

## Achieving superior products using food freeze drying

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Water is generally the main 'ingredient' in food and has a considerable impact on both physico-chemical and mechanical properties, which constantly change during storage [2,5]. The products shelf life is limited by the biological activity of microorganisms and water deeply affects it. Bacteria and moulds easily grow and proliferate in humid conditions [2,5], resulting in quicker spoilage and organoleptic degradation [8]. However, not all water present in food has the same influence on the growth of microorganisms [1, 5]. In fact, interaction of water with the material structure can be different: part of it strongly interacts with the material matrix in a dynamic equilibrium, while the remaining, which is the majority, becomes more available for physical, chemical and microbiological reactions [2].

In order to reduce the water content and activity, drying is carried out. This process will have an impact on the final food attributes and quality and, therefore, it should be performed rationally.

The most simple and common drying method is air-drying, which relies on evaporation of water from the product at around 65-85°C, though industrial processes have been proposed at lower temperatures (~20°C), often in conjunction with vacuum technologies [4]. An increased apparent density, low porosity, microstructural damages and degradation of the product properties (e.g. aroma, colour and nutrients profile) characterise the resulting dried material [8].

Drying can be assisted by microwaves and radio frequencies that will produce fast heating due to the electrostatic interactions with water molecules. Although input energy can be tailored proportionally to the material moisture content [10], the food structure most likely damages and 'puffs' [4, 7].

Another common dehydration technique for liquid products (e.g. beverages) based on evaporation, is spray drying. The process consists in producing fine solid particles from atomised liquid droplets that pass through a hot convective gas. This technique is relatively cheap and fast. However, there are some disadvantages, such as powder caking, stickiness, nutrient degradation and structural damages with a subsequent loss in quality [3].

Freeze drying (lyophilisation) is the most widely used alternative to evaporative techniques. The process is based on ice crystal sublimation, allowing to achieve a high-quality product [9]. The process consists in freezing the product and decreasing both pressure and temperature below the water triple point (0.01 °C and 6.1 10-3 bar). The resulting sublimation at negative temperatures limits both chemical reactions (e.g. Maillard-browing reaction) and product degradation [8]. Pre-treatments in hypertonic solutions are often carried out to better preserve the structure on freezing and drying as well as to enhance the product appearance and rehydration [4, 6].

Once food is dried, it rehydrates directly in mouth with saliva or, it is reconstituted before consumption, such as ready meals, vegetables, pasta and cereal and food/dairy powder. Rehydratability is a key factor to consider after drying. The water to be recovered should be similar to the quantity before dehydration. To optimise and tailor the rehydration rate and extent, the correct freezing and drying parameters should be chosen. In fact, not only the chemical properties of the material are key for an optimal reconstitution, such as wettability and hydrophilicity, but also the resulting dried structure, mainly in terms of shrinkage, shape and porosity. It is accepted that freeze drying gives a great opportunity to achieve a premium food quality by designing both the freezing and drying steps.

In *Table 1* and *2*, impacts of common drying technologies on quality and process are reported.

Table 1: Impact of common food drying techniques on food quality

Table 2: Impact of common food drying techniques on food proce	SS
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		Air drying	Vacuum assisted drying	Spray drying	Freeze drying
	Process time	~	~	~	?
Pro	Equipment cost (Pilot Scale)	~	×	×	×
	Return of Investment (Product quality vs investment)	×	×	~	~
K	Continuous process	~	?	~	?
?	Negative Temperatures (Quality retention)	×	~	×	~
epends on pplication	Efficiency and yield	×	1	×	1
	Solid products	~	1	×	~

Freeze drying can be beneficial for a wide range of products, such as fruit and vegetables, dairy, pet food, beverages, meat, fish and seafood as well as nutraceuticals (e.g. sport supplements probiotics).

Since freeze drying produces superior products, the range of potential users has been grown exponentially to fulfil the request of military units and special forces during space and naval missions or in remote bases, tourism, athletes for extreme sports and expeditions, organisations such as UNESCO in places that are hit by disasters or conflicts, international humanitarian movements (e.g. Red Cross).

Biopharma has recently expanded their facilities to allow for food freeze drying, introducing services built on a quality by design (QbD) approach that aim to assist in achieving the customer requirements in terms of shelf life, appearance and physico-chemical properties. An initial characterisation of the thermal behaviour of the pre-lyophilised product is essential in order to design a tailored drying cycle and make the product more suitable for the process. Without it, the risk is to use a freeze drying process that dries the product, but not in a safe and efficient way, affecting irreversibly the quality. Technologies such as freeze drying microscopy (Lyostat), electrical impedance analysis (Lyotherm) and differential scanning calorimetry (DSC) are typically used to fully understand the food thermal behaviour. *Figure 1* shows an example of Lyostat and Lyotherm analysis results.





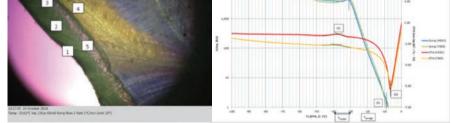


Figure 1: (A) Example of Lyostat analysis of fruit juice. The dried layer is formed at different temperatures (1, 2, 3). The collapse of the product starts at 5. (B) Example of Lyotherm graph of ice cream. Softening events are observed at Z1, whereas at Z2 and D2 there is complete melting

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Knowing the thermal stability of the product allows to prevent deleterious events such as boiling and 'puffing', shrinkage and collapse, clump formation, increased density, slowed sublimation kinetics, slowed rehydration, colour change and nutrient loss. After this thermal characterisation, food can be reformulated or treated with hypertonic solutions to improve the final appearance after drying as well as the mechanical and chemical properties. Finally, after the proof of concept and/or the process optimisation, the dried material is evaluated with post-process analyses (e.g. chemical and nutritional testing, moisture content and water activity analysis, microscopy, microbiological tests and mechanical characterisation). Figure 2 shows Biopharma Group's systematic approach.



Figure 2: Biopharma Group's systematic approach in freeze drying.

On completion of the product and process development, the customer benefits from a full report that can be applied to common production freeze dryers used by Biopharma and other Contract Manufacturing Organisations. It also contains key information regarding the most suitable packaging to achieve the required shelf life and stability.

For further information and to read the full article, please visit www.biopharma.co.uk

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