electric
515	field treatment on immersion freezing, thawing and selected properties of apple tissue.
516	Journal of Food Engineering, 146, 8-16. doi:
517	http://dx.doi.org/10.1016/j.jfoodeng.2014.08.013
518	Witrowa-Rajchert, D., & Lewicki, P. P. (2006). Rehydration properties of dried plant tissues.
519	International Journal of Food Science & Technology, 41(9), 1040-1046. doi: 10.1111/j.1365-
520	2621.2006.01164.x
521	Wu, Y., & Zhang, D. (2014). Effect of pulsed electric field on freeze-drying of potato tissue.
522	International Journal of Food Engineering, 10(4), 857-862.

Rehydration time [min]	1		5		15		60		
Freezing temperature [°C]	-40	-4	-40	-4	-40	-4	-40	-4	
Energy input [kJ/kg]				stra	wberry				
untreated	3.27±1.50a	2.10±0.69a	4.69±2.06a	2.70±0.87a	5.95±2.33a	3.37±1.01a	8.62±2.91a	5.54±1.38a	
0.3	6.48±1.51ab	4.44±1.73ab	8.87±2.56ab	5.84±2.28ab	10.65±2.68ab	7.50±2.79ab	13.96±3.17ab	10.92±3.08ab	
0.7	11.16±3.48b	6.66±4.29ab	13.21±3.65b	8.89±4.99ab	14.73±3.29bc	11.03±5.46ab	17.69±2.99bc	15.68±6.34b	
1.5	10.94±4.50b	4.55±0.83b	13.99±3.79b	6.64±1.18b	16.07±3.38c	8.46±1.68b	19.61±3.18c	12.94±2.51b	
Energy input [kJ/kg]	bell pepper								
untreated	6.62±0.94a	7.68±1.4a	9.56±1.22a	11.56±1.4a	12.73±1.79a	15.54±2.24a	18.99±2.02a	22.71±1.93a	
1.5	8.93±1.05a	14.85±5.29ab	13.64±2.07a	23.06±6.64b	18.13±2.64ab	29.48±7.8b	24.44±3.9ab	38.99±8.37b	
3	11.15±4.07a	15.21±4.68ab	16.16±3.85a	21.43±5b	21.32±3.92b	27.47±4.91b	29.83±3.14b	35.94±3.48b	
6	24.29±7.37b	18.26±6.34b	30.5±7.42b	24±6.55b	35.24±7.19c	29.69±5.21b	41.35±6.52c	37.71±1.73b	

Table 1 Rehydration capacity (%) of untreated and PEF pre-treated (1 kV/cm) freeze-dried strawberry and bell pepper over a rehydration time of 60 min at 20°C. Different letters in the same column of each raw material indicate significant differences (p<0.05)



Figure 1 Cell disintegration index of PEF pretreated (1 kV/cm) strawberries and bell peppers related to different specific energy inputs. Different letters for each material indicate significant differences (p<0.05)

Johnalbreit

Temperature [°C] -5 -10 -15 -20 -25 Time [h]

Figure 2 Temperature behaviour of the outer (dotted line) and inner (full line) layers of PEF treated \blacksquare (1kV/cm 0.7 kJ/kg) and untreated \blacksquare strawberries during freeze-drying, freezing temperature -40 °C.

Jonuugibie



Figure 3 Volume reduction (%) resulting from freeze-drying of untreated and PEF pretreated (1 kV/cm) strawberries (a) and bell peppers (b) frozen at -40 (\square) and -4 °C (\square). Significant differences for equal freezing temperature (-40 °C=a, -4 °C=a*) is indicated by different letters (p<0.05)



Figure 4 Texture measurement of PEF pretreated (1 kV/cm) and untreated freeze-dried strawberries (a) and bell peppers (b) immediately after processing (\square) and 30 days of storage (\square). Different letters indicate significant statistical differences (p< 0.05)

Highlights

- Quantitative determination of shrinkage phenomena was performed by innovative 3D scanning method
- PEF treatment significantly improved the freeze-drying process due to the enhanced mass transfer
- Volume loss and firmness were reduced and rehydration capacity increased for strawberries and bell pepper
- PEF effects are more pronounced at lower freezing temperatures as usually applied during industrial quick freezing processes
- The impact of PEF on selected physical parameters is already detectable at low energy inputs of 0.3 kJ/kg

Journal

Conflict of interest

We know of no conflicts of interest associated with this publication, and there has been no significant financial support for this work that could have influenced its outcome. As corresponding author, I confirm that the manuscript hast been read and approved for submission by all the named authors.

Sumalprophy

Author contribution statements

TF, MG and HJ conceived and planned the experiments.

TF and MG carried out the experiments and contributed to sample preparation.

TF, MG, PP and HJ contributed to the interpretation of the results.

TF and MG took the lead in writing the manuscript.

All authors provided critical feedback and helped shape the research, analysis and manuscript.