Effect of Pulsed Electric Field on Collapse Phenomenon of Apple Tissue in Vacuum Freeze Drying

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Abstract: The effects of pulsed electric field (PEF) on drying characteristics and collapse phenomenon during vacuum freeze drying of apple were investigated. The effects of PEF treatment on drying time, specific energy consumption and rehydration ratio were studied separately with different temperature. Results showed that the board temperature should below the collapse temperature of apple to increase the drying rate without destroying the quality during sublimation processing, and the collapse temperature of the apple after PEF treatment was 75°C, the board temperature should be as high as possible at the maximum allowable temperature of 85-95°C during the desorption processing according to the drying experiment and microstructure analysis. Moreover, the influence mechanism of PEF treatment on collapse phenomenon was discussed from the perspective of moisture migration and mechanics. Therefore, PEF treatment can improve the diffusion rate, and increase the collapse temperature.

1. Introduction
Freeze drying is very popular in processing of foodstuffs and widely used for food preservation. However, this process is very energy intensive and long drying time [1, 2]. Therefore it is necessary to optimize and improve the freeze drying process. The freeze drying process is conventionally divided into three stages: pre-freezing; sublimation drying under vacuum, known as primary drying; followed by desorption, or secondary drying. After primary drying, residual moisture content may be as high as 7% [3]. During the sublimation drying process, in order to improve the drying rate, the drying temperature should be as high as possible. However, the drying temperature should be lower than the collapse temperature to avoid denaturing, or collapsing phenomenon. When the temperature of the sample is increased by 1°C during the drying process, the sublimation drying time will be shortened by 13%, and the drying rate can be increased by 11% [4]. Hence, the drying temperature must be kept below and approach the collapse temperature to shorten the process time.

Collapse phenomena such as structural collapse and volume contraction (shrinkage) often occur sublimation drying process. Collapse and shrinkage of the pore network have been found to depend on the water content and temperature and to occur above glass transition temperatures [5]. Venir et al. showed the collapse temperature of anhydrous sample was 28 ± 2°C [6]. The freezing curve and Glass transition lines were developed using Clausius-Clapeyron and Gordon-Taylor models by Mauric et al. The effective molecular weight of solids in apple was 75.6, and glass transition temperature of solid
was 41.3℃ [7]. Barresi et al. studied the effect of the drying temperature and composition on the product appearance and final characteristics of pure substances, they found that if glass transition temperature is exceeded during the sublimation drying, a collapse of the cake structure can occur, the product cake loses its stiff pore structure and undergoes collapse. The collapse temperature and the glass transition temperature are closely related and the collapse temperature is normally a few degrees centigrade higher than glass transition temperature [8]. Ohori et al. investigated the impact of the ramp rate during the sublimation drying process on the properties and drying behavior of lyophilized cakes. The lyophilized cakes were found to collapse as the ramp rate decreased. And the ramp rate is potentially a critical parameter in controlling collapse, and an investigation of the ramp rate may be necessary for the establishment of a successful lyophilization process [9]. Shishehgarha et al. reported that the percentage of collapsed strawberries increased with process temperature. At heating temperatures higher than 50°C, the strawberry dry layer temperature was higher than the estimated glass transition temperature of dried fruit, increasing the risk of collapse [10].

Usually, in order to improve the permeability of the cells, save the energy and drying time, the cell structure of the fruits and vegetables is needed to be pretreated. Combined vacuum freeze-drying and pulsed electric field (PEF), as a new processing technology for drying of fruits and vegetables, have the advantage of minimal deterioration of original color, flavor, texture, or nutritional values [11]. Furthermore, PEF treatment can improve the collapse temperature of fruits and vegetables to a certain extent [12]. The aim of this study was to evaluate the effect of PEF treatment on the collapse temperature of apple tissue in the processes of vacuum freeze-drying.

2. Material and method

2.1 Sample preparation
Apples were purchased in a local supermarket (Taiyuan, China). The moisture content, measured by drying 25 g of the fresh apple tissue at 105 °C to constant weight, was about (85.89±0.50)wt.% according to the direct drying method (GB 5009.3-2016, China) using the air blowing thermostatic oven. The apples were sliced parallel to the main axis of the apple, then cut into 20 mm long, 20 mm width and 10 mm thick.

2.2 PEF treatment
PEF treatment was applied using a monopolar PEF generator (BTX ECM 830, Harvard Apparatus; Holliston, MA). The PEF generator provided pulses of a near-rectangular shape. The following protocol was used in PEF experiments: E =1000 V/cm, n = 35, ti = 120 μs, Δt =500 ms. Untreated samples by PEF as the control group.

2.3 Vacuum freeze drying experiment
Vacuum freeze drying experiment was carried out by JDG-0.2 vacuum freeze-drying machine (Kejin Vacuum Freeze-drying Technics Co. Ltd., China). The parameters of vacuum freeze drying experiment were set as follow: the pre-freeze temperature was -35°C for 10 h, the board temperature was set to 65°C, 75°C, 85°C, respectively, vacuum degree was 40 Pa during the sublimation drying processing, the board temperature was 95°C, and the vacuum degree was 30 Pa during the desorption drying processing.

Drying time (h) was determined while the final moisture content of dried samples did not decrease anymore during desorption drying process. Specific energy consumption (kJ·g⁻¹) was equal to the ratio of electricity consumption (kJ) over the initial mass (g).

2.4 Rehydration experiments
Rehydration ratio was determined by immersing a weighed amount of dried samples into hot water at 20°C for 8 h. The soaked samples were blotted with a paper towel for 4-5 times to remove excess water. The rehydration ratio was calculated by following formula:
Rehydration ratio = \frac{g_f}{g_d}

Where \( g_d \) and \( g_f \) are weights of the samples before and after rehydration, respectively.

2.5 Microstructure

After vacuum freeze drying, the structure of dried apple that untreated and PEF treated were randomly chosen for microscopic examination by SEM (SM-7100F, JEOL, Japan). The micrographs were recorded at 100× magnification to observe the change of cell microstructure.

3. Results and discussion

3.1 Influence of different drying conditions on drying characteristics

Table 1 represents that the effect of different drying temperature on drying time, specific energy consumption and rehydration ratio of PEF treatment apple tissue, and the untreated sample as the control group. In Table 1, the board temperature was the sublimation drying temperature. From Table 1, the drying time was decrease with increasing the board temperature, the drying time was saved 1.13 h when the board temperature was increased from 65℃ to 75℃, and however, the drying time was only saved 0.23 h when the board temperature was increased from 75℃ to 85℃. This indicated that when the board temperature reached a certain value, increased the board temperature has no significant effect on drying time.

When the temperature was 65℃, 75℃, 85℃, the specific energy consumption was 329.03 kJ·g\(^{-1}\), 300.75 kJ·g\(^{-1}\), 289.86 kJ·g\(^{-1}\) respectively, while that of the control group was 360.41 kJ·g\(^{-1}\) at the temperature of 75℃. Therefore, PEF processing could greatly reduce the energy consumption. In an electric field, the evaporation rate of water was significantly enhanced while a low amount of heat was produced. Thus the energy consumption was reduced and the temperature of materials did not rise [13]. As shown in Table 1, the drying time, specific energy consumption of apple tissue that pretreatment by PEF was reduced by 22.84%, 16.55%, respectively, over the results of the untreated samples at the temperature of 75℃.

The rehydration characteristics of the dried product were used as a quality index because they could indicate the physical and chemical changes of samples during drying. With the increasing of the drying temperature, the rehydration ratio decreased, while the rehydration ratio of untreated samples was 4.71 at the temperature of 75℃. This is mainly because of PEF treatment could increase the cell membrane permeability, improve the drying rate, and avoid the phenomenon of collapse, it can short the drying time and increase the rehydration ratio to some extent.

| Temperature (℃) | PEF treatment | Untreated |
|-----------------|---------------|-----------|
| 65℃             | 75℃           | 85℃       | 75℃       |
| Drying time (h) | 7.38          | 6.25      | 6.02      | 8.10      |
| Specific energy consumption (kJ·g\(^{-1}\)) | 329.03 | 300.75 | 289.86 | 360.41 |
| Rehydration ratio | 7.55       | 7.51      | 6.09      | 4.71      |

The table refers to the board temperature in the sublimation drying processing.

3.2 Influence of different drying conditions on macroscopic and microscopic structure

The macroscopic and microscopic structure of the dried apple under different drying conditions were presented in Figure 1 and Figure 2. After the pretreatment of the electric field, there was no collapse of the apple sample, the macroscopic structure of the sample was well preserved. Based on the obtained results, there were no differences in macroscopic structure between Figure 1A and Figure 1B.
Microscopic structure can also observe the same result according to the Figure 2A and Figure 2B. Compared to the appearance of the dried apple with the board temperature of 65°C and 75°C, the apple sample, pretreated by PEF, appeared a slight collapse phenomenon at the board temperature of 85°C (Figure 1C). Therefore, the percentage of the collapsed apple have increased with the process temperature. However, the macroscopic structure of untreated sample appeared an obvious collapse phenomenon at the board temperature of 75°C as shown in Figure 1D. The sample structure collapsed, dented inward, and there is hardening phenomenon on the surface of the samples.

After the electric field treatment, the permeability of the cell membrane is increased, the water transmission speed increased, and the drying rate improved. After freezing, deformation of the cells occurs and some cell wall was disrupted. The cell breakage was presumably caused by ice crystals. During the sublimation stage, the orderly polyhedral arrangement was lost and more cell wall was disrupted. In desorption stage, the cells were found to be further damaged [14]. It was found that there were no significant changes in cell structure between Figure 2A and Figure 2B. Due to the percentage of the collapsed apple have increased with the process temperature, when the board temperature was 85°C, the microstructure of apple tissue occurred the collapse and shrink (Figure 2C). This means that the drying temperature of 85°C exceeded the collapse temperature of apple that pretreated by PEF. Therefore, when the drying temperature greater than the collapse temperature, it will affect the appearance of drying samples, meanwhile, it also affect the final moisture content, and the dried samples were not beneficial for storage, and poor in rehydration.

![Figure 1](image)

Figure 1. The appearance of the dried apple under different drying conditions. Photographs show the appearance of dried apple during sublimation drying processing. (A) 65°C, 40 Pa, PEF treatment, (B) 75°C, 40 Pa, PEF treatment, (C) 85°C, 40 Pa, PEF treatment, (D) 75°C, 40 Pa, untreated.
Figure 2. Microstructure of the dried apple under different drying conditions. Photographs A, B, C, D show cross-sections of dried apple prepared by (A) 65°C, 40 Pa, PEF treatment, (B) 75°C, 40 Pa, PEF treatment, (C) 85°C, 40 Pa, PEF treatment, and (D) 75°C, 40 Pa, untreated, respectively.

In Figure 2D, the untreated sample had an evident collapse phenomenon, the structure had a relative integrity, but the cells had obvious folds. During the vacuum freeze drying processing, the apple tissue had low cell permeability. With the increase of temperature, moisture evaporated from their surfaces, and when heating plate provided heat more than the heat that needed for moisture diffusion. The moisture could not evaporate in time, which led to the collapse phenomenon and the drying rate decreased. According to the results of experiment and above analysis, the apple collapse temperature was 75°C after PEF treatment.

3.3 Influence mechanism of PEF treatment on drying apple
According to the drying aspect, PEF treatment can accelerate the moisture evaporate and avoid collapse. During the drying processing, water movement will occur from areas of high water potential to areas of low water potential, fruits and vegetables cellular structure acts as a semi-permeable membrane, and the water potential on both sides of the semipermeable membrane determines the direction of water diffusion. Therefore, it is very important to increase the water potential difference between the intracellular and extracellular for improving the drying rate of fruits and vegetables. After PEF applied, cell membranes with short duration (µs – ms level) could form reversible or irreversible electroporation on biological cell membranes to reduce the influence of the interaction among the molecules, and the free-water can move to the surface, hence, PEF treatment can accelerate the transport process of water molecules, and avoid the collapse phenomenon.

From the perspective of mechanics, the collapse phenomenon is mainly caused by the change of mechanical properties and micro-structure of fruits and vegetables. The water vaporization was enhanced remarkably by PEF, which could make the material even drying and uniform force, so the final material is completely when dried, the skeleton structure of the material is preserved. However,
the untreated samples, due to the low permeability of the cell membrane, with the influence of the pressure of the freeze-drying chamber and thermal stress, the cells appeared slight collapse. Nevertheless, this collapse phenomenon resisted the further diffusion of water molecules, and then led to a significant collapse. It is precisely because of this cumulative effect of the collapse phenomenon, the drying rate was decreased, drying time was prolonged, and the residual water in the cells is difficult to evaporate, which is not favorable for the storage of fruits and vegetables.

4. Conclusions

The vacuum freeze drying experiment was performed on apples with different drying temperatures and the micro-structure was analyzed by SEM. The results showed that when the board temperature was set to 75°C during sublimation processing, the drying time of dried apple after PEF treatment was 6.25 h, the specific energy consumption was 300.75 kJ·g⁻¹ and the rehydration ratio was 7.51, meanwhile, the cell structure was well preserved. When the board temperature was set to 85°C, the sample pretreated by PEF appeared a collapse phenomenon. However, the untreated sample, when the board temperature was set to 75°C during sublimation processing, the drying time was 8.10 h, the specific energy consumption was 360.41 kJ·g⁻¹, and the rehydration ratio was 4.71. Moreover, the microstructure of apple tissue occurred the collapse and shrink, and the skeleton structure was lost. Therefore, the collapse temperature of the apple after PEF treatment was 75°C.

The influence mechanism of PEF treatment on collapse phenomenon was discussed from the perspective of moisture migration and mechanics. The results showed that the PEF can not only change the permeability of the cell membrane, but also change the water potential difference between the intracellular and extracellular., so as to PEF treatment can improve the diffusion rate, and increase the collapse temperature. Therefore, the board temperature should below the collapse temperature of the fruits and vegetables to increase the drying rate of the fruits and vegetables without destroying the quality during sublimation processing, the board temperature should be as high as possible at the maximum allowable temperature of 85-95°C during the desorption processing.

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