

Review

Recent Trends in Pretreatment of Food before Freeze-Drying

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Abstract: Drying is among the most important processes and the most energy-consuming techniques in the food industry. Dried food has many applications and extended shelf life. Unlike the majority of conventional drying methods, lyophilization, also known as freeze-drying (FD), involves freezing the food, usually under low pressure, and removing water by ice sublimation. 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. Although the FD process allows obtaining dried products of the highest quality, it is very energy- and time consuming. Thus, different methods of pretreatment are used for not only accelerating the drying process but also retaining the physical properties and bioactive compounds in the lyophilized food. This article reviews the influence of various pretreatment methods such as size reduction, blanching, osmotic dehydration and application of pulsed electric field, high hydrostatic pressure or ultrasound on the physicochemical properties of freeze-dried food and drying rate.

Keywords: freeze-drying; size reduction; blanching; osmotic dehydration; pulsed electric field; high hydrostatic pressure; ultrasound; drying rate; quality

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 [1]. 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 [2]. At 0 °C and pressure of 611.73 Pa, the three states of water, namely vapour, liquid and ice, occur in aggregation [3]. 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 [4]. 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 [5,6]. Vacuum FD process is commonly carried out at a low temperature (shelf temperature below 50 °C) [7] 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 [8–11]. Vacuum FD is especially recommended for delicate, thermal-sensitive and high-value food, the physical and nutritional properties of which should be maintained [12]. The absence of liquid water, low oxygen access in the drying chamber and application of low temperatures result in dried products of excellent quality [13]. In general, FD involves three stages: freezing, primary drying and secondary drying. The steps of the FD process were described in a recent study [14]. FD can also be performed under atmospheric pressure at a low-temperature range

(−30 to −60 °C) with low-humidity air [15,16]. However, such a process is usually very slow and takes up to three times longer duration than vacuum FD [6].

Lyophilization allows almost complete removal of water from food [17]. In industrial conditions, freezing is mostly performed in a lyophilizer, whereas in the laboratory scale, food is often frozen in a refrigerator [5,18,19]. 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 [25–27] 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. Pretreatment of Food before FD

2.1. Size Reduction and Pureeing

One of the oldest methods of food pretreatment applied before drying is size reduction. This is carried out before FD and involves classical cutting of material into pieces [31] or grinding into puree [32]. Such processes increase the surface areas of the dried materials and accelerate the drying rate [33] while changing the properties of the dried products [34]. Taskin [10] studied the FD process of whole and ground black chokeberry fruits. He found that the time and energy consumption of the process was reduced more than twofold by chokeberry puree drying in comparison to whole-fruit dehydration. Venkatachalapathy and Raghvan [33] found that freeze-dried puree made from strawberries had a better quality than the microwave-dried puree. Another study showed that freeze-dried orange puree [35] is ideal for obtaining freeze-dried crunchy snacks. Tylewicz et al. [36] analysed freeze-dried kiwi puree for the production of fruit bars and found that the products obtained by FD had a higher total phenolic and flavonoid content than those obtained by air drying. Rudy et al. [32] studied the FD process of whole and pulped cranberries and proved that pureeing of cranberries before drying reduced the FD time by about half and resulted in a dark red-coloured final product with a higher antioxidant capacity in comparison to the FD of whole fruits. Dziki et al. [18] explored the FD process of whole and pulped kale leaves and observed that pulping decreased the drying duration by 40%. This effect was the most visible when a temperature of 20 °C was applied during FD. Moreover, dried kale obtained from freeze-dried puree was characterized by lower lightness and hue angle, but higher browning index in comparison to that obtained from whole leaves. On the other hand, whole-dried leaves had higher chlorophyll content than pureed and dried leaves. Importantly, pulping exerts little or no influence (depending on the drying temperature) on the total phenolic content and antioxidant activity. A study [37] analysed the FD process of sliced and whole cranberries and revealed that the drying time was decreased about fourfold when strawberries were sliced into 5 mm samples, and about threefold when the slices had a thickness of 10 mm. Figure 1 shows the relation between water content and FD time of whole bananas and banana puree. The same mass (500 g) of banana was freeze-dried at 60 °C according to the method presented in the study by Dziki et al. [18]. The laboratory freeze-dryer was

equipped with a system for recording the mass changes occurring during FD (Figure 2). For banana puree, about fourfold less FD time was observed compared to whole bananas. This indicated that FD of puree not only reduces the duration of the process but also the energy requirements. Therefore, pureed food is often subjected to FD instead of whole or sliced samples of raw materials [34,38,39]. However, the number of studies concerning such a method of food pretreatment before FD is limited (Table 1).

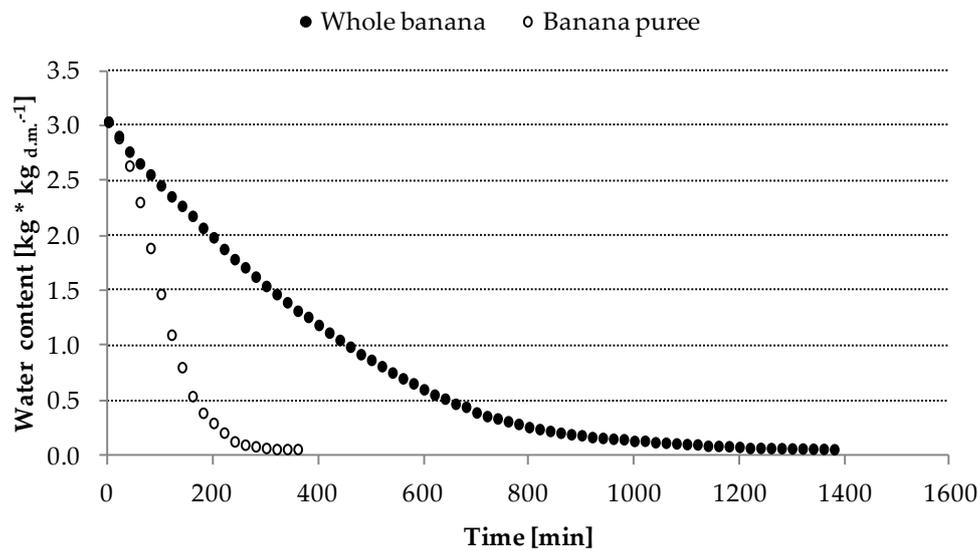


Figure 1. Relation between Freeze-drying (FD) time and water content in whole bananas and banana puree, (temperature of plates: 60 °C, pressure in the drying chamber: 100 Pa); d.m.—dry mass.



Figure 2. Measuring stand for the recording of mass changes of sample during FD.

Table 1. Methods of pretreatment of food before FD, drying conditions and effect of pretreatment.

Raw Material	Method and Conditions of Pretreatment	FD Conditions	Main Effect	References
Black chokeberry	SR *, cutting and puree preparation	−50 °C, 52 Pa	Reduced DT and energy	[10]
Cranberries	SR, puree	30, 50 and 70 °C, 52 Pa	Reduced DT, changed colour, enhanced AA	[32]
Kale	SR, puree of leaves	20, 40 and 60 °C, 52 Pa	Reduced DT, darker leaves	[18]
Strawberries	SR, slices of strawberries 5 mm and 10 mm	30, 40, 50, 60 and 70 °C	Reduced DT	[37]
Strawberries	OD, fructose, 50 °C, 60°B, 3 h	Not included	Reduced DT, increased rehydration, better structure	[40]
Strawberries	OD, fructose+ calcium chloride, fructose+ calcium lactate, (fructose 50°B and calcium chloride or calcium lactate (1% <i>w/w</i>))	−100 °C, 200 Pa	Higher rehydration capacity, better colour and texture, preserved structure	[41]
Pumpkin	OD, syrup solution (glucose equivalent DE containing 663 g/kg of sugars), 20 °C, 3 and 20 h,	10 °C, 63 Pa	Decreased water activity and water content in freeze-dried samples	[42]
Apples	OD, 60° B sucrose or sorbitol solutions at 60 °C, 8 h	From −40 to −45 °C, 133 Pa	Reduced DT but decreased TPC and AA	[43]
Chinese yam	OD, Sucrose with concentrations of 30%, 40% and 50% <i>w/w</i>	Pulsed-spout microwave FD, 50 °C, 80 Pa; microwave power 2 W/g; duration of spouting 0.1 s, with an interval of 20 min.	Decreased drying rate and increased rehydration ratio	[44]
Raspberries	OD, Fruit/sugar (sucrose) ratio: 1.27 for dry infusion and 0.36 for wet infusion.	−55 °C, 4 Pa.	Decreased porosity, volume and rehydration ratio	[45]
Apples	OD, two 60% solutions: sucrose and sucrose+chokeberry juice concentrate (1:1), 40 and 60 °C, for 30 and 120 min.	25 °C, 100 MPa	Increased water activity and changed colour	[46]
Elephant foot yam	BL, sodium metabisulphite solution, 75 °C	30 °C	Changed physic-chemical and functional properties	[47]
Pumpkin	BL, water (100 °C, 1 min)	10 °C, 103 Pa	Reduced DT	[42]

Table 1. Cont.

Raw Material	Method and Conditions of Pretreatment	FD Conditions	Main Effect	References
Guava fruit	BL and ultrasounds (37 kHz, 240 W, 65 °C, 5 and 10 min)	40 °C, 75 Pa	Reduced DT, better colour preservation, decreased TPC and AA	[26]
Tomato	BL, steam (100 °C, 5 min)	−56 °C, 8 Pa	Increased TPC, lycopene and β-carotene content but decrease ascorbic acid content	[48]
Chinese yam fruit	BL, water (85 °C, 1 min)	−40 °C, 100 Pa, with assistance of microwaves (0.225 W/g)	Increased porosity of dried samples	[49]
Broccoli	BL, microwave (800 W, 2 min)	from −45 °C to −50; <20 Pa	Increased content of phenolics and chlorophyll	[50]
Red pepper	BL, water (90 °C, 1 min); microwave (1.5 min, 650 W)	20, 40 and 60 °C, 62 Pa	Reduced DT, but decreased TPC and AA	[51]
Onion	BL, water (70 °C, 1, 3 and 5 min)	temperature of FD no included, 6 Pa	Reduced content of bioactive compounds and AA, increased lightness and yellowness	[52]
Apples	BL, water (90°, 1 min)	−20 °C, 1 h, −10 °C, 1 h, 0 °C, 1 h, 10 °C, 2 h, 20 °C for 2 h, 30 °C, 2 h, finally 40 °C	Reduced DT, decreased ascorbic acid content, increased lightness	[53]
<i>Thunbergia laurifolia</i> leaves	BL, water (100 °C, form 1 to 7 min); steam-microwave BL (900 W, form 1 to 7 min)	35 °C, 15 Pa	Increased content of bioactive compounds, higher rehydration ratio, better preservation of colour	[54]
Papaya leaves	BL, water (96–98 °C, 1.5 min)	−100 °C, 10 Pa	Increased TPC	[55]
Sea cucumber	PEF, (optimum parameters: 22.5 kV for 70 Hz, 5 min)	Not included	Reduced DT, increased rehydration ratio	[56]
Red bell pepper and strawberries	PEF, the field strength 1.0 kV/cm, the number of pulses was 20 and 200, treatment time 2.0–28.6 ms	−4 ± 2 °C and −40 ± 2 °C, 12,500 Pa, time 72 h	Reduced firmness, higher rehydration capacity	[57]

Table 1. Cont.

Raw Material	Method and Conditions of Pretreatment	FD Conditions	Main Effect	References
Apple	PEF, pulse duration of 40 ms and pulse width of 10 ms. The interval between the pulses 0.5 s (2 Hz). Energies of 0.5 and 1 kJ/kg and a field strength of $1.07 \text{ kV}\cdot\text{cm}^{-1}$	40 °C, 100 Pa.	Reduced DT, increased AA	[58]
Apple	PEF, pulses strength 1000, 1250, 1500 $\text{kV}\cdot\text{cm}^{-1}$ Pulse duration 60, 90, 120 μs , Pulses number 15, 30, 45	I steep 70 °C, 40~45 Pa II steep 90 °C, 30~35 Pa	Reduced DT, increased hydration capacity	[59]
Apple	800 $\text{V}\cdot\text{cm}^{-1}$, 0.1s	40 °C, 1000 Pa	Better preservation of shape and increased porosity	[60]
Potato	PEF, electric field strength 1000, 1250, pulse width, pulses number were 60, 90, and 120 μs , and 1500 $\text{V}\cdot\text{cm}^{-1}$; pulse duration 500 ms.	75 °C, 40–45 Pa.	Reduced DT	[61]
Okra	US, 40 kHz, the power density is 25 W/L, and the ultrasound time is 30 min, samples were frozen -20 °C for 24 h, then thawed by using ultrasonics	Mbar, main drying at 18 °C, final drying at 20 °C, vacuum pressure of 52 Pa	Reduced FD time and total energy consumption, increased AA activity and lower degradation of chlorophyll	[62]
Carrot root	US, 45 kHz, the ultrasound power 150, 240, and 300 W, treatment time 30 min.	60 °C, 80 Pa	Reduced DT and increased level of β -carotene	[63]
Apple, carrot, eggplant	US intensity $10.3 \text{ kW}\cdot\text{m}^{-3}$ and $20.5 \text{ kW}\cdot\text{m}^{-3}$, transducer, radiator with 21.9 kHz average frequency,	Atmospheric FD, -19 °C , air velocity of 1, 2 and $4 \text{ m}\cdot\text{s}^{-1}$	Reduced DT and FD energy	[64]
Red bell pepper	US, 76, 90 and 110 W,	vacuum pressure of 46 Pa, drying at 10 °C	Reduced DT	[65]
Eggplant	US, 25 and 50W, 21.9 kHz, US 0.3 and $20.5 \text{ kW}\cdot\text{m}^{-3}$	$-5, -7.5, -10\text{ °C}$, air velocity $2.5 \text{ m}\cdot\text{s}^{-1}$, atmospheric FD	Reduced DT no destructive effect on AA	[66,67]
Sweet potato	US at 25 °C, 30 kHz. Power of ultrasound: 200, 400 and 600 W, respectively (duration 30 min)	Drying temperature was 50 °C, pressure 80 Pa.	Reduced DT and improved colour and texture	[68]
Strawberry chips	US, 200 W, 40 kHz, duration 25 min	4 °C, 10 Pa, for 20 h	and enhanced antioxidant properties	[11]

Table 1. Cont.

Raw Material	Method and Conditions of Pretreatment	FD Conditions	Main Effect	References
Orange peel	US, 20.5 kW/m ³	−10 °C, atmospheric pressure	Reduced DT and do not change the fibre functional properties	[69]
Quince	US, 28 kHz, 50 W, time 10, 20, and 30 min	−25 °C, 48 Pa	Reduced hardness and shrinkage, increased rehydration ratio and AA	[70]
Button mushrooms	US, 12.3 and 24.6 kW·m ^{−3}	−10 °C, 2 m·s ^{−1}	Reduced DT and lightness, decreased hardness and chewiness, of rehydrated samples	[71]
Strawberries	HHP, from 0 to 250 MPa, 10 min	−50 °C, 10 Pa	Increased redness, lightness, and total content of anthocyanin, reduced DT	[11]
Jabuticaba (peel and seed)	HHP, 200, 350 and 500 MPa; 1; 5.5 and 10 min	−50 °C, 65 Pa	Increased content of TPC and higher AA	[72]
Shrimp	HHP, 550 MPa, 10 min, prefrozen at −80 °C (3 h)	Primary drying: −35 °C, 10 Pa for 3 h, secondary drying at 50 °C, 10 Pa for 19 h	Acceleration of moisture migration, improved drying efficiency	[73]
Garlic	HHP, 400, 500, 600 MPa, 5 min	Not included	Inactivation of microorganism, increasing the content of TPC and AA, less pungent odour, more bright and less yellow powder	[74]
Korean ginseng	HHP, 600 Pa, 5 min; UVT (254 nm, 35 W, 25 mW·cm ^{−2} , 10 min) and HHP combination (300, 400, 500, 600 Pa, 5 min)	−30 °C, 37 Pa for 20 h and −76 °C, 1 Pa for 2 h	Inactivation of microorganism, increasing the content of TPC but no change in AA and colour	[75]
Edible flowers	HHP, Pansies: 75, 150 and 450 MPa, 5 and 10 min, Centaurea: 75, 100, 200 and 300 MPa, 5 min, Borage: 75 MPa, 1 and 5 min, Camellia: 75 MPa, 1 and 5 min, and 100 MPa for 5 min	Not included	Increased level of bioactive compounds	[76]

* SR—size reduction, OD—osmotic dehydration, BL—blanching, PEF—pulsed electric field, US—ultrasound, HHP—high hydrostatic pressure, DT—drying time, TPC—total phenolics content, AA—antioxidant activity, FD—freeze-drying.

2.2. Blanching

Blanching is an important and widely known pretreatment method prior to vegetables and fruits processing. The main aim of this process is to inactivate the enzymes, shorten the drying time and enhance the quality of dried products [77]. Furthermore, blanching allows removing pesticide residues and toxic substances from food [78] while increasing microbiological purity [79]. Several methods of blanching are used, such as hot water blanching [42], steam blanching [80], superheated steam impingement blanching [81], microwave blanching [82], ohmic blanching [83], infrared blanching [84] and vacuum-steam pulsed blanching [85]. Hot water blanching is the simplest and the most popular method applied during food processing [86]. Numerous works have discussed the possibility of performing pretreatment of food before FD by using blanching. For instance, Suriya et al. [47] blanched elephant foot yam (*Amorphophallus paeoniifolius*) slices with sodium metabisulphite solution before FD and found that blanching changed the physicochemical and functional properties of freeze-dried and powdered yam. The authors noted that amylose content and water solubility increased, whereas foam capacity and foam stability decreased in comparison to yam flour obtained from unblanched plants. Moreover, blanching significantly changed the pasting properties of yam flour and increased the hardness of gels resulting from freeze-dried and powdered yam. Another study [42] investigated the effect of blanching of pumpkin (*Cucurbita maxima*) on the course of FD and properties of the dried product. The authors of the study blanched the osmotically dehydrated pumpkin in water and observed that blanching facilitated the removal of water during FD. They explained this phenomenon by the damage of the cell walls caused by pumpkin pretreatment. In addition, the freeze-dried material was characterized by lower moisture content in comparison to unblanched pumpkin. Jorge et al. [48] carried out water blanching of tomato before FD and found that blanching increased the content of phenolics, lycopene and β -carotene but decreased the content of ascorbic acid and sugars. Contrary to this report, Krzykowski et al. [51] observed that water blanching and microwave blanching of red pepper slightly but significantly reduced the total phenolic content and antioxidant activity of dried and powdered pepper. Importantly, they observed that drying time was reduced by about 30% for blanched fruits. Another group of authors [26] studied the influence of ultrasound (US)-assisted blanching on the FD process in guava. They proved that such kind of pretreatments reduced, to a large extent, the peroxidase activity and, to a lower extent, the polyphenol oxidase activity. The time of FD and the final moisture content of the dried product were significantly decreased after US-assisted blanching. Additional pretreatment had a positive influence on colour retention by dried fruits. However, blanching reduced the total phenolic content (about 15%) in dried fruits as well as the antioxidant activity. Interestingly, higher antioxidant activity was observed when blanching was performed for a longer time. Phahom et al. [54] used water blanching and steam-microwave blanching for medicinal herb leaves of laurel clockvine (*Thunbergia laurifolia* Linn.) prior to FD. They showed that blanching increased the rehydration ratio of freeze-dried leaves due to the increase in intercellular spaces and cell permeabilization. Blanching also increased the content of caffeic acid and quercetin in freeze-dried leaves (from 0.73 to 1.63 mg·g_{d.m.}⁻¹ and from 5.92 to 7.86 mg·g_{d.m.}⁻¹, respectively). Importantly, the antioxidant capacity of dried leaves was found to be increased after blanching and the colour of freeze-dried product was better preserved in comparison to leaves that were freeze-dried without pretreatment. Another study [55] analysed the effects of blanching on the drying process in papaya (*Carica papaya* L.) leaves. The results showed that blanching significantly increased the total phenolic content in freeze-dried leaves (about 20%) but had only a slight influence on colour coordinates and the content of ascorbic acid in dried samples. Wang et al. [49] observed that blanching of Chinese jam increased the porosity of freeze-dried material. Ferreira et al. [50] applied microwave blanching before the FD process in broccoli and found that blanching increased the content of phenolic compounds (about 50%) as well as chlorophylls (a and b; about 8%) during the extraction of dried broccoli. However, the process had no influence on broccoli drying time. Contrary to this study, Ren et al. [52] found that blanching onions in hot water (70 °C, 3 min) led to a decrease in the total phenolic and flavonoid contents (about 15%) in freeze-dried onions. Additionally, they showed that

longer blanching time decreased the total antioxidant activity of freeze-dried onions expressed as ferric reducing power but had no significant influence on DPPH (2,2-diphenyl-1-picrylhydrazyl) scavenging activity. Onions blanched after FD appeared more bright and yellow and contained less quercetin in comparison to freeze-dried control sample. Wang et al. [53] also found that blanching apples before FD significantly increased the lightness of the dehydrated product. Interestingly, the method of FD had a significant influence on the redness of freeze-dried apples. The products obtained after vacuum FD had several-fold decreased redness (from 6.72 for control FD sample to 1.83 for blanched apples), whereas in the case of plate freezing, a reverse relation was observed (redness increased to 8.33). In addition, blanching increased the porosity of freeze-dried samples and decreased the content of ascorbic acid (about 25%) and hardness of the freeze-dried samples (about 50%). The time of drying was about 10% shorter when blanching was followed by vacuum FD. Taking all these results into account (Table 1), it can be concluded that blanching poses different effects on the drying course and quality of freeze-dried products depending on the kind of raw material used and process conditions applied.

2.3. Osmotic Dehydration

Osmotic dehydration (OD) refers to the process of water removal from fruits and vegetables and increasing the solute content in food. During OD, water is removed from material mostly by flow through capillaries and diffusion. Different osmotic agents are used for OD based on the properties of the product [87]. The main aim of OD is to enhance the nutritional and sensory quality of fruits and vegetables [88]. This method of pretreatment also allows reducing the drying time [89] and decreases the drying cost, and thus has a positive impact on the environment [90]. On the other hand, OD may cause the loss of proteins, minerals and vitamins in the resulting product [6,91]. It involves relatively slow mass transfer and is influenced mainly by the permeability of cell membranes and cell structure [92]. After OD, the water activity of the product is reduced only to about 0.9, but the dried food should have a water activity of less than 0.6 [93]. Thus, OD is often applied as a pretreatment before other dehydration methods. However, only a limited number of studies have investigated the influence of OD on the FD process to date. Prosapio and Norton [40] studied the influence of OD on the FD process in strawberries. They compared four osmotic agents (maltodextrin, maltose, fructose and sucrose) and selected fructose for further experiments. Their results showed that OD caused about twofold reduction in drying time and significantly increased the rehydration ratio of freeze-dried fruits. Additionally, they found that the samples that underwent OD retained a better structure and higher force required to puncture than unprocessed freeze-dried strawberries. In another work, the same authors [41] used US-assisted OD as a pretreatment method before FD in strawberries. Fructose along with a firming agent was used as an osmotic agent, while calcium chloride and calcium lactate were used as firming agents. The pretreatment applied was found to enhance the rehydration capacity of the freeze-dried samples and improve the texture of strawberries after rehydration. Moreover, the colour and structure were better protected in the freeze-dried sample in comparison with the non-pretreated fruits. Importantly, the use of US caused a reduction in the OD time. Another group of authors [42] studied the influence of OD on FD in pumpkin and found that a longer OD duration resulted in a decrease in water content and water activity in freeze-dried pumpkin. Cieurzyńska et al. [46] analysed the effect of OD performed before FD in apples. They used sucrose and chokeberry juice+sucrose (1:1) as osmotic agents. Their results showed that OD caused an increase in the water activity of freeze-dried fruits and significantly increased the total colour difference (ΔE) between the control and OD samples; in particular, when OD was performed by using chokeberry juice+sucrose, the ΔE was between 40 and 50. On the other hand, Assis et al. [43] freeze-dried osmotically dehydrated apples in sorbitol and sucrose solutions and found that the drying rate was slightly increased when sucrose solution was used as an osmotic agent and the colour of the dried fruits was better preserved. However, OD caused a several-fold reduction in the total phenolic content and antioxidant activity of freeze-dried apples. Chakraborty et al. [91] studied the effect of the FD process in kiwi followed by OD using sucrose with different concentrations. Their study showed that OD reduced the vitamin content

in fruits by about 10%. Another group of authors [44] applied pulsed vacuum OD before drying in Chinese yam, using sucrose with different concentrations as an osmotic agent. They observed that OD increased the drying rate by 8.4% and 16.8% compared to untreated yam. The highest increase was observed when osmotic solutions with a sucrose concentration of 40% were used. OD had a slight influence on the colour coordinates of dehydrated yam but decreased the dehydration rate. The lowest dehydration ratio was achieved when OD was performed with a 50% concentration of sucrose. The authors of the study explained this phenomenon by the fact that a high concentration of osmotic agent can cause shrinkage of tissues and decrease the porosity, leading to a decrease in the dehydration ratio. The study conducted by Sette et al. [45] revealed that OD performed using sucrose in raspberries resulted in a lower rehydration rate and a significant reduction in the firmness of freeze-dried fruits in comparison to the control samples. They explained this fact by the decrease in porosity and degradation of polysaccharides during OD, as a result of which plasticity and softness of tissues increased. Additionally, they found that OD samples presented a lower glass transition temperature and more reduced volume in comparison to the control fruits. According to the presented data, OD is an important process that can be used before FD with the aim of enhancing the quality of dried fruits as well as reducing the drying time (Table 1).

2.4. 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⁻¹ to 20–80 kV·cm⁻¹) are used in a short time (from a few nanoseconds to a few milliseconds) [94]. 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 [95]. This method can also be used for pretreatment of food before drying [96]. Generally, PEF enhances the quality of dried products [97] and accelerates the drying rate [57,61,98]. In recent years, several works have studied the effects of PEF on the FD process (Table 1). Lammerskitten et al. [58] 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 [59] found that PEF decreased the FD time of apples by about 23% in comparison to apples dried without pretreatment. Additionally, Parniakov et al. [60] showed that PEF pretreatment preserved the shape of freeze-dried apples and increased their porosity by 86 times. In another study [57], 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% [57]. Bai and Luan [56] 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 (Table 1), 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.

2.5. 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 [99]. Application of US for drying accelerates this process significantly [100]. 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 [101,102]. In particular, US with a power of 1 W·cm⁻² and a frequency of 20–100 kHz is recommended for enhancing the drying rate of food [64,66,103]. In addition, US can be used independently as a method of food dehydration, especially in the case of heat-sensitive raw materials [104] because of the moderate increase in the temperature of dried products in comparison

to other techniques [64,67]. US can also be used as a pretreatment method before FD. Xu et al. [62] 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. [64] 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 [68] 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. [67] 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 [63]. 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 β -carotene (from 22.7% to 32.0%) and had no negative influence on the sensory scores of dried products. Ren et al. [52] 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. [65] 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 [69] 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 [70] 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. [71] carried out atmospheric FD in button mushrooms (*Agaricus bisporus*) 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 kW·m⁻³, respectively. Moreover, the use of US with a power of 24.6 kW·m⁻³ decreased the hardness and chewiness of rehydrated mushrooms but caused about a twofold increase in the rehydration time of dried *A. bisporus*. 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 (Table 1). However, the adequate power of US and time of pretreatment have to be optimized for different products.

2.6. High Hydrostatic Pressure

The application of high hydrostatic pressure (HHP) is a non-thermal method of food preservation. In this process, water is used as a pressure medium, with pressure in the range of 100–1000 MPa [105,106]. Food processing by HHP results in the inactivation of microorganisms [107] and modifies the enzyme activity [108]. Moreover, this process retains the food shape and does not require the use of chemical additives [106]. HHP also increases the shelf-life of food and is recommended for heat-sensitive products [109]. During HHP pretreatment, the cells in the material are destroyed [110,111] and the metabolic reactions that are crucial for cell maintenance are inhibited [112]. These changes also have an influence on the drying rate and the properties of dried products [72,113,114]. Application of HHP increases the cell membrane permeability, enhances diffusion and increases the mass transfer during drying [28]. To date, only a few works have analysed the influence of HHP on the FD process.

For instance, Zhang et al. [115] studied the vacuum FD process of strawberries that were pretreated by applying a different HHP. In the microscopic images of freeze-dried strawberries, they observed microscopic holes or channels in the dried material pretreated by HHP and also noted the damaged tissue structure. In addition, they studied the microstructure of dried strawberries and observed that with the increase in the value of HHP the porosity of the product increased. The average pore areas changed from 22,864 μm^2 (control sample without HHP) to 49,581 μm^2 (250 MPa). This phenomenon accelerated the FD course. The authors found that such kind of pretreatment decreased the FD duration from 37 to 28 h. A reverse linear relationship was observed between HHP and drying duration. Additionally, as the value of HHP increased, the redness and lightness of the dried strawberries were also found to be increased. Most importantly, as the level of pressure increased, the content of anthocyanins increased from 3.72 mg cyanidin-3-glucoside equivalent (C-3-G equiv)/g for the control sample (no pretreatment) to 13.54 mg C-3-G equiv/g (HHP = 250 MPa) [115]. On the other hand, Pimenta Inada et al. [72] studied the FD process in jaboticaba (*Myrciaria jaboticaba*) peel and seeds and found that the HHP pretreatment was ineffective in increasing the total phenolic content and antioxidant capacity of jaboticaba powder. A value of pressure higher than 200 MPa resulted in a decrease in the total phenolic content (from 17% to 43%). Interestingly, FD at $-50\text{ }^\circ\text{C}$ and oven-drying at $75\text{ }^\circ\text{C}$ resulted in dried products with a similar total phenolic content, but with a different phenolic profile. Park et al. [74] used HHP for the manufacture of garlic powder by FD. They observed that pressurization of garlic before FD resulted in about a threefold decrease in the number of total aerobic bacteria from 4.22 log colony-forming unit (CFU)/g fw (for untreated garlic) to 1.52 log CFU/g fw (HHP = 600 Pa) in the final product. A similar tendency was found in the case of yeast and mould counts. Moreover, the authors showed that HHP increased the total phenolic content and antioxidant activity of freeze-dried garlic. Furthermore, the pungent fragrance of garlic powder was decreased after using HHP as a result of a decrease in alliinase activity and the content of diallyl disulfide. Moreover, after pressurization, the lightness of freeze-dried garlic powder was increased and yellowness was decreased. In another study [116], the FD process of ginseng paste (*Panax ginseng* Meyer) pretreated by HHP was studied. Additionally, before HHP, the authors used titanium dioxide-UVC photocatalysis (TUV) as a pathogen inactivation technique [75]. Their results [116] showed that the combined pretreatment with TUV and HHP had no influence on colour changes in freeze-dried ginseng paste and did not change the antioxidant activity (DPPH radical scavenging activity) but significantly reduced (about twofold) the microbiological contamination of ginseng powder. Importantly, an increase of about 70% in total phenolic content was observed. The authors also noted the strong anti-inflammatory effect of freeze-dried ginseng when the combination of UVT and HHP was used. A previous study [73] used HHP for shrimp pretreatment before FD. The authors of the study observed that HHP improved drying efficiency and moisture migration from the exterior to the interior part of shrimp. In addition, HHP improved the fragility of freeze-dried shrimp. Fernandes et al. [76] studied the effect of HHP on the properties of edible flowers and found that this pretreatment method significantly increased the total phenolic and flavonoid contents in freeze-dried flowers. However, this effect disappeared after 20 days of storage of flowers. To sum up, HHP can effectively influence the FD process of food. Most importantly, the pretreatment reduced the drying rate and induced the production of bioactive compounds in freeze-dried food (Table 1).

3. Conclusions

FD is among the best methods used for food preservation and is widely applied especially for the dehydration of fruits and vegetables. It allows obtaining high-quality dried products with shelf life extended for up to a few years. On the other hand, such a method of food preservation is energy consuming and requires expensive equipment. Moreover, FD can negatively affect the properties of some dried products. The present review indicates that adequate pretreatments can significantly reduce the drying time and improve the quality of freeze-dried products. In particular, non-thermal technologies such as pureeing, OD and the use of HHP, US or PEF can be used as pretreatment methods

before FD. These methods enhance the mass and heat transfer during dehydration and often positively affect the properties of dried products. However, some of them (HHP, US and PEF) are expensive. Importantly, the described pretreatments can be used before vacuum FD, as well as atmospheric FD which is very time consuming. Thus, future studies should focus on finding ways to accelerate the ice sublimation process without any loss in the quality of the product. Although much research has been carried out on increasing the rate of the FD process and preserving the nutritional and sensory properties of lyophilized food, the process is still considered a challenge, and studies are needed to optimize the conditions of pretreatment for different kinds of raw materials.

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