Trends in Approaches to Assist Freeze-Drying of Food: A Cohort Study on Innovations

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ABSTRACT
Freeze drying has been a very successful technology in the food and pharma industries, particularly owing to the benefits it offers in terms of product quality. It is most recommended for high-value foods and those that contain heat-sensitive ingredients. Over the years, to meet industry requirements, several variants of the freeze-drying technology have been developed, even to meet specific drying applications. The process of freeze-drying is highly energy-intensive and time-consuming. Nevertheless, innovative techniques are available with the potential to produce products at reduced drying times and lower cost, while being environment-friendly and maintaining freeze-dried quality. This review summarizes advances in the application of infrared, microwave, ultrasound, pulsed electric field, and other techniques as pre-treatments and/or to assist conventional freeze-drying processes. Freeze drying combined with other techniques can provide more benefits in terms of energy, time, and cost savings. In this review, comparative studies have been presented to describe these aspects. Such techniques to assist the freeze-drying process can be linked well with sustainable food processing strategies, particularly considering a significant reduction in energy requirements.

Introduction

Drying is a key unit operation and possibly the oldest food preservation method.[1] The prime focus of drying is the preservation of the product with the associated conversion of a liquid or moist product into powders, flakes, or solids, without significantly compromising its physio-chemical properties. Drying also extends the shelflife of food products by inhibiting microbial growth, enzymatic activity, and reduces or avoids the use of chemicals.[2] However, conventional drying approaches result in physical and chemical quality degradation, reducing the food’s acceptability to consumers.[3] Accordingly, improved drying approaches are desirable for food and agro-products to produce products with better quality, ideally with shorter drying time, higher capacity, better process control, lower cost, and improved operational safety. Additionally, drying techniques must be non-polluting and have minimal impact on the environment.[4]

Freeze-drying (FD), lyophilization, or sublimation drying has gained interest and has long been known as a premium drying technique as it provides good retention of vital food properties. It also improves the stability of the product. The absence of liquid water and the reduced temperatures during sublimation ceases the majority of deteriorative reactions, e.g., microbiological reactions which...
produce high-quality dried products. The approach is used for heat-sensitive food samples, including bioactive constituents and microbial cultures. It causes minimal shrinkage of the product and results in excellent rehydration capacity, color retention, and superior taste. Generally, less than 20–25 g/100 g of moisture content and water activity below 0.6 can facilitate control of microbial proliferation and enzymatic activity in foods. FD products can be easily reconstituted with water because of their highly porous structure.

Although FD is a well-accepted drying technique for producing superior quality products, high energy, and time requirements remain a challenge. Long drying times are associated with low vapor pressure driving forces for mass transfer and difficulties in transferring heat effectively in a vacuum system. Similarly, an appropriate selection of processing conditions is very important to improve the efficiency of energy utilization. The study conducted by Fissore, Pisano, & Barresi for freeze-drying of coffee presented an exergy analysis to represent the effectiveness in energy utilization. These authors have proved that the exergy losses can be reduced if high chamber pressure and low values of heating fluid temperature are adopted. While pressure and temperature are crucial parameters, appropriate process control of the drying chamber is necessary to avoid the occurrence of sonic flow (choking flow) in the duct connecting the chamber and the condenser especially when the sublimation flux is too high.

The capital, operational, and maintenance costs of freeze-drying units are four to eight times higher than conventional drying units such as hot air drying. High capital and energy requirements are associated with the need to maintain condenser systems running at the very low temperatures needed to provide the low water vapor pressure environment. For instance, in a study conducted on the drying of okra snacks, the specific energy consumption of a typical FD process was reported to be around 75 kWh/kg H₂O since it is a slow process, as compared to hot air drying which required ~50 kWh/kg H₂O. However, by combining freeze-drying with microwave vacuum drying, the energy consumption values could be lowered to ~20 kWh/kg H₂O. The cost of freeze-drying depends upon the type of raw material, the capacity of the plant, and the duration of the cycle, amongst other factors. Hence, improvements in the FD method can significantly enhance drying rates and conserve energy without compromising product quality. Therefore, to overcome the shortcomings of FD, a judicious

Figure 1. Phase diagram of water \([T_m(w)]:\) melting or freezing temperature of pure water; \(T_n:\) ice nucleation temperature; \(T_g(w):\) glass transition of pure water; \(T_m(s):\) melting or freezing temperature of the solute; \(T_g(s):\) glass transition temperature of the solute; \(T_g:\) glass transition temperature of the maximally freeze-concentrated solution (for an amorphous solution); \(T_c:\) collapse temperature; \(T_{eu}:\) eutectic temperature of a frozen crystalline solution. (Source: Assegehegn et al., 2018).

5. The approach is used for heat-sensitive food samples, including bioactive constituents and microbial cultures. It causes minimal shrinkage of the product and results in excellent rehydration capacity, color retention, and superior taste. Generally, less than 20–25 g/100 g of moisture content and water activity below 0.6 can facilitate control of microbial proliferation and enzymatic activity in foods. FD products can be easily reconstituted with water because of their highly porous structure.

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combination of freeze-drying with other techniques can prove beneficial. In recent years, various techniques such as ultrasonic treatment, pulsed electric field, and osmotic dehydration have been successfully used to meet these requirements to overcome the challenges associated with conventional FD processes. For example, atmospheric freeze-drying significantly reduces energy consumption. Atmospheric freeze-drying is a combination of freeze-drying and conventional drying. In freeze-drying, more energy is required as a large vacuum is provided to the frozen material for the sublimation of the frozen solvent. This frozen material can also be dried without the application of a large vacuum. Thus, the basic approach behind atmospheric freeze-drying is to increase the energy efficiency by maintaining the high product quality. Similarly, for liquid foods, an organic solvent can modify the product structure which eventually changes the rate of sublimation.\textsuperscript{[13,14]}

Accordingly, this is the focus of this review, discussing the science behind such combined approaches and presenting key findings of recent studies in comparison with the FD process alone. Such hybrid techniques focus on harnessing the synergistic benefits of one or more assisting technique, in terms of retention of food quality at reduced cost and time requirements. Apart from these innovations, several other advances have been reported in the context of freeze-drying of foods.

**Fundamentals of freeze-drying**

The FD process can be explained using the phase diagram of water. There are three main processes involved in FD: freezing, primary drying, and secondary drying. The freezing step can either be performed outside the freeze dryer using dedicated freezing equipment or in the freeze dryer itself if the shelves are fabricated in such a way as to allow coolant fluid to pass within them. For smaller products, the freezing step can be performed under a vacuum because of the pressure drop. During freezing the temperature of the liquid in the sample is lowered below the freezing point temperature. Phase change occurs upon the nucleation of ice crystals and subsequent crystal growth. Water is thus converted into the ice with the effect that dissolved solutes become more concentrated in the remaining unfrozen water. Cooling rate, solute concentrations, and temperature critically affect the crystallization process (f). Ultimately, a point is reached when no further development of ice crystals is possible\textsuperscript{[15]} which corresponds to the point when the amorphous non-ice phase passes through the glass transition into the glassy state.

Once the freezing process has completed, drying begins by exposing the frozen material to a low water vapor pressure environment. In practice, this is achieved by the use of low-temperature condensers to remove water from the headspace, either in the same or a connected chamber, and with vacuum applied to improve the rates of mass transfer (although this does reduce heat transfer). Initially, ice is removed by sublimation and this is known as primary drying. Sublimation begins at the surface and a sublimation front then moves into the material. The speed of the front heavily depends on the temperature-pressure combinations maintained. If performed properly the non-ice phase is maintained in the glassy state such that when ice crystals sublime they leave behind voids and the frozen shape of the material is maintained, i.e. avoids so-called “collapse”. Thus, freeze-dried products show low shrinkage and highly porous structures.\textsuperscript{[12]} Once the sublimation front has passed, secondary drying occurs in which left-over moisture in the glassy matrix is removed by desorption. This secondary moisture removal is important as without it the material will not be dry enough to withstand the sample collapsing (and destroying porosity) when the temperature of the sample ultimately rises. The primary and secondary drying processes overlap as at any particular location secondary drying will start as soon as the primary sublimation front has passed through. Slightly confusingly, “primary” and “secondary” drying is also routinely used to refer to phases in the actual operation of freeze dryers. The “primary drying phase” refers to the main freeze-drying period when shelf temperatures are kept low (and the dominant mode of drying is primary sublimation). The “secondary drying phase” is the final period of freeze-drying after all the ice has gone, only secondary drying is occurring, and the further loss of moisture from the glassy matrix allows the temperature to be gradually raised without collapsing the sample. Nevertheless, structural collapse or loss of cake
structure, and interfacial effects along the ice/freeze-concentrate interface can result in significant effects on the stability of key ingredients and in the overall quality of the food product.\textsuperscript{[16]} Thus, there are five main processing elements involved with freeze-drying: freezing, sublimation, desorption, vacuum pumping, and vapor condensation.\textsuperscript{[15]} The time and energy requirements in each stage are heavily dependent on product characteristics, dryer design and configurations, and process variables. The energy required to thaw the condenser must also be considered. Further, requirements regarding the moisture content of the final product are product specific.

The control of the residual moisture content of the FD sample is very important. Over-drying may damage the products, leading to a loss of protein activity during storage. It can also degrade bioactive compounds and have negative implications on natural pigments. Furthermore, high residual moisture contents accelerate product degradation over time.\textsuperscript{[13]} There are various studies conducted to control residual moisture content. Oddone et al.\textsuperscript{[17]} achieved 1.5\% residual moisture content with the help of vacuum-induced nucleation; the process is known to help in the development of larger pores and could lower drying time by up to 55\%. Another study was conducted by Mphahlele et al.\textsuperscript{[18];} they reported that the residual moisture content of pomegranate peel powder was \(0.087 \pm 0.002\) kg water/kg dry matter after 16 hours of the drying period. In general, the residual moisture content of crystallizing products ranges from 1\% (e.g., glycerin) and 10\% (e.g., mannitol) but for an amorphous product it ranges from 15\% to 20\%.\textsuperscript{[17,19]} Though often not highlighted, the storage of freeze-dried products is important, critically affecting the quality and stability of the dried products. This is because FD products are highly porous and extremely hygroscopic.\textsuperscript{[20]} In most cases, FD aims for long-term product storage, and this explains the need to arrest deteriorative biochemical and microbiological reactions using rigorous packaging and storage practices. Appropriate shelflife studies, including methods involving accelerated shelflife testing, have been used to approximate product stability during storage. Owing to the high costs involved, the application of FD to most (low value) food products is not feasible. The technology is commercially viable for biological formulations, therapeutic drugs, and high-value foods. Nevertheless, FD has gained popularity for other applications as well, including the use of domestic benchtop units. The following sections elaborate on various techniques being used in combination with FD, and selected innovative strategies that supplement FD processes, to meet specific product requirements.

\textbf{Assisting freeze-drying with novel technologies}

The process of FD involves complex and multi-faceted issues that link product characteristics, changes in the product during the freezing/drying stages, mechanisms of heat and mass transfer, whilst considering the quality of the end-product. In particular, the drying stage is a time-intensive operation, as moisture from the product (in the form of ice or water) has to be removed from the inner layers of the product with only a very small vapor pressure driving force for mass transfer. There is clear merit in researching methods to assist the FD to achieve lower drying times and lower energy requirements to remove the same level of moisture (Table 1). Techniques that have gained importance for such applications during the past decade are elaborated below. The techniques can be classified according to the point in which they can be applied: (i) as a pretreatment, (ii) during the freezing step, or (iii) during the actual drying stage. For example, pulsed electric field processing, explosion puffing, and foaming are pretreatments for changing the material structure to enhance subsequent mass transfer, osmotic dehydration is a pretreatment to reduce the moisture content to lower the freeze-drying duty.\textsuperscript{[45]} The method of freezing (particularly control of nucleation) affects the ice microstructure that forms which in turn affects subsequent freeze-drying rates.\textsuperscript{[46]} The application of microwave and infrared radiation is used to boost sublimation rates by supplying heat. Ideally, it is required to focus on the synergistic benefits of different approaches and to minimize the limitations of individual techniques. Accordingly, a precise choice has to be made in selecting technologies and in the various process and product parameters during the freeze-drying process. Importantly, from the
Table 1. Advantages of combined drying treatments over conventional FD.

| Technique | Products              | Key advantages over conventional FD                                                                 | Implications on product quality                                                                 | References |
|-----------|-----------------------|------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------|------------|
| IR        | Pear                  | • Higher dehydration time • Decreased drying time by 14.3–42.9% • Better rehydration capacity      | • More retention of total antioxidant activity in products • Lower damage to the phenolic content | [21]       |
|           | Banana snacks         | • Rapid drying • 70% reduction in drying time • IR lamp used minimum electrical energy and saved electrical energy consumption | • Crisper samples • Same color as compared to FD                                                | [22]       |
|           | Cordyceps militaris   | • Reduction in 7.21–17.78% of drying time • 11.88–18.37% of the energy consumption                | • Enhanced retention of cordycepins, total phenolics, hydroxyl radical scavenging activity, reducing power, 3-octanone, 3-octanol and 1,3-octadiene | [23]       |
|           | Aloe vera             | • 8.5 W/g IR power, 80.24°C product temperature and 6.67 hours drying time gives minimum moisture content of 3.49%, maximum wettability of 52.84 s and maximum yield of 3.87% | • Better retention of color and texture • Higher retention of antioxidant                           | [24]       |
| MW        | Okra snacks           | • Decreased drying time by 75.36% • Reduced energy consumption by approximately 71.92%            | • Higher retention of color, lower apparent density, and lower foaming stability                    | [12]       |
|           | Duck egg white protein powder | • Reduced drying time • Increased drying rate                                                | • Survival rate and membrane integrity were the same for cultures                                 | [25]       |
|           | Lactic acid bacteria  | • Reduction of drying time by 80% • Higher energy efficiency                                     | • Solubility is 60%                                                                              | [26]       |
|           | Crude protein from Ginkgo biloba L. | • The drying time is reduced to 5 hours                                                  |                                                                                                   | [27]       |
| US        | Button mushroom       | • Reduced drying time up to 74%                                                              | • No significant effect of ultrasound on color, texture, rehydration, and cell damage              | [28]       |
|           | Barley grass          | • Reduction in drying time by 14% • Reduced energy consumption by 19%                           | • Reduced total microbial count by 33% • High flavonoid and chlorophyll content                    | [29]       |
|           | Microbial transglutaminase | • Reduced drying time                                                                      | • Increased enzymatic activity • Improved stability in extreme pH levels and high temperature • Smaller particles and better particle morphology | [30]       |
| PEF       | Apple tissue          | • Enhanced relative pore size by ≈86 • Rehydration capacity by ≈1.3 • Accelerated cooling and drying • Improved efficiency of drying | • Preserve the shape of the final product                                                           | [31]       |
|           | Potato tissue         | • Increased drying rate by 14.31% • Reduced drying time by 31.47% • Decreased energy consumption by 16.59% |                                                                                                   | [32]       |
|           | Apple                 | • Decreased specific energy consumption by 20.46% • Reduced drying time by 22.50% • Increased productivity per unit area by 28.50% • Increased drying rate by 27.02% |                                                                                                   | [33]       |
Table 1. (Continued).

| Technique | Products | Key advantages over conventional FD | Implications on product quality | References |
|-----------|----------|--------------------------------------|---------------------------------|------------|
| EPD       | Restructured carrot–potato chips | • Minimize energy consumption  
• Reduced drying time | • Superior texture  
• More desirable color | [34] |
|           | Jackfruit chips | • Improved efficiency of drying  
• Reduced drying time | • Decreased color deterioration and the product was more similar to fresh jackfruits  
• Relatively more retentions of ascorbic acid, phenolics, and carotenoids | [35] |
|           | Mango, pitaya and papaya fruit chips | • Increased drying rate and reduced cost | • High retentions of ascorbic acid, total phenolic, total carotenoids, and total flavonoids  
• Minimized browning discoloration and pigment degradation  
• More expansion ratio | [36] |
|           | Jackfruit bulb chips | • Shorter drying time  
• Less operating cost | • FD-EPD showed the highest expansion ratio (119%), best color and texture compared with the FD-EPD and AD-EPD dried samples  
• More antioxidant capacities and higher retention of phenolics and carotenoids | [37] |
|           | Restructured carrot–potato chips | • Reduced energy consumption  
• Lower production cost  
• Reduced drying time  
• Improve the efficiency of dehydration | • Pleasant crispness and flavor | [38] |
| OD        | Apricots | • Reduced drying time | • Vitamin C lost by 21%  
• Total sugars increased to 57%  
• Retain structural and mechanical property | [39] |
|           | Strawberry | • Decreased processing time | | [40] |
| SD        | Vanillin | • Better thermal stability | • Spherical size particle with fine pores | [41] |
|           | Coffee | • Better solubility | • Enhanced retention of volatiles by 93% | [42] |
|           | Lactobacillus plantarum | • Better cell viability | • Improved flowability and lower hygroscopicity | [43] |
|           | Lactobacillus casei | • More protective | • Fine probiotic powder | [44] |

sustainability point of view, it is vital to consider the environmental impact of such hybrid techniques. These methods are now presented in more detail, and in order of the point of application.

**Pretreatment**

**Ultrasound pretreatment**

Ultrasound (US) technology is a non-thermal approach and is particularly interesting as it can be applied in all three points of application. US waves are acoustic pressure waves that alternately create compression and expansion in a material. US waves, both high (5–10 MHz) and low (20–100 kHz) frequencies have found a range of applications in food processing and non-destructive food quality evaluation. It can be very violent on a microscale-level and can disrupt the material structure, and hence, can be used as a pretreatment if this disruption provides easier routes for mass transfer. It can also act as a nucleation enhancer and thus a means to control freezing (and the resulting ice structure). Finally, US can be applied in the freeze-drying process itself, predominantly atmospheric freeze-drying...
(where it is transmitted through the gas) but is also possible in conventional freeze-drying (where it is transmitted through the shelves), to enhance heat and mass transfer during freeze-drying.\textsuperscript{[48]}

The use of US as a pretreatment for FD has been found to reduce subsequent drying times and retain or even improve product quality. US pretreatment improves the subsequent mass transfer in both solid and liquid products by permanently changing the structure of the material, by either creating internal voids and/or disrupting cell membranes.\textsuperscript{[37,48]} Cellular disruption by US is known to result in microbial inactivation. Despite the material disruption, Islam et al.\textsuperscript{[49]} found that ultrasound pretreatment was able to even improve the quality of freeze-dried pears, in terms of better texture and porosity.

Application of US as pretreatment during drying also helps to reduce enzyme activity such as ascorbate peroxidase (APX), peroxidase (POD), and polyphenol oxidase (PPO). In a model system, around 70% inactivation of POD was observed and the activity of APX and PPO were reduced by 12–20% and 20–30% respectively while untreated samples showed higher enzymatic activity.\textsuperscript{[50,51]} In another study, Rodriguez et al.\textsuperscript{[52]} found that sonicated samples possessed 58% reduced PPO activity than untreated fresh apple samples. Focusing on lowering energy consumption during FD process, Cao et al.\textsuperscript{[29]} applied US as a pretreatment to barley grass at different power levels (10, 30, 45, 60 W/L) for 10 minutes. Figure 2 shows the energy consumption of FD and FD samples pretreated with US. These researchers found that energy consumption and drying time declined significantly in the case of US-assisted FD, as compared with conventional FD. For US treated samples (45 W/L), drying time and energy consumptions were 14% and 19% lower, respectively, and the authors explained that US pretreatment increases the porosity of the material, in turn, enhancing mass transfer. UT enhanced the stability of the samples and produced high-quality products. They also observed that US treatment reduces the microbial load (40 MPN/100 g to 20 MPN/100 g). Ideally, microbial cell membranes become damaged, directly inactivating spoilage microorganisms which prevents loss of nutritional substances.\textsuperscript{[53]}

\textit{Pulsed electric field–assisted freeze-drying}

Pulsed electric field (PEF) processing of foods involves the application of short duration high voltage electrical pulses to a product that is between two electrodes. Polarization effects cause permeabilization of cell membranes, thereby accelerating mass transfer, but crucially keeps the semi-rigid cell wall structure intact. PEF is a proven non-thermal approach for sterilization of biological cells, without causing unacceptable alterations in food tissues.\textsuperscript{[54,55]} In most cases, PEF has been adopted successfully
with liquid foods with good conductivity characteristics. Studies on the applications of PEF treatment with FD are limited, including reports on potatoes, and apples. Lammerskitten et al. studied the effect of PEF pretreatment on the kinetics of FD apple tissue. PEF improved the freeze-drying kinetics and decreased the treatment time by 57% compared to untreated apple slices. The pretreatment also helped in enhanced absorption of water. Similarly, in another study conducted by Fauster et al. the effect of PEF pretreatment on the physical properties of bell peppers and strawberries was evaluated. A decrease in shrinkage was observed for bell peppers and strawberries in comparison to the untreated samples. PEF is a highly effective pretreatment requiring less energy inputs to enhance the quality of FD products. These researchers have confirmed that PEF can be an effective pretreatment for conventional FD processes. Both Jalté et al. and Wu and Zhang found optimal parameters of electric field strength, pulse width, and pulse number to be 1500 V cm⁻¹, 120 μs and, 45 pulses, respectively. At this optimum condition, energy consumption reduced by 17%, drying time reduced by 31%, and drying rate improved by 14%. Similar improvements (although not quantified) were seen by Jalté et al., who used field strength of 400 V cm⁻¹ (alternating polarity), 100 μs pulse width, and approx. 1000 pulses. They also found marked improvements in the quality of the freeze-dried potato, being more uniform in color and shape, and with less shrinkage.

In another study on apple, Barba et al. explained the effects of PEF-assisted FD using microscopy and capillary impregnation analysis. First, the samples were PEF pretreated (100 pulses of 800 V cm⁻¹ for 1000 μs). Subsequently, vacuum cooling was given to the samples to reduce the temperature to a sub-zero level for FD. A monopolar PEF generator capable of producing pulses of a near-rectangular shape was used in this study. PEF significantly improved the FD drying time. Final products analyzed using the capillary impregnation test revealed larger pores in the case of PEF pretreated samples, justifying its associated effects, even in terms of improvement in rehydration capacity, as compared with untreated samples. In their study, these researchers reported an increase in relatively pore size and rehydration capacity by 86% and 1.3%, respectively. Like Jalté et al., found with potatoes, Parniakov also found less surface shrinkage with the PEF pretreated samples after freeze-drying. PEF treatment enhances the freezing and thawing rates, thus decreasing process times. Importantly, PEF reduces the impact of food freezing on the environment, making it a sustainable technology.

**Freeze-explosion puff drying**

Explosion puffing drying (EPD), also known as instant controlled pressure drop texturing (DIC, French for Détente InstantanéeContrôlée) is an emerging concept. This technique is based on thermo-mechanical effects associated with samples rapidly subjected to saturated steam (about 0.1–0.6 MPa) followed by a sudden pressure drop up to vacuum (about 5 kPa). In general, the following steps are used a) sample is predried to attain the required moisture content b) water equilibrium c) providing steam pressure with high-temperature d) sudden pressure drop to vacuum d) final drying. These steps can also be described in three stages. Such sudden pressure drop causes auto-vaporization of volatile compounds, swelling, porous structure, and rupture of some cell walls. Notably, the rapid cooling of the product stops thermal degradation. DIC technology is well suited to heat-sensitive food products and is known to yield products with low-density, high-expansion characteristics, and porous microstructures. DIC gives controlled texture expansion and the last phase of drying can be achieved more rapidly, thus reducing energy consumption and hence processing costs. Considering quality and energy consumption, a combination of DIC and FD has been perfectly adapted to jackfruit chips, jackfruit bulb chips, mango, pitaya, and papaya fruit chips, and carrot–potato chips. In a comparative drying study among instant controlled pressure drop-assisted freeze drying (FD-DIC), instant controlled pressure drop-assisted hot air drying (AD-DIC), and freeze-drying (FD) performed on jackfruit bulb chips, proved that chips treated by FD-DIC showed an excellent quality than other drying methods. In this method, samples were first freeze-dried, during FD, treatment pressure was maintained at 0.1 kPa and the condenser temperature was –56°C; secondary drying was performed at 25°C. A part of the samples was dried using
a freeze dryer till the moisture content reaches the final value and another batch was shifted to the processing vessel of an experimental DIC processor to reduce the moisture.

Researchers also compared combined freeze-explosion puff drying (FD-EFD) and various other drying techniques, concluding that FD-EFD can be a better alternative to conventional FD. A study on restructured carrot-potato chips[38] explained that convective hot air drying combined with EFD is not an appropriate technique owing to significant shrinkage effects in the samples during the drying period. However, FD-EFD did not have such issues and yielded products with pleasant crispness and flavor. Yi et al.[37] examined the effect of AD (air drying), FD, infrared (IR) drying, microwave (MW) drying, and vacuum drying (VD) for explosion puff drying (EPD) on the qualities of jackfruit (Artocarpus heterophyllus L.) chips. IR and MW will also be discussed in later sections but FD required the longest time for drying, followed by the FD-EPD process (Fig. 3). These treatments – AD-EPD, FD-EPD, IR-EPD, MV-EPD, and VD-EPD – required 53%, 83%, 21%, 20%, and 46% of the total drying time, respectively. Among all treatments, FD retained the highest amount of ascorbic acid, phenolics, and carotenoids because of the absence of oxygen and due to lower drying temperatures during the FD process. Further, the absence of liquid water in the FD process is key merit. On the other hand, FD-EPD showed the highest content of these compounds compared to those from AD-EPD, IR-EPD, MV-EPD, and VD-EPD. It could be concluded that combined drying methods of FD-EPD can provide samples with high quality, similar to that of FD products. In addition, FD-EPD requires relatively lower production cost as compared to FD. Hence, FD-EPD can be an alternative method to dry foodstuffs, to maintain high quality, without compromising on drying time and operational cost benefits.

**Osmotic dehydration–assisted freeze-drying**
In osmotic dehydration (OD), the equilibrium condition is retained on both sides of the membrane by evacuating water from lower solute concentration to higher concentration.[65] OD is an inexpensive method for partial removal of water from food samples by contacting with a low water activity

Figure 3. Effect of pre-drying treatments on the total drying time of jackfruit chips.[37]
solution, with good retention of color, flavor, and nutritional quality. Whilst it is not possible to lower moisture contents down to the low levels required for storage using OD, it can be used as an energy-efficient pretreatment to reduce the moisture content so that less water need removing in the subsequent drying step. This reduces overall energy consumption and helps to maintain product quality.\textsuperscript{40,66}

Recent studies on pear and onion showed that OD reduced the drying time of conventional oven-drying by 42\% and 40\%, respectively.\textsuperscript{66} In another study, OD treatment was observed to reduce the drying time, apart from retaining vitamin C content and the color of chili.\textsuperscript{67} Similarly, Prosapio and Norton\textsuperscript{40} studied the effect of OD on oven-drying and FD of strawberries. OD reduced the drying time to 7 hours whereas, FD without pretreatment required 15 hours to dry the sample. Images of cortex cells in fresh and rehydrated strawberries obtained by confocal scanning laser microscopy are shown in Fig. 4. The images in Figs 4b and 4c are visible and comparable with Fig. 4a. This explains that processing did not alter the microstructure of the material. In Fig. 4d, cells are difficult to identify and appear broken; whereas, in Fig. 4e OD + oven-dried samples were partially broken. OD pretreatment can effectively reduce processing time and have implications on the structure of foods, apart from the nutrition content, sensory characteristics, and functional properties. Further, it is also known to prevent oxidative browning and inhibit enzymatic activity.\textsuperscript{68,69}

\textbf{Freezing}

The way that freezing is performed is important to freeze-drying processes as the ice crystal microstructure formed during freezes translates into the final porous structure of the dried material and affects the rate of mass transfer of water through the pores during drying.\textsuperscript{64,68} Thus the method of freezing, particularly the rate of freezing, significantly affects the rate of subsequent freeze-drying. Furthermore, the freezing step must be reproducible so that subsequent freeze-drying is also reproducible. Supercooling is also one of the most important key parameters which have a significant impact on ice crystal size.\textsuperscript{68–72}

In most cases, there is a straight decision to be made between slow and fast freezing with slow freezing favoring larger crystals/pores, and faster subsequent freeze-drying but with more likelihood of damage to cells or proteins. Some novel technologies have been developed in the pharma sector to control ice nucleation with techniques such as ice fog,\textsuperscript{19} rapid depressurization, vacuum-induced surface freezing,\textsuperscript{73} and electric field-induced nucleation.\textsuperscript{74} The ice fog technique helps to control the ice nucleation during the freeze-drying. In this technique, the temperature of the product is reduced to the anticipated ice nucleation temperature after that nitrogen gas is passed with help of a copper coil which is immersed in liquid nitrogen and then passed to the product chamber which resulted in the formation of dense ice fog. This ice fog is forced into vials which initiate the crystallization.\textsuperscript{75,76} Various research groups optimized the process condition for the formation of ice to get a better result which can applied for industrial applications.\textsuperscript{77,78} The vacuum-induced surface freezing (VISF) technique utilizes the reduced pressure inside the drying chamber for a short time. The reduced pressure evaporates water partially which leads to product temperature and promotes the nucleation of ice.\textsuperscript{73} Oddeno et al.\textsuperscript{79} used an applied improved method of VISF by isolating the drying chamber after the depressurization. These techniques are mainly applicable where the material to be frozen is liquid rather than a moist solid, which is not always the case with food systems. However, modification in the freeze-drying equipment is necessary for the application of these intensification techniques which can be quite expensive.\textsuperscript{80} In terms of food systems, available techniques include control of freezing rate, ultrasound controlled nucleation, and spray freezing.

\textbf{Effect of freezing rate}

The effect of freezing rate has on subsequent drying behavior has been known for decades. Karel and Flink\textsuperscript{81} identified slow freezing as producing larger pores and faster sublimation rates. Pardo et al.\textsuperscript{82}
demonstrated that different freezing rates inversely affected the ice crystal sizes of frozen coffee solutions. Slow freezing rates (using still air at –40°C) were found to give a higher quality coffee product in terms of
volatile retention during subsequent freeze-drying. Ceballos et al. investigated using different freezing rates (immersing container into baths of different temperatures) before freeze-drying soursop fruit pulp. They found that slower freezing (0.5°C/min) yielded lower final moisture, better solubility, faster rehydration, better color, and better retention of ascorbic acid. Woo and Mujumdar summarized the scope of using electric and magnetic fields to assist the freezing process, explaining mechanisms of ice nucleation and crystal growth. These researchers also explained the concept of magnetocaloric freezing. In recent work, Cheng et al. reviewed different hybrid freezing approaches, focusing on immersion and plate type freezers for a range of food products. Understandings from these concepts can supplement the rationale to develop improved FD technologies.

**Ultrasound-assisted nucleation**

Many researchers reported that there are many benefits of ultrasound technologies in food processing such as ultrasound-assisted drying, ultrasound-assisted osmotic dehydration, ultrasound-assisted freeze, and ultrasound microbial inactivation. Ultrasound can help to enhance the dehydration rate, improving food quality, increasing water diffusivity, sugar diffusivity, forming large crystal ice crystals, and sterilization. US can be used as a nucleation inducer during freezing and has been applied to many systems. In a couple of cases, this has been applied with a subsequent freeze-drying step. Cheng et al. found that applying US to strawberries increased the nucleation temperature and shortened the freezing time. Unfortunately, it also had the effect of increasing the freeze-drying time. This was in contrast to Dai et al. who found that freeze-drying times of carrot were shorter when using US in the freezing step. They attributed this to the higher nucleation temperature producing larger crystals and thus an easier route for mass transfer.

**Spray freezing**

A further method to improve freeze-drying times is to make the frozen particles smaller. One method available to liquid foods is spray freezing, which when combined with FD is known as Spray Freeze Drying (SFD). It is an attractive drying technique which produces powder product with high porosity. SFD consists of three important steps namely

1. **Droplet generation (atomization):** a liquid food is atomized and the bigger volume is broken down into tiny droplets;
2. **Solidification (freezing):** the atomized droplets are frozen with a cryogenic medium (such as liquid nitrogen);
3. **Sublimation drying:** sublimation of the frozen particles occurred.

The typical steps involved in SFD are shown in Fig. 5. The main competitor technique is spray drying, and SFD has some advantages and disadvantages compared to this and conventional FD. In spray drying (SD), drying temperature is high, whereas in FD drying time is long. SFD provides both these benefits and also much better control over particle size than FD. This technique has many applications for thermally sensitive compounds for example bovine serum albumin, vitamin E, docosahexaenoic acid, and fish oil. The significance of SFD over FD and SD has been proved by Ishwarya and Anandharamakrishnan. They have performed comparative studies on physical and aroma characteristics of coffee dried using SFD with those from the FD and SD techniques. It was observed that the SFD retains the sensitive low-boiling aromatic components of coffee during processing; however, these components were lost in the initial phases of the SD and FD method. Figure 6 demonstrates the SEM pictures of the SFD, FD, and SD samples of coffee. These indicate the distinction in the microstructure with the different drying processes. In SFD coffee samples, a spherical image with a rough surface was found and in SD and FD samples spherical shape with a smooth surface was observed. Hence, with less processing time SFD produces particles with more porosity, high stability, and enhanced quality when contrasted with SD and FD.
Ultrasonic atomization in spray freezing

A variant of spray freeze-drying technology is to use ultrasonic atomizers to produce a more homogeneous spray. Ultrasonic Spray Freeze Drying (USFD) yields porous particles following stages of ultrasonic atomization, rapid freezing, and lyophilization. The approach has been extensively used for the production of powders such as partially purified microbial transglutaminase (mTGase) powder\textsuperscript{[30]} and liposomal dry powder.\textsuperscript{[96]} In the production of partially purified microbial transglutaminase (mTGase) powder, Isleroglu et al.\textsuperscript{[30]} observed that USFD process gives higher enzymatic activity.
and smaller particle size as compared to conventional FD. It was also found that USFD better retains the samples at extreme pH levels and high temperatures in comparison to the conventional FD process.

**Drying**

Ratti[97] explained the energy cost breakdown for a typical freeze-drying process. The sublimation process contributes to around 45% of the total requirement, followed by vacuum (26%), condensation (25%), and freezing (4%). In other words, it is extremely vital to focus on the energy requirements during the sublimation stage. Hybrid approaches can boost typical drying mechanisms and thereby prove beneficial.

**Infrared-assisted freeze-drying**

Infrared (IR) radiation corresponds to the part of the electromagnetic (EM) spectrum with wavelengths ranging from 700 nm to 1 mm (430 THz to 300 GHz). IR radiation is generally absorbed in a thin surface layer. The depth to which IR radiation penetrates depends upon the wavelength of radiation and product characteristics such as its absorbance behavior. Heat is transferred from the surface of the food to inner portions through conduction during IR drying. IR drying has been extensively used for various products such as fruits, vegetables, meat, and fish, as it offers rapid heating, faster drying rate, short process time, low energy cost, and high retention of nutrients[84,85]. The application of IR with FD provides rapid drying, reduced drying time, and increased energy efficiency. Figure 7 shows a schematic model for IR–assisted FD process[98]. The IR is applied during the drying phases to supply the energy required for the sublimation of ice, with significantly higher effects as compared with heat supplied through the shelves.

In recent years, many researchers have examined the effect of IR–assisted FD for drying numerous food samples such as strawberries, sweet potatoes[99] shiitake mushrooms, [100] amongst others. Wu, Zhang, and Bhandari[23] explained that the application of IR–assisted FD decreases drying time by 7 ~ 18% and saved energy by 12 ~ 18% in comparison with conventional FD. When IR radiation penetrates the surface layer of the food sample, it produces molecular vibrations and gives thermal energy to speed up the drying rate during FD. Khampakool, Soisungwan, and Park[22] conducted a comparative study between FD- and IR-assisted FD using banana snacks as the product. Table 2 presents drying time, drying rate, vacuum energy,

![Figure 7. A physical model for IR-assisted FD process. (Redrawn with permission from Bae et. al., 2010).][98]
Table 2. Drying time, drying rate, vacuum energy, and energy consumption for different IR assisted FD for the preparation of banana slices.

| Drying method          | Total drying time (min) | Drying rate (g/hr) | Vacuum energy (J) | Electrical energy consumption (×10^3 MJ) |
|------------------------|-------------------------|--------------------|-------------------|-----------------------------------------|
| FD                     | 696                     | 3.3                | 680,160           | 27.0                                    |
| IRAFD-2.7 kW/m^2 at 20% WR | 330                   | 7.0                | 324,511           | 13.0                                    |
| RAFD-2.7 kW/m^2 at 20% WR to 4.0 kW/m^2 at 0°C | 294                   | 8.0                | 288,107           | 11.6                                    |
| Continuous IRAFD-2.7 kW/m^2 | 213                   | 10.9               | 209,340           | 8.4                                     |

Source: Reproduced with permission from Khampakool, Soisungwan and Park, 2019.

and electrical energy consumption of FD and various trials involving IR-assisted FD. Energy consumption relies on the time required for drying the food samples. The highest electrical energy consumption of 27 MJ was required for FD as it had the longest drying time of 11.5 hours. However, IR-assisted FD had evident reduction in electrical energy consumption as the process involved reduced drying time owing to improved drying rates. Continuous IR-assisted FD (2.7 kW/m^2) process consumed less electrical energy, followed by IR-assisted FD (2.7 kW/m^2) at 20% WR to 4.0 kW/m^2 at 0°C and IRAFD-2.7 kW/m^2 at 20% WR. Continuous IR-assisted FD showed reduction in drying time up to 210 minutes, with 70%-time savings.

The IR component of the EM spectrum is further subdivided into near IR (NIR, wavelengths between 0.75 and 1.4 μm), mid-IR (MIR, wavelengths between 3 and 8 μm), and far IR (FIR, wavelengths between 15 and 100 μm). In a study conducted on pears, using FD and mid-IR-assisted FD, products processed using the latter technology exhibited softer texture, better rehydration capacity, and improved retention of phenol content and antioxidant activity, as compared to conventionally FD pears. IR-assisted FD is a promising approach for producing high-quality dried products at a relatively lower cost.

Microwave-assisted freeze-drying

Microwave (MW) radiation represents the part of the EM spectrum with frequency ranging between 300 MHz and 300 GHz (1 mm to 1 m wavelength). Microwave drying generates heat directly inside the product which leads to higher internal heating. This is similar to IR heating but penetration distances are typically much higher. The application of MW technology to FD first began in the 1950s and became more widespread in the 1970s. Microwave Freeze Drying (MFD) has been a topic of development since then, due to its obvious potential for providing heat energy to sublimating materials in a vacuum system. For example, Ma and Pelte were able to freeze-dry 1.5 cm thick beef slices, and ¼–¾ inch beef cubes with the help of MW. It was known from these early studies that localized sample overheating and corona discharges were issues that limited the power that could be applied. It is critical that melting is avoided as once part of a sample melts, the increased absorption of MW of water compared to ice results in even more sample heating and melting. The focus is on the dielectric properties of food materials and the use of MW to assist FD is perhaps better studied than other techniques.

Since then a large number of studies have been published highlighting the potential of MFD to provide products of high quality at reduced drying time and cost. Studies on sea cucumber, button mushrooms, re-structured mixed potatoes with apple chips, banana cubes, and okra snacks showed that drying time requirements for these products when processed with MFD are significantly shorter than conventional FD. Despite its unique advantages, there exist certain challenges with MFD as it can cause ice melting and overheating due to non-uniform temperature distribution. To solve this problem in MFD, Wang et al. designed MW-assisted pulsed fluidized bed freeze-drying (MPFFD) system. In MPFFD, MW heating is given to the FD sample and it is pulsed at the same time, providing uniformity in drying, and improvements in product quality (Fig. 8). Recently, the effects of using MPFFD on the quality of Cordyceps militaris
and stem lettuce slices were studied. In these studies, researchers concluded that MPFFD samples dried more evenly than the samples dried in steady-spouted beds. In MPFFD, drying time requirements were around 72% lower in comparison to FD; however, for stem lettuce slices a more modest reduction in drying time of only up to 20% was observed in comparison to MFD. Similar results were reported for banana cubes by Jiang et al.\cite{108} Recently, a comparative study was conducted using five different drying methods: hot air drying (AD), FD, microwave vacuum drying (MVD), hot air drying combined with microwave vacuum drying (AD-MVD), and combined drying consisting of FD and MVD (FD-MVD) for the processing of functional okra snacks.\cite{11} As shown in Table 3, FD samples required the longest drying time of 41 hours with the lowest drying rate of 22 g/(hr·kg), followed by FD-MVD, AD, AD-MVD, and MVD. FD also showed the highest energy consumption of 76 kWh/kg H₂O. Whereas, FD-MVD had a relatively lower energy consumption of 21 kWh/kg H₂O. This study also concluded that FD-MVD can better retain antioxidants, color, texture, and sensory qualities, as compared to the other four methods. The potential of MVD for drying of okra snacks can be extended to other product ranges.

**Ultrasound-assisted freeze-drying**

In conventional drying, the application of US waves directly stimulates rates of heat and mass transfer and can lower drying times. US also induces ice nucleation during the freezing process.\cite{110} This approach has also been found to benefit rates of (non-conventional) atmospheric FD (AFD), which

**Table 3. Drying time, drying rate, and specific energy consumption for different MW-assisted FD methods for the preparation of functional okra snacks.**

| Drying method | Drying time (hr) | Drying rate [g/(hr·kg)] | Specific energy consumption (kW·hr/kg) |
|---------------|------------------|--------------------------|----------------------------------------|
| AD            | 8.2 ± 0.41       | 110.39 ± 5.52            | 49.2 ± 1.46                           |
| FD            | 41.23 ± 1.05     | 21.91 ± 0.56             | 75.85 ± 1.79                          |
| MVD           | 0.25 ± 0.02      | 3635.05 ± 290.8          | 4.38 ± 0.22                           |
| FD-MVD        | 10.16 ± 0.51     | 89.10 ± 4.47             | 21.3 ± 1.07                           |
| AD-MVD        | 2.15 ± 0.11      | 421.08 ± 21.54           | 10.75 ± 0.54                          |

Source: Reproduced with permission from Jiang et al., 2017.\cite{12}
uses very low humidity air to freeze-dry material. For example, Carrion et al.\textsuperscript{[111]} were able to dramatically shorten drying times for button mushrooms by up to 74%. Similar research was performed on the effect of US-assisted atmospheric FD to understand the effects on the antioxidant properties of eggplant.\textsuperscript{[112]} US treatments proved highly efficient in accelerating the atmospheric FD process, whilst providing improvements in the quality of eggplant. US, however, is more difficult to apply to conventional vacuum FD systems as US cannot be transmitted through a vacuum. It is nevertheless possible to transmit US to a freeze-drying material through the shelves using a technique called contact ultrasound, although the advantage is less obvious. Schössler et al.\textsuperscript{[110]} were able to reduce the drying times of red bell pepper cubes by only 11.5%.

**Conclusion**

The use of supplementary technologies has shown significant benefits when combined with conventional freeze-drying. Techniques can be classified into pretreatments (ultrasonic treatment, pulsed electric field, and osmotic dehydration), modification of freezing (ultrasonic nucleation, control of freezing rate, spray freezing), or direct application to the freeze-drying process itself (microwave, infra-red). However, these treatments have mainly been demonstrated on a research scale. There will be cost implications for use on a commercial scale but these may be outweighed by the potential savings in processing time, energy efficiency, and in some cases quality enhancements. In the future, more work should be targeted for developing continuous freeze-drying processes because the shift towards continuous freeze-drying provides several advantages in terms of process efficiency and product quality. With more studies on simplifying and optimizing the operations, such techniques can be taken up in commercial operations.

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