

# Freeze-Drying

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Freeze-dried materials are especially recommended for the production of spices, coffee, dried snacks from fruits and vegetables and food for military or space shuttles, as well as for the preparation of food powders and microencapsulation of food ingredients.

freeze-drying

grinding

osmotic dehydration

pulsed electric field

ultrasound

high hydrostatic pressure

## 1. Introduction

Drying is a commonly used process to extend the shelf life of food. The most popular method of food dehydration is hot air drying, in which the food material is exposed to a stream of hot air. This method is simple and relatively cheap, but the quality of products obtained after drying is often significantly lower <sup>[1]</sup>. Freeze-drying (FD), also called lyophilization, is among the best methods of food preservation. Moreover, the FD technology is the most widely used for the preservation of bacteria for producing starters and probiotics <sup>[2]</sup>. At 0 °C and pressure of 611.73 Pa, the three states of water, namely vapour, liquid and ice, occur in aggregation <sup>[3]</sup>. This state of equilibrium is called the triple point. Below this point, the removal of water from the material can only occur as a result of sublimation <sup>[4]</sup>. Such a phenomenon is possible under adequate temperature and pressure (below the triple point to enable the conversion of ice into vapour), when water molecules have enough energy to break free from the frozen material, but the conditions cannot support the formation of a liquid <sup>[5][6]</sup>. Vacuum FD process is commonly carried out at a low temperature (shelf temperature below 50 °C) <sup>[7]</sup> and low pressure (below the vapour pressure at the ice surface). Typically, the vacuum levels applied in the FD range between 7 and 70 Pa <sup>[8][9][10][11]</sup>. Vacuum FD is especially recommended for delicate, thermal-sensitive and high-value food, the physical and nutritional properties of which should be maintained <sup>[12]</sup>. The absence of liquid water, low oxygen access in the drying chamber and application of low temperatures result in dried products of excellent quality <sup>[13]</sup>. In general, FD involves three stages: freezing, primary drying and secondary drying. The steps of the FD process were described in a recent study <sup>[14]</sup>. FD can also be performed under atmospheric pressure at a low-temperature range (−30 to −60 °C) with low-humidity air <sup>[15][16]</sup>. However, such a process is usually very slow and takes up to three times longer duration than vacuum FD <sup>[6]</sup>.

Lyophilization allows almost complete removal of water from food <sup>[17]</sup>. In industrial conditions, freezing is mostly performed in a lyophilizer, whereas in the laboratory scale, food is often frozen in a refrigerator <sup>[5][18][19]</sup>. The rate of freezing significantly influences ice formation and determines the drying rate. A faster freezing rate results in the

formation of small ice crystals. The size of ice crystals has a considerable impact on lyophilization. Sublimation of fast-frozen food, with small-sized ice crystals, occurs rapidly in the first drying period but is slower in the second period of lyophilization [17].

FD is also widely used in the pharmaceutical and cosmetic industries [20][21]. Because of its high costs (up to five times higher than hot air drying [22]), this process is mainly recommended for the preservation of heat-sensitive materials [6]. It is also widely used for the microencapsulation of bioactive compounds of food [23][24]. The reduction in FD costs with high-quality products is still considered a challenge. However, adequate food pretreatment can significantly decrease the energy consumption associated with FD [25][26][27] and improve the quality of dried food [28][29]. FD allows obtaining products of very good quality, with a low final moisture content of 1–4% [5]. The obtained materials are brittle and easy to grind, and therefore, FD can be used to produce powders from various biological substances [30].

Pretreatment of food before drying serves two purposes: reduces the drying time and improves the quality of the dried material. This review aims to point out the recent trends in the pretreatment of food before FD and show how the different methods of pretreatment influence the properties of the dried materials and drying rate.

## 2. Pulsed Electric Field

The pulsed electric field (PEF) method is based on the permeabilization (electroporation) of cell membranes when electric pulses (usually ranging from 100–300 V·cm<sup>-1</sup> to 20–80 kV·cm<sup>-1</sup>) are used in a short time (from a few nanoseconds to a few milliseconds) [31]. When PEF is applied for food processing, the electric pulses induce the plasmolysis of biological cells [28]. PEF is a non-thermal technology, which is widely used to reduce the content of microorganisms in food [32]. This method can also be used for pretreatment of food before drying [33]. Generally, PEF enhances the quality of dried products [34] and accelerates the drying rate [35][36][37]. In recent years, several works have studied the effects of PEF on the FD process. Lammerskitten et al. [38] studied the influence of PEF and FD process in apples and found that this method of pretreatment reduced the drying time by about 25% and increased the rehydration capacity of freeze-dried apples. Similarly, Wu and Guo [39] found that PEF decreased the FD time of apples by about 23% in comparison to apples dried without pretreatment. Additionally, Parniakov et al. [40] showed that PEF pretreatment preserved the shape of freeze-dried apples and increased their porosity by 86 times. In another study [35], PEF was used for the pretreatment of red bell pepper and strawberries before FD. The authors of the study found that PEF pretreatment increased the rehydration capacity of the dried material by up to 50% while firmness was reduced by up to 60% [35]. Bai and Luan [41] found that PEF reduced the drying time (by about 16%) and increased the rehydration ratio of sea cucumber. However, taking into account the possibility of using PEF as a food pretreatment method before FD, the number of publications on this topic is limited, and the use of PEF should be more extensively studied as a pretreatment for different materials before FD.

## 3. Ultrasound

US technology is widely used for enhancing the rate of different processes in the food industry, especially cutting and slicing, filtration, freezing and crystallization, thawing extraction, pickling and drying [42]. Application of US for drying accelerates this process significantly [43]. The effect of US is mainly mechanical and not thermal. The use of US generates surface tensions in capillaries, as a result of which micro-channels are formed and the loss of water from the sample during drying can occur more easily [11]. Moreover, US improves the freezing process by increasing the size of ice crystals formed before FD [44][45]. In particular, US with a power of  $1 \text{ W}\cdot\text{cm}^{-2}$  and a frequency of 20–100 kHz is recommended for enhancing the drying rate of food [41][46][47]. In addition, US can be used independently as a method of food dehydration, especially in the case of heat-sensitive raw materials [48] because of the moderate increase in the temperature of dried products in comparison to other techniques [47][49]. US can also be used as a pretreatment method before FD. Xu et al. [50] used a US-freeze-thawing pretreatment to improve the FD efficiency of okra, and found that in okra pretreated with this method the retention of bioactive compounds was the highest and drying time was reduced. Merone et al. [47] applied US during atmospheric FD of apples, carrots and eggplants and observed that the use of US decreased the FD energy by up to 50% and reduced the drying time by up to 70%. Similar results were obtained by another group of authors [51] when they used US before FD in sweet potato. They found that the reduction in drying time increased with the increase in the power of US. In addition, they found that the US-treated samples showed higher hardness and fracturability after drying. Colucci et al. [49] studied the influence of US intensity on the antioxidant potential of eggplants and found that US caused no destructive effect on this parameter in freeze-dried eggplants. Similar results were observed by Zhang et al. [11] when US was used before vacuum FD in strawberry chips. Another team of authors studied the influence of US on the FD kinetics and quality of carrots [52]. They noted that as the power of US increased the drying time of carrot slices decreased from 20.7% to 23.7%. Importantly, US caused an increase in the content of  $\beta$ -carotene (from 22.7% to 32.0%) and had no negative influence on the sensory scores of dried products. Ren et al. [53] showed that US pretreatment of onions before FD increased their content of phytochemicals from 1% to 20% (flavonoids, quercetin, phenolic compounds) and enhanced the antioxidant activity of dried onions. However, prolonged sonication had a deleterious effect on these compounds and the antioxidant activity of the product. Schössler et al. [54] applied US throughout the FD process of red bell pepper and found that US increased the temperature of pepper and decreased the drying time by about 12%. The quality of the dried product (rehydration, colour, ascorbic acid content) after US-assisted FD did not differ significantly in comparison to pepper dried without US. Another team [55] studied the influence of US-assisted method on the atmospheric FD process of orange peel and revealed that US significantly accelerated the drying process. The FD time was decreased by about 57% without any effect on the functional properties of the fibre in the peel. Other researchers [56] used US as a method of pretreating quince slices before FD. They reported that US caused a decrease in the rate of shrinkage and the hardness of quince (by about 30%), whereas the rehydration ratio was increased by about 50%. Importantly, the total phenolic content and antioxidant activity of freeze-dried quince were higher when US was used before dehydration. The best-quality dried slices were obtained when the time of US pretreatment was 20 min. Carrión et al. [57] carried out atmospheric FD in button mushrooms (*Agaricus bisporous*) with the assistance of US. They found that the drying time was reduced by about two times and three times when the power of US was 12.3 and 24.6  $\text{kW}\cdot\text{m}^{-3}$ , respectively. Moreover, the use of US with a power of 24.6  $\text{kW}\cdot\text{m}^{-3}$  decreased the hardness and chewiness of rehydrated mushrooms but caused about a twofold increase in the rehydration time of dried A.

*bisporous*. Additionally, the US-assisted atmospheric FD decreased the lightness and increased the redness of mushrooms. The presented data show that US can significantly accelerate the drying process and enhance the quality of dried products. However, the adequate power of US and time of pretreatment have to be optimized for different products.

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