# Freeze-Drying

Subjects: Food Science & Technology
Contributor: Dariusz Dziki

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.

Keywords: freeze-drying ; grinding ; osmotic dehydration ; pulsed electic 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 [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][9][10][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][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$^{-1}$ to 20–80 kV·cm$^{-1}$) 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 tenses 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 W·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 β-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 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. 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.

## References


1. Ciurzyńska, A.; Lenart, A. Freeze-drying—Application in food processing and biotechnology—A review. Pol. J. Food Nutr. Sci. 2011, 61, 165–171.
2. Iaconelli, C.; Lemetais, G.; Kechaou, N.; Chain, F.; Bermúdez-Humarán, L.G.; Langella, P.; Gervais, P.; Beney, L. Drying process strongly affects probiotics viability and functionalities. J. Biotechnol. 2015, 214, 17–26.
3. Harguindeguy, M.; Fissore, D. On the effects of freeze-drying processes on the nutritional properties of foodstuff: A review. Dry. Technol. 2020, 38, 846–868.
4. Jiang, H.; Zhang, M.; Mujumdar, A.S.; Lim, R.X. Comparison of drying characteristic and uniformity of banana cubes dried by pulse-spouted microwave vacuum drying, freeze drying and microwave freeze drying. J. Sci. Food Agric. 2014, 94, 1827–1834.
5. Shukla, S. Freeze Drying Process: A Review. Int. J. Pharm. Sci. Res. 2011, 2, 3061–3068. [Google Scholar]
6. Bhatta, S.; Janezic, T.S.; Ratti, C. Freeze-drying of plant-based foods. Foods 2020, 9, 87.
7. Munzenmayer, P.; Ulloa, J.; Pinto, M.; Ramirez, C.; Valencia, P.; Simpson, R.; Almonacid, S. Freeze-drying of blueberries: Effects of carbon dioxide (CO2) laser perforation as skin pretreatment to improve mass transfer, primary drying time, and quality. Foods 2020, 9, 211.
8. Caglar, N.; Ermis, E.; Durak, M.Z. Spray-dried and freeze-dried sourdough powders: Properties and evaluation of their use in breadmaking. J. Food Eng. 2021, 292, 110355.
9. Vanbillemont, B.; Nicolaï, N.; Leys, L.; De Beer, T. Model-based optimisation and control strategy for the primary drying phase of a lyophilisation process. Pharmaceutics 2020, 12, 181.
10. Taskin, O. Evaluation of Freeze Drying for Whole, Half Cut and Puree Black Chokeberry (Aronia melanocarpa L.). Heat Mass Transf. Stoffuebertragung 2020, 56, 2503–2513.
11. Zhang, L.; Liao, L.; Qiao, Y.; Wang, C.; Shi, D.; An, K.; Hu, J. Effects of ultrahigh pressure and ultrasound pretreatments on properties of strawberry chips prepared by vacuum-freeze drying. Food Chem. 2020, 303, 125386.
12. Mustafa, I.; Chin, N.L.; Fakurazi, S.; Palanisamy, A. Comparison of phytochemicals, antioxidant and anti-inflammatory properties of sun-, oven- and freeze-dried ginger extracts. Foods 2019, 8, 456.
13. Ratti, C. Hot air and freeze-drying of high-value foods: A review. J. Food Eng. 2001, 49, 311–319.
14. Zhang, Z.; Liu, X.Y. Control of ice nucleation: Freezing and antifreeze strategies. Chem. Soc. Rev. 2018, 47, 7116–7139.
15. Tolstorebrov, I.; Eikevik, T.M.; Sæther, M. Influence of thermal properties of brown seaweeds (Saccharina Latissima) on atmospheric freeze-drying process in fluidized bed. Refrig. Sci. Technol. 2019, 2019, 2996–3003.
16. Nakagawa, K. Food drying at sub-zero temperature: Importance of glassy phase on product quality. Sci. Eng. Health Stud. 2018, 12, 125–137.
17. Oyinloye, T.M.; Yoon, W.B. Effect of freeze-drying on quality and grinding process of food produce: A review. Processes 2020, 8, 354.
18. Dziki, D.; Polak, R.; Rudy, S.; Krzykowski, A.; Gawlik-Dziki, U.; Różyło, R.; Miś, A.; Combrzyński, M. Simulation of the process kinetics and analysis of physicochemical properties in the freeze drying of kale. Int. Agrophysics 2018, 32, 49–56.
19. Hua, T.C.; Liu, B.L.; Zhang, H. Freeze-Drying of Pharmaceutical and Food Products; Woodhead Publishing: Great Abington, Cambridge, UK, 2010; p. 280. ISBN 9781845697464.
20. Fissore, D.; Pisano, R.; Barresi, A.A. Process analytical technology for monitoring pharmaceuticals freeze-drying–A comprehensive review. Dry. Technol. 2018, 36, 1839–1865.
21. Morais, A.R.D.V.; Alencar, É.D.N.; Xavier Júnior, F.H.; Oliveira, C.M.D.; Marcelino, H.R.; Barratt, G.; Fessi, H.; Egito, E.S.T.D.; Elaissari, A. Freeze-drying of emulsified systems: A review. Int. J. Pharm. 2016, 503, 102–114.
22. Bhandari, B.; Bansal, N.; Zhang, M.; Schuck, P. Handbook of Food Powders: Processes and Properties; Woodhead Publishing: Sawston, Cambridge, UK, 2013; p. 655. ISBN 9780857098672.

23. González-Ortega, R.; Faieta, M.; Di Mattia, C.D.; Valbonetti, L.; Pittia, P. Microencapsulation of olive leaf extract by freeze-drying: Effect of carrier composition on process efficiency and technological properties of the powders. J. Food Eng. 2020, 285, 110089.

24. Lopes, L.A.A.; Carvalho, R.D.S.F.; Magalhães, N.S.S.; Madruga, M.S.; Athayde, A.J.A.A.; Portela, I.A.; Barão, C.E.; Pimentel, T.C.; Magnani, M.; Stamford, T.C.M. Microencapsulation of Lactobacillus acidophilus La-05 and incorporation in vegan milks: Physicochemical characteristics and survival during storage, exposure to stress conditions, and simulated gastrointestinal digestion. Food Res. Int. 2020, 135, 109295.

25. Plaza, A.; Cabezas, R.; Merlet, G.; Zurob, E.; Concha-Meyer, A.; Reyes, A.; Romero, J. Dehydrated cranberry juice powder obtained by osmotic distillation combined with freeze-drying: Process intensification and energy reduction. Chem. Eng. Res. Des. 2020, 160, 233–239.

26. Alvarez, C.; Ospina, S.; Orrego, C.E. Effects of ultrasound-assisted blanching on the processing and quality parameters of freeze-dried guava slices. J. Food Process. Preserv. 2019, 43, 14288.

27. Feng, Y.; Ping Tan, C.; Zhou, C.; Yagoub, A.E.G.A.; Xu, B.; Sun, Y.; Ma, H.; Xu, X.; Yu, X. Effect of freeze-thaw cycles pretreatment on the vacuum freeze-drying process and physicochemical properties of the dried garlic slices. Food Chem. 2020, 324, 126883.

28. Witrowa-Rajchert, D.; Wiktor, A.; Sledz, M.; Nowacka, M. Selected emerging technologies to enhance the drying process: A review. Dry. Technol. 2014, 32, 1386–1396.

29. Alipoorfard, F.; Jouki, M.; Tavakolipour, H. Application of sodium chloride and quince seed gum pretreatments to prevent enzymatic browning, loss of texture and antioxidant activity of freeze dried pear slices. J. Food Sci. Technol. 2020, 57, 3165–3175.

30. Różyło, R. Recent trends in methods used to obtain natural food colorants by freeze-drying. Trends Food Sci. Technol. 2020, 102, 39–50.

31. Barba, F.J.; Parniakov, O.; Pereira, S.A.; Wiktor, A.; Grimi, N.; Boussetta, N.; Saraiva, J.A.; Raso, J.; Martin-Belloso, O.; Witrowa-Rajchert, D.; et al. Current applications and new opportunities for the use of pulsed electric fields in food science and industry. Food Res. Int. 2015, 77, 773–798.

32. Wu, X.; Wang, C.; Guo, Y. Effects of the high-pulsed electric field pretreatment on the mechanical properties of fruits and vegetables. J. Food Eng. 2020, 274, 109837.

33. Ghosh, S.; Gillis, A.; Levkov, K.; Vitkin, E.; Golberg, A. Saving energy on meat air convection drying with pulsed electric field coupled to mechanical press water removal. Innov. Food Sci. Emerg. Technol. 2020, 66, 102509.

34. Rybak, K.; Samborska, K.; Jedlinska, A.; Parniakov, O.; Nowacka, M.; Witrowa-Rajchert, D.; Wiktor, A. The impact of pulsed electric field pretreatment of bell pepper on the selected properties of spray dried juice. Innov. Food Sci. Emerg. Technol. 2020, 65, 102446.

35. 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 2020, 121, 108937.

36. Wu, Y.; Zhang, D. Effect of pulsed electric field on freeze-drying of potato tissue. Int. J. Food Eng. 2014, 10, 857–862.

37. Yamada, T.; Yamakage, K.; Takahashi, K.; Takaki, K.; Orikasa, T.; Kamagata, J.; Aoki, H. Influence of Drying Rate on Hot Air Drying Processing of Fresh Foods Using Pulsed Electric Field. IEEJ Trans. Electr. Electron. Eng. 2020, 15, 1123–1125.

38. Lammerskitten, A.; Mykhailyk, V.; Wiktor, A.; Toepfl, S.; Nowacka, M.; Bialik, M.; Czyżewski, J.; Witrowa-Rajchert, D.; Parniakov, O. Impact of pulsed electric fields on physical properties of freeze-dried apple tissue. Innov. Food Sci. Emerg. Technol. 2019, 57, 102211.

39. Wu, Y.; Guo, Y. Experimental study of the parameters of high pulsed electrical field pretreatment to fruits and vegetables in vacuum freeze-drying. In International Conference on Computer and Computing Technologies in Agriculture, Proceedings of the CCTA 2010: Computer and Computing Technologies in Agriculture IV, Nanchang, China, 22–25 October 2010; Springer: Heidelberg, Germany, 2010; Volume 344, pp. 691–697.

40. Parniakov, O.; Bals, O.; Lebovka, N.; Vorobiev, E. Pulsed electric field assisted vacuum freeze-drying of apple tissue. Innov. Food Sci. Emerg. Technol. 2016, 35, 52–57.

41. Santacatalina, J.V.; Fissore, D.; Cárcel, J.A.; Mulet, A.; García-Pérez, J.V. Model-based investigation into atmospheric freeze drying assisted by power ultrasound. J. Food Eng. 2015, 151, 7–15.

42. Bai, Y.; Luan, Z. The effect of high-pulsed electric field pretreatment on vacuum freeze drying of sea cucumber. Int. J. Appl. Electromagn. Mech. 2018, 57, 247–256.

43. Bhargava, N.; Mor, R.S.; Kumar, K.; Sharanagat, V.S. Advances in application of ultrasound in food processing: A review. Ultrason. Sonochem. 2021, 70, 105293.

44. Kowalski, S.J.; Mierzwa, D.; Stasiak, M. Ultrasound-assisted convective drying of apples at different process conditions. Dry. Technol. 2017, 35, 939–947.

45. Cheng, X.; Zhang, M.; Xu, B.; Adhikari, B.; Sun, J. The principles of ultrasound and its application in freezing related processes of food materials: A review. Ultrason. Sonochem. 2015, 27, 576–585.

46. Dai, C.; Zhou, X.; Zhang, S.; Zhou, N. Influence of ultrasound-assisted nucleation on freeze-drying of carrots. Dry. Technol. 2016, 34, 1196–1203.

47. Merone, D.; Colucci, D.; Fissore, D.; Sanjuan, N.; Carcel, J.A. Energy and environmental analysis of ultrasound-assisted atmospheric freeze-drying of food. J. Food Eng. 2020, 283, 110031.

48. de la Fuente-Blanco, S.; de Sarabia, E.R.-F.; Acosta-Aparicio, V.M.; Blanco-Blanco, A.; Gallego-Juárez, J.A. Food drying process by power ultrasound. Ultrasonics 2006, 44, e523–e527.

49. Colucci, D.; Fissore, D.; Rossello, C.; Carcel, J.A. On the effect of ultrasound-assisted atmospheric freeze-drying on the antioxidant properties of eggplant. Food Res. Int. 2018, 106, 580–588.

50. Xu, X.; Zhang, L.; Feng, Y.; Zhou, C.; Yagoub, A.E.G.A.; Wahia, H.; Ma, H.; Zhang, J.; Sun, Y. Ultrasound freeze-thawing style pretreatment to improve the efficiency of the vacuum freeze-drying of okra (Abelmoschus esculentus (L.) Moench) and the quality characteristics of the dried product. Ultrason. Sonochem. 2021, 70, 105300.

51. Wu, X.F.; Zhang, M.; Ye, Y.; Yu, D. Influence of ultrasonic pretreatments on drying kinetics and quality attributes of sweet potato slices in infrared freeze drying (IRFD). LWT 2020, 131, 109801.

52. Fan, D.; Chitrakar, B.; Ju, R.; Zhang, M. Effect of ultrasonic pretreatment on the properties of freeze-dried carrot slices by traditional and infrared freeze-drying technologies. Dry. Technol. 2020. Available online: https://www.tandfonline.com/doi/abs/10.1080/07373937.2020.1815765 (accessed on 30 October 2020).

53. Ren, F.; Perussello, C.A.; Zhang, Z.; Kerry, J.P.; Tiwari, B.K. Impact of ultrasound and blanching on functional properties of hot-air dried and freeze dried onions. LWT—Food Sci. Technol. 2018, 87, 102–111.

54. Schössler, K.; Jäger, H.; Knorr, D. Novel contact ultrasound system for the accelerated freeze-drying of vegetables. Innov. Food Sci. Emerg. Technol. 2012, 16, 113–120.

55. Mello, R.E.; Fontana, A.; Mulet, A.; Correa, J.L.G.; Cárcel, J.A. Ultrasound-assisted drying of orange peel in atmospheric freeze-dryer and convective dryer operated at moderate temperature. Dry. Technol. 2020, 38, 259–267.

56. Yildiz, G.; Izli, G. The effect of ultrasound pretreatment on quality attributes of freeze-dried quince slices: Physical properties and bioactive compounds. J. Food Process Eng. 2019, 42, e13223.

57. Carrión, C.; Mulet, A.; García-Pérez, J.V.; Cárcel, J.A. Ultrasonically assisted atmospheric freeze-drying of button mushroom. Drying kinetics and product quality. Dry. Technol. 2018, 36, 1814–1823.

58. Carrión, C.; Mulet, A.; García-Pérez, J.V.; Cárcel, J.A. Ultrasonically assisted atmospheric freeze-drying of button mushroom. Drying kinetics and product quality. Dry. Technol. 2018, 36, 1814–1823.