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# The use of lyophilization for fruit and vegetable preservation. A review

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**Abstract.** The lyophilization or freeze-drying (FD) is one of the most important technologies for obtaining dried food products and is mainly used to produce high-quality fruit and vegetable products. FD has two major drawbacks: low productivity (slow process, batch operation) and relatively high specific energy consumption, which has increased the need for improvements using innovative technologies. The main aspects of the physical basics and the three steps (freezing, primary drying, and secondary drying) are briefly presented. A new variant of FD, atmospheric freeze-drying, is also mentioned. A short overview is provided of the latest developments in FD techniques, highlighting their most characteristic and important features: how well they preserve the original texture, colour, nutritional value, and rehydration capacity. The review also discusses different coupled methods, including the integration of FD systems with other techniques such as ultrasound, microwave, infrared radiation, pulsed electric fields, and carbon dioxide laser perforation. The review evaluates the advantages and disadvantages of freeze-drying, discusses the expected quality of freeze-dried products, and identifies parameters that should be measured to assess the product's shelf life.

**Keywords and phrases:** freeze-drying, pretreatments, infrared and microwave heating, berry fruits

# 1. Introduction

Nowadays, approximately 80% of the world's population lives in environments where raw vegetables and fruits experience harmful changes within 10–30 hours, and meats within a few hours. Therefore, in many cases, preservation of food is necessary even when it is available fresh. One of the oldest methods of preservation is drying, which is achieved by reducing the moisture content of the material. In hot-air drying methods, moisture reduction occurs at temperatures higher than ambient air, requiring heat transfer to the product, which can occur through conduction, convection, or radiation. Currently, the gentlest drying method is lyophilization because lyophilized products have better quality than other dried products, as the temperatures used for water removal are lower, denaturation processes do not occur, and due to the gradually deepening sublimation, internal diffusion does not take place (*Salehi & Kashaninejad*, 2018; *Wang et al.*, 2019; *Bhatta et al.*, 2020).

# 2. Lyophilization

## 2.1. How does lyophilization affect product quality?

Food quality refers to the overall properties of food that make it suitable for fulfilling regulatory standards and consumer expectations. The most important requirements for food include edibility for human nutrition, nutritional value, biological value, sensory appeal, and food safety. Quality control determines the quality; it can reduce rejects and increase the production value at the manufacturing facility (*Al Faruq et al.*, 2025; *Csapó et al.*, 2016).

A fundamental requirement when measuring a specific quality parameter is to use appropriate testing methods and tools. When choosing these, key aspects include speed, simplicity, reproducibility, and accuracy. The testing method can be standardized, based on instrumental measurements, or non-standardized operational testing methods, which are suitable for application in production.

## 2.2. What processes occur during drying?

The physical and chemical changes within the material define the mechanism of the drying process. For agricultural and food industry materials, the modes of water binding, energy inputs, sorption relationships, drying rate conditions, heat and mass transfer processes, and changes caused by drying are of significant importance (*Almási*, 1977). The forms of water binding include: chemically bound water, adsorbed water, capillary-bound water, osmotic-bound water, and free water.

Drying only occurs if the moisture in the drying material can evaporate into the surrounding environment. If the vapour content of the surrounding gas exceeds that within the material, instead of drying, the material will gain water and become wetter. Whether during drying or wetting, the process continues until equilibrium is reached (*Dalgleish McNair*, 1990). At equilibrium, the vapour pressure of the water in the drying material equals the partial vapour pressure of water in the surrounding air. In this state, the moisture content of the drying material corresponds to the equilibrium moisture content (*Liapis & Bruttini*, 2014).

## 2.3. How to describe the heat and mass transfer processes in drying?

The moisture content and temperature of the material to be dried vary with location and time. If the drying is intensive, pressure changes within the material depend on position and time. In colloid-capillary-porous materials, the movement of liquid or vapour occurs under the influence of moisture and pressure gradients. Non-isothermal drying involves moisture migration driven also by temperature gradients. Molecular diffusion, influenced by increased trapped air pressure and capillary suction, occurs in the opposite direction of heat flow (in the direction of the heat gradient).

The moisture gradient is initially large and decreases significantly as the moist zones penetrate deeper. Its importance can be explained by the uneven migration of moisture within the materials, causing the interior to swell while the exterior shrinks. In cases of high moisture gradients, surface cracking and fragmentation of the material can occur (*King*, 1970).

The vapour pressure of solid and liquid materials depends on temperature: they increase exponentially with temperature. It is characteristic of the equilibrium established between condensed phase and vapour. When a liquid or solid is placed in a closed, isothermal environment, evaporation or sublimation causes the vapour pressure to increase until a dynamic equilibrium is reached between the phases. During this process, the same number of molecules transitions from one phase to the other per unit time, so evaporation or sublimation rate equals condensation or deposition rate. Temperature changes affect both directions simultaneously; at equilibrium, their rates become equal, but the quantity of vapour phase shifts in favour of vapour as the vapour pressure increases (*Lombrana*, 2009).

## 2.4. Processes in drying and wetting operations

During normal drying processes, part of the moisture from the material is transferred to the air and removed through air flow. During conditioning, the target moisture content is achieved by contacting the material with moist air. Both drying and wetting involve material transfer between two phases: one is the air, and the other is water trapped within the cells, pores, or particles of the solid material. The transfer processes include moisture migration within the solid material, transfer at the interface between the solid and the drying air, and the movement of water vapour molecules in the air (*Lorentzen*, 1975).

Simultaneously with the material transfer, a significant heat flow occurs in the opposite direction, because evaporation is an endothermic process involving heat removal. The solid material cools down, leading to heat flow from the warmer drying air into the cooler material. This process consists of the following sub-processes: convective heat exchange in the air, heat transfer at the interface between air and the drying material, and conduction within the drying material itself. Both material transfer and heat transfer processes are heavily influenced by the thin, stationary boundary layer of air at the phase separation surface. Through this layer, molecular diffusion drives material flow, and conduction facilitates heat flow (*Mellor*, 1978).

## 2.5. What changes occur in the composition of food during drying?

Even with the gentlest drying method, it is impossible to fully preserve the original properties: the structure of the material is somewhat damaged, resulting in irreversible changes to the cell walls. In the case of vegetables and fruits, skin formation occurs, while nuts develop cracks and splitting. The application of heat can reduce the protein content of the original material, which also affects the taste. A characteristic change during drying is shrinkage, which deteriorates the product's aesthetic appearance. Regarding storage of dried materials, the moisture content after drying is of significant importance, as it influences the equilibrium moisture content (ERP). The ERP value determines the storage requirements for the product (*Morrison & Hartel*, 2007).

## 2.6. What drying methods are known? Why do we dry food?

Food industry materials are dried either to extend their shelf life or to facilitate subsequent processing steps. Drying is the opposite operation of moistening. Moistening involves adjusting the moisture content of materials to a specified level. In most cases, the temperature of the material is also controlled along with its moisture content. The storage of food industry materials in a controlled environment with a specified temperature and humidity is called conditioning. Conditioning supports chemical and biological processes within the materials that are favourable for consumption or further processing (*Ratti*, 2008).

### *2.6.1. Convective drying*

Convective drying occurs as a result of the interaction between the material and the drying medium. The drying medium (usually warm air) transfers heat to the material due to its temperature, and the heat transferred carries away the moisture leaving the material. The balance between the heat supplied to the material and the moisture removed occurs in a flowing medium. The climate conditions that create convection are determined by the characteristics of the inlet side of the dryer. Convective drying processes can be classified based on the pressure prevailing in the chamber, the type of drying medium used, the mass flow relations, the flow of the material and the drying medium, and the construction and type of drying apparatus (*Ratti*, 2008).

### *2.6.2. Contact drying*

In contact drying, the moisture gradient within the material causes vapour to flow to the surface via conduction. The resulting thermodiffusion aligns with the direction of moisture movement, increasing moisture transfer. The thermal resistance and the heat flux supplied to the evaporation zone are significantly influenced by the layer thickness and its thermal conductivity, making contact drying advantageous for thin layers (*Ratti*, 2008).

### *2.6.3. Radiant heating*

In radiant heating, heat transfer occurs within the infrared wavelength range. In this process, photons absorbed by the atoms of the material generate vibrations that produce heat, initiating moisture migration from the interior. Different materials absorb radiation best at different wavelengths. Wet materials generally have a high reflection factor. Most of the internal radiation is absorbed by moisture beneath the surface, leading to moisture release under a vapour pressure higher than the ambient vapour pressure. The intensity and duration of radiation control the drying process. The radiation source and the mode of irradiation allow infrared rays to penetrate into the material, enhancing heat and mass transfer. To eliminate cracks and internal stresses caused by moisture gradients, oscillating irradiation is typically used (*Ratti*, 2008).

### *2.6.4. High-frequency processes*

During the high-frequency process, the material to be dried is placed between the plates of a flat capacitor. The water removal process resulting from this has three distinct stages: the first stage is heating, the second is the constant drying rate stage, and the third is the decreasing drying rate stage. Water removal begins when the high-frequency heating starts, partly due to capillary effects and partly because of the higher temperature of the internal core material. Due to the risk of high temperatures occurring, it is common to dry the material under vacuum. The specific energy consumption of this operation is higher than that of contact or convective drying methods (*Ratti*, 2008).

### *2.6.5. Freeze-drying (lyophilization)*

The term "lyophilization" (or "freeze-drying") refers to the rapid water-absorbing properties of the finished product produced with this technology. Therefore, it is more accurate to use the term "freeze-drying" or "sublimation drying" instead, but this term is highly anchored in industrial terminology. Freeze-drying indicates that the food is dried while frozen, and sublimation drying emphasizes the method of water removal, implying that the water in the food sublimates directly from ice to vapour without passing through the liquid phase.

By the end of the 20$^{th}$ century, lyophilization evolved from laboratory use to an industrial process. Freeze-drying is a modern and efficient technology suitable for producing high-quality products. It ensures long shelf life for foods and pharmaceuticals. These lyophilized products, packaged in moisture-proof, light-proof, and oxygen-free packaging, retain their quality for years. The biological activity of heat-sensitive materials is best preserved with this method, and using it improves the product's taste, texture, and appearance. Although freeze-drying is a time- and cost-intensive process compared to other drying methods, it is one of the gentlest techniques for heat-sensitive materials (*Ratti*, 2001).

The final moisture content of products will attain values between 1% and 4%, which prevents the growth of mould and bacteria, as well as the enzymatic biochemical reactions. During processing of high-moisture content material, initially the temperature of the material is lowered below the eutectic point until freezing, when solid state is reached. Subsequently, the solvent is removed from the frozen product by sublimation under appropriate pressure and temperature conditions.

#### 2.6.5.1. Physical basics of lyophilization

The lyophilization is a coupled process of ice formation inside the food material, during the freezing process, followed by the drying of the material by sublimation of the free water (primary drying) and the evaporation of the remaining bound water, at higher temperature (secondary drying, referred also as desorption drying phase (*Ma et al.*, 2023)). The evolution of the process parameters (pressure and temperature) during a typical freeze-drying process is represented in *Figure 1*.

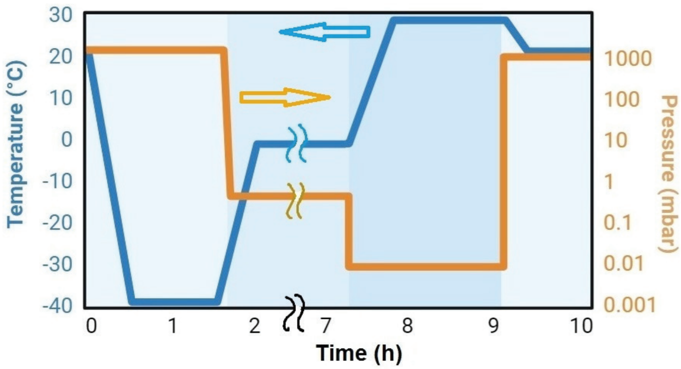


Figure 1. The variation trend of operation parameters (temperature and pressure) during a typical lyophilization process

The sublimation of ice requires that the single-component system (water) should be situated under the triple point ($t_{tr}$ = 0.0098 °C, $p_{tr}$ = 611.7 Pa), in conformity with the phase diagram of water (*Figure 2*).

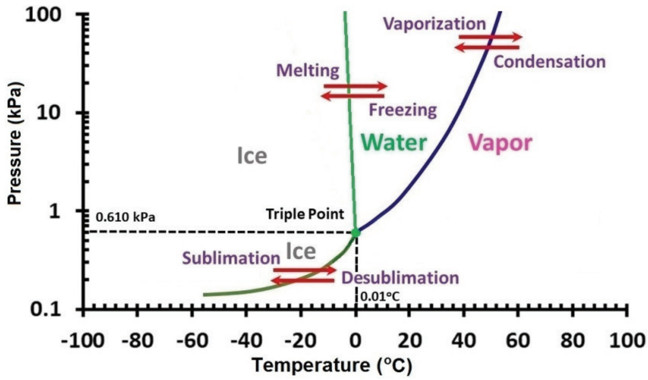


Figure 2. The phase diagram of water

The phase diagram suggests that under atmospheric pressure, the ice cannot sublimate, but that is contrary to the natural phenomenon that could appear under specific atmospheric conditions (*Jambon-Puillet et al.*, 2018). The explanation of this discrepancy is that the phase diagram is applied for pure water as single component, when the partial pressure of the water in the gas phase is equal to the total pressure of the system. When air is present, the partial pressure of the water could be very low (dry air), even at atmospheric pressure. This phenomenon was proposed firstly as a basis for a drying method in 1959 (*Meryman*, 1959) but firstly used in food industry in a Norwegian–Hungarian collaboration only in year 2005 (*Claussen et al.*, 2007). In classic vacuum freeze-drying (VFD), the food is frozen and then subjected to sublimation at low vacuum pressure.

In atmospheric freeze-drying (AFD), the sublimation is driven by a partial pressure gradient occurring between the drying air and the sample surface. This drying method is performed below the freezing point of the food sample under atmospheric pressure, using low temperature and dry air (*Naliyadhara & Trujillo*, 2025). Indeed, also in this case, the process parameters (temperature, water partial pressure) should be maintained under triple-point condition. The main drawback of the process is the prolonged drying time leading to ice thawing and product shrinkage (*Azizpour et al.*, 2025). Despite AFD saving about 30% of energy and that simpler equipment is needed (lack of vacuum system), in comparison even with FD, it is slower. The air temperature is in the interval of -10 °C and -3 °C, these values being a compromise between the quality of the products and the economic aspect of the process.

With infrared- or microwave-assisted heating, the process could be accelerated with approximately 50%. The utilization of ultrasound improves the mass transfer and could provide an average decrease of processing duration about 60% (*Xu et al.*, 2024). To increase product quality, a multistep temperature profile was proposed, including cooling, freezing, primary and secondary drying, termed as low-temperature drying. As quality factors, the degree of shrinkage and retention of L-ascorbic acid followed (*Nakagawa et al.*, 2021).

Therefore, to lyophilize a food product, its water content must first be frozen, and the water vapour pressure above it must be reduced below the surface vapour pressure. During sublimation, the system's temperature decreases, and without external heat input, the process would stop due to the phase change latent heat. The lyophilization process consists of two main steps: freezing of the material to be dried and sublimating the ice, which requires maintaining low water vapour pressure and supplementing the sublimation heat (*Snowman*, 1997).

In foods, water exists in various bound forms, and freezing occurs according to the mechanisms of multi-component solutions. It is characteristic that the solution does not freeze at a well-defined freezing point but over a temperature range, resulting in ice crystals and a eutectic mixture in the system after freezing. In some fruits, the eutectic point cannot be identified; the concentrated solution between ice crystals solidifies in an amorphous (glassy) state rather than forming crystalline structures. Materials with high $T_g$ are difficult to lyophilize because they become plastic under heat input and tend to foam and then collapse. Fruits with high fructose content fall into this category (*Csapó & Csapóné*, 2004). The glass transition temperature ($T_g$) has an important effect on the drying kinetics through internal mass transfer mechanism (*Joardder et al.*, 2024) related to the variation of the pore structure.

The size of ice crystals formed during freezing also influences the lyophilization process. Larger crystals are advantageous for sublimation but can negatively affect tissue structure. Rapid freezing produces small ice crystals along with the frozen solution; however, this results in longer drying times compared to materials containing larger crystals. Small crystals favour aroma retention because, after sublimation of the ice, the larger internal surface area and the formation of internal voids mechanically hinder the movement of volatile molecules. The fine crystal structure formed during rapid freezing provides better thermal conductivity towards the sublimation front but limits mass transfer, making it harder for water vapour to escape from the system. In the freezing process, the control of ice crystals' growth is crucial to reduce the structural damage of the cellular microstructure.

To achieve this, a small (in comparison with plant or animal cell) ice crystal formation is needed. This ice crystal size could be obtained only by homogeneous nucleation. For this reason, a variety of innovative freezing processes is developed, ultrasound-, microwave-, magnetic-field-, and high-pressure-assisted freezing procedures having great application potential in the food industry (*Loayza-Salazar et al.*, 2024). When the lyophilization process is conducted properly, the non-ice phase is left in the glassy state; consequently, following the ice crystal's sublimation, the cavities formed maintain the original form, without collapsing. Therefore, in the secondary drying phase, the temperature should be kept under the glass transition temperature ($T_g$) of the material (*Waghmare et al.*, 2022).

In cases when during the drying process a critical temperature ($T_c$) is exceeded, the structure may collapse, causing porosity reduction, which decreases the specific surface and, simultaneously, the vapour effective diffusivity through the porous material. The glass transition temperature ($T_g$) and critical collapse temperature ($T_c$) are closely related, as the $T_c$ could be 2–20 °C higher than $T_g$, depending mainly on the raw material composition (*Bhatta et al.*, 2020). The consequences are the increase of the duration of the secondary drying, deterioration of the structure of the product, and their rehydration capacity. Moreover, the higher moisture content of the final product may cause lower storage stability and lead to microbiological risks (*Nowak & Jakubczyk*, 2020).

The freezing equipment and the freezing method are primarily determined by the shape, size, and consistency of the starting product, that is, whether it is granular, solid, liquid, or paste-like. Freezing takes place in a device separate from the sublimator. Several freezing methods and devices exist to adapt to the starting material: fluidized bed freezer (also known as vortex bed freezer), freezing roll (coolant bath or refrigerant), freezing band (coolant bath), and freezing tray (cold air).

#### 2.6.5.2. The sublimation process

In normal drying, water leaves the system through direct transition from liquid to vapour, implying inner liquid transport trough capillaries. During sublimation drying, because the water is in a frozen state, solution transport is not possible. In this process, the sublimation front moves from the surface towards the interior of the material, meaning that as the frozen layer's thickness decreases steadily, the dry layer's thickness increases constantly. The vapour and heat transport must pass through this continuously growing dry layer during the operation.

The speed of sublimation and thus of lyophilization is determined by two transport processes: energy transport from the heating element to the sublimation front and water vapour transport towards the vacuum chamber or the ice condenser. Since these processes occur through the frozen or already dry layer of the food, the thickness of this layer is crucial for the process rate. Accordingly, the speed can be increased by reducing particle size and packing density (*Ratti*, 2001, 2008).

2.6.5.3. What are the technical conditions for lyophilization?

The fundamental technical requirement is a hermetically sealed chamber capable of maintaining low pressure conditions necessary for sublimation. The low pressure in the chamber is generated using a vacuum pump. A basic requirement for the pump is that it can produce lower pressure than the vapour tension within the frozen product. In industrial practice, the principle of multi-stage vacuum is commonly employed.

Sublimation must be maintained through continuous heat supply, and the water vapour produced during sublimation must be removed from the system or condensed to maintain pressure. Typically, a low-temperature ice condenser is used as a technical solution, where the condensed water reverts to ice (*Almási*, 1977; *Liapis & Bruttini*, 2014).

2.6.5.4. Technology of freeze-drying

During lyophilization, water leaves the food without altering its shape or components. Ensuring these conditions fundamentally depends on the preparation process, pre-freezing, the temperature during lyophilization, the low pressure created in the vacuum chamber, and the placement and temperature of the condenser. Therefore, the usability of the lyophilized product depends on the proper design of these technological steps and appropriate rehydration techniques. The phases of the process include: preparation for freezing, freezing of the food, and freeze-drying.

In the lyophilization phase, the sublimation of the frozen ice crystals occurs. Often, different lyophilization procedures vary only in the method of heating and the continuous removal of vapour. Heating can be radiative or conductive, and vapour removal can be achieved through direct suction, high-capacity pumps, adsorption or chemical binding, or re-freezing on a low-temperature surface.

Complete drying occurs when the temperature at the geometric centre of the material rises to match the heating surface temperature. The end of lyophilization is indicated by the temperature in the core of the material. When the ice crystals in the centre sublimate as well, the temperature of the material matches that of the heating plates, signalling that the process is complete (*Dalgleish McNair*, 1990; *Jafar & Farid*, 2003).

2.6.5.5. Practising lyophilization

The material to be dried is portioned onto metal trays after preparation and then placed into the lyophilizer, which is a large chamber, often with a transparent door that is airtight. After freezing the material, the air is evacuated from the container containing the product with a vacuum pump, creating a low-pressure environment. The latent heat of sublimation is supplied through contact, convection, radiation, or high-frequency heating. As a result, the ice crystals evaporate through the material towards the ice condenser. The temperature during this process is generally kept at a maximum of 20 °C in well-designed equipment. After the drying process – usually lasting 24 to 48 hours –, the vacuum is restored, and the dried product is removed from the trays (*King*, 1970; *Lombrana*, 2009). A typical industrial batch freeze dryer is represented in *Figure 3*.

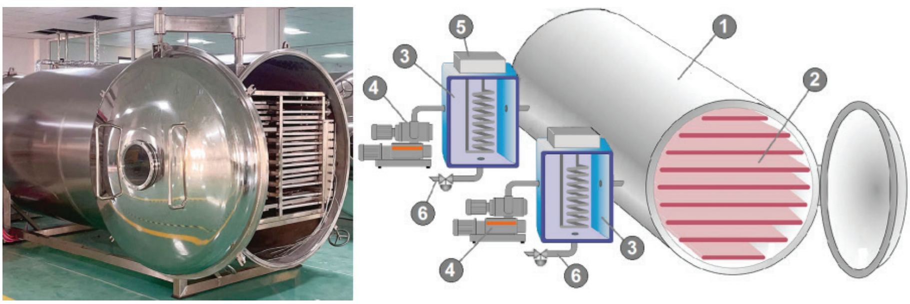


Figure 3. Industrial-scale vacuum freeze-dryer and its construction scheme

Notes: 1. drying chamber; 2. heating plates; 3. desublimation chamber; 4. vacuum pump; 5. cooling/de-icing cycle control unit; 6. water evacuation valve and tube.

To assure the uninterrupted evacuation of the humidity during one-batch drying, each freezing chamber is connected to two condenser (desublimation) chambers, working alternately in freezing/defrosting mode to ensure the cyclic evacuation of the liquid water from the installation (*Pedersen*, 2025). To obtain a product with required quality (porous texture without shrinkage and low residual moisture), besides cost-effectiveness, the value of process parameters (pressure and temperature profile) should be well known and precisely controlled (*Juckers et al.*, 2025). Moreover, these are closely coupled through the sublimation, as the process is highly endothermic. Furthermore, product temperature is the function of sublimation rate, chamber pressure, as well as time-variable heat transfer coefficients, as the thickness of the ice layer changes during the drying process.

2.6.5.6. What are the advantages and disadvantages of lyophilization?

Advantages include that it is a sterilization-capable method, since microbial growth ceases below a water activity of $a_w = 0.6$, and the products obtained this way have a water activity of $a_w = 0.2$. During the process, proteins are either minimally damaged or remain intact, and flavour compounds and vitamins are preserved as well. The product retains its original shape, colour, and texture. Rehydration of these products is faster and more complete compared to traditional drying methods. Properly treated materials can be stored for years under suitable conditions, and due to weight reduction, transportation costs are decreased.

However, there are several challenges in the industrial implementation of lyophilization techniques, among which perhaps the most significant is the control of sublimation heat. Additionally, compared to conventional drying, a lower productivity could be resulted, and the preparation of products incurs additional costs on top of the already significant energy requirements of the technology. Designing, building, or purchasing a lyophilizer involves substantial financial investments. Therefore, it is used primarily for preserving or producing food products where preservation costs constitute only a small part of the product's price, and where quality is the top priority (*King*, 1970). Since in a vacuum environment no convection occurs, the heat and mass transfer are extremely slow, resulting in a long production cycle with high energy consumption and costs generally 4-6 times higher than that of hot air drying (*Sun et al.*, 2019).

2.6.5.7. What will be the quality of lyophilized products?

Freeze-drying is considered one of the gentlest preservation methods, and it is regarded as the most delicate among the drying processes. During lyophilization, vitamins, minerals, aroma compounds, and nutrients remain virtually unchanged. As a result, lyophilized foods have high nutritional value. Although some microorganisms survive during freeze-drying (except in some cases), their growth on low water activity products is impossible, and the microbiological status of lyophilized foods remains unchanged (*Donno et al.*, 2025).

Vitamins withstand lyophilization without loss. However, merely preserving vitamin content during drying is not sufficient; the product is considered acceptable only if it retains its quality during storage. Lyophilized products can be stored at ambient temperature. Some researchers suggest that residual moisture content and oxygen in the packaging environment can influence the degradation of vitamin C, and thus nitrogen atmosphere packaging is recommended. During the process, the contents of vitamins A, $B_2$, and E are also largely preserved, and after rehydration, the taste of the products is close to that of fresh fruit (*Donno et al.*, 2025).

At low pressure, the decrease of the boiling point of the aroma compounds (mainly esters, lactones, and aldehydes) appears, but the aroma loss is lower in comparison with classic hot air drying, mainly due to the considerably lower processing temperature. This phenomenon is given by other physical factors, as follows. The increased relative volatility of the aroma compounds relative to water would increase the aroma loss by distillation only when liquid water is present in the system. As in FD the water is in solid form, this phenomenon does not increase significantly the aroma losses in the primary drying phase, but it does increase significantly the aroma losses during the secondary drying period. Moreover, during hot air drying, the structure of the plant material is more deteriorated. Water removal causes product damage in two ways: by steam formation, the cell and tissue structure are disrupted, and the capillary force of the water meniscus in the porous structures of the product leads to shrinkage or structural collapse during water vaporization (*Nakagawa et al.*, 2021).

In freeze-drying, when the freezing process is properly conducted, structure deterioration is minimal. Some exceptions still exist, such as the case of waxy skinned berries (e.g. blackcurrant, blueberry), when the peel barrier – without special pretreatment – can lead to serious structure collapse (*Pedersen*, 2025), followed by increased aroma loss. Conversely, it is true that during the freeze-drying process, a significant portion of these compounds remains, although the processing technology and especially the ripeness of the product before freezing can influence significantly the aroma profile of the final product. In most cases, the aroma of the final products is more intense than that of the initial raw material due to concentration increase, even if the minor aroma loss appears during the process.

In terms of colour, it is generally observed that slow freezing results in a darker appearance of the product, while rapid freezing produces a lighter colour. However, the colour of the finished product appears duller compared to fresh products, often due to water loss and light refraction changes. This phenomenon disappears in rehydrated products (*Ratti*, 2001, 2008).

#### 2.6.5.8. What is the role of water activity in preserving the quality of lyophilized foods?

The most important parameter for food preservation (next to temperature) is water activity (*Chirife & Fontana Jr.*, 2020). The water activity of food is related basically to the chemical potential of the water inside the food material. At equilibrium, this potential is equal with the chemical potential of the water in the environment, being a temperature-dependent property of food. Water activity is a well-established parameter for both microbial growth inside the foods and glass transition temperature (*Roos*, 2020). Among the characteristic microorganisms responsible for food spoilage, bacteria require the highest water activity ($a_w$), generally needing $a_w \approx 0.95$ for their metabolic functions. Yeasts function normally within the range from $a_w = 0.88$ to 0.95. Moulds and some halophilic bacteria operate within a water activity range from $a_w = 0.74$ to 0.88. One group of yeasts, osmophilic, can function at water activities in the range of $a_w = 0.69$–0.74. Some xerotolerant (xerophilic) mould strains that are particularly drought-tolerant can sustain certain metabolic activities in the range of $a_w = 0.65$–0.69. When water activity drops below $a_w = 0.65$, osmophilic yeast strains can still function, but at $a_w = 0.60$ activity is at the lower limit of their viability. Water activity also influences microbial thermal death (*Csapó & Csapóné*, 2004).

Among the biochemical processes leading to food spoilage, the activity of native enzymes in plant tissues is strongly dependent on water activity. Enzymatic activity reaches its maximum at around $a_w = 0.90$, then rapidly declines to about $a_w = 0.75$, and decreases slowly at lower water activities, reaching its minimum near $a_w = 0.4$. The inactivation of enzymes by heat is also dependent on water activity; the rate of inactivation decreases with lower water activity. In freeze-dried products, the water activity level, depending on the product, is less than $a_w = 0.2$ (*Csapó & Csapóné*, 2004*; Bhatta et al.*, 2020).

#### 2.6.5.9. Application of lyophilization technology in the food industry

Freeze-dried products have a significantly lower weight, and the loss of dry matter is considerably less compared to other preservation methods. Lyophilization is used only for products where high quality is essential, and the costs of preservation constitute only a small part of the final price. Examples include coffee extracts, tea infusions, instant soups, nutritional powders, fruit used for flavouring cereals, and space foods for astronauts.

Currently, freeze-drying is an expensive, time-consuming, and elaborate process. The equipment cost is roughly three times higher than that used for other drying methods, and it requires substantially more energy. Therefore, this method is mainly used for the dehydration of heat-sensitive materials.

Some foods can be very well freeze-dried, but not all foods are suitable for this process. Freeze-drying is typically applied to coffee, fruits, fruit juices, vegetables, herbs, food flavours, fish, seafood, eggs, and dairy products. Different types of foods, including multi-ingredient dishes like stews or soups, can also be freeze-dried.

As a result, freeze-dried foods are suitable for outdoor activities, travel, camping, military nutrition, survival food storage, and space exploration. Larger food items (such as fruits or meats) usually need to be cut into smaller pieces before freeze-drying. Heat-sensitive foods like fruits are especially suitable for freeze-drying and have become popular as snacks or as ingredients in breakfast cereals. Meat and seafood must be cooked before freeze-drying, and it should be noted that for some foods freeze-drying is not cost-effective (*Kerekes et al.*, 2008).

2.6.5.10. What does primarily influence the long-term preservation of your freeze-dried product?

Freeze-dried products are typically crispier and have a rehydration rate four to six times higher than traditional air-dried foods. The key advantages of freeze-drying include significant recovery of volatile compounds, preservation of the structure and surface, high yield, long shelf life, and reduced weight for storage, transportation, and handling. Besides the duration of the freeze-drying process, quality factors such as packaging method, packaging material quality, and storage time also play a crucial role. The packaging material must be an excellent barrier to water, oxygen, and aromas because if vacuum packaging allows water or air to permeate, it can significantly degrade the food's quality (*Dalglish McNair*, 1990; *Csapó & Csapóné*, 2004; *Sagara et al.*, 2005).

For the shelf life of freeze-dried products, environmentally friendly and energy-efficient vacuum packaging is important. Water absorption measurements of freeze-dried samples can help determine the quality properties of various packaging materials and assess the suitability of storage conditions. Using water activity measurements provides a more precise understanding of the adsorption and desorption areas and timeframes of the freeze-dried products. These data allow us to determine the shelf life, proper storage methods, and necessary packaging for different types of freeze-dried foods (*Csapó & Csapóné*, 2004).

So, what are the properties and factors that influence decisions about how long a freeze-dried food can be stored, and what components may change during storage? The most critical factor is maintaining a very low water activity ($a_w \approx 0.2$) because under these conditions neither chemical nor biochemical nor microbiological changes occur during storage. If the freeze-drying process was performed optimally, achieving this water activity, or even a lower one, is easily attainable. The next most important aspect is using packaging that can prevent water and oxygen ingress over an extended period. The packaging must be light-proof, as light energy can initiate various chemical reactions. It is advisable to use vacuum packaging or perform the process in a nitrogen atmosphere (*Dalgleish McNair*, 1990; *Kerekes et al.*, 2008; *Ratti*, 2001).

2.6.5.11. The parameters that should be measured to verify the product's shelf life

The parameters that should be measured to verify the product's shelf life and to determine whether any changes have occurred during long-term storage that could indicate a reduction in product quality are the following. The most important parameter is the equilibrium relative humidity, but even more crucial is the measurement of water activity. If the water activity is around 0.2, we can be confident that the lyophilization was successful, and no processes that could compromise the product's quality have taken place during storage. Measuring the dry matter content can further reinforce our conclusions drawn from water activity measurements.

In the case of fruits and vegetables, measuring the L-ascorbic acid (vitamin C) content can also be useful for estimating changes during lyophilization and storage. If the lyophilization technology is perfect and the packaging and storage conditions are optimal, the degradation or transformation of vitamin C will be minimal. However, in the presence of limited oxygen, the ascorbic acid would convert partially to dehydroascorbic acid and then to diketogulonic acid, and such transformations can indicate suboptimal storage conditions or long storage durations (*Csapó et al.*, 2020).

## 3. Freeze-drying of vegetables and fruits

After discussing the principles of freeze-drying, we turn to the freeze-drying of vegetables and fruits, as many fruits and vegetables are dehydrated to extend shelf life, preserve quality, and reduce packaging costs. Dehydration reduces transportation weight, preserves the original taste and sensory properties of the product, as well as nutrients, because during freeze-drying (FD) water is sublimated from frozen samples under low pressure without exposing the food to high temperatures (*Alves-Filho et al.*, 2006; *Chang et al.*, 2006; *Chu et al.*, 2021; *Hammami & René*, 1997; *Salehi,* 2023; *Naliyadhara & Trujillo*, 2025).

The main advantage of FD is its ability to preserve vitamins, nutrients, colour, aroma, and flavour, maintaining most of the food's original properties. Heat-sensitive, biologically active compounds remain intact, rehydration rate is higher, and the volumetric density of the resulting product is lower (greater porosity). These properties are especially important for fruits and vegetables. This process results in the higher preservation of phytochemicals and bioactive compounds (*Bhatta et al.*, 2020; *Grace et al.*, 2022; *Coşkun et al.*, 2024).

A disadvantage of the technology is that FD takes longer to fully dry, which results in higher energy consumption and higher costs (*Al Faruk et al.*, 2025). Newly developed methods and various combination techniques accelerate drying and save energy. FD is combined with technologies such as ultrasound, microwave, vacuum, pulsed microwave vacuum, $CO_2$ laser perforation, pulsed electric field (PEF), and infrared radiation (*Duan et al.*, 2007; *Cui et al.*, 2008; *Jiang et al.*, 2013, 2021; *Lin et al.*, 1998; *Rodrigez et al.*, 2015; *Wang et al.*, 2018; *Giancaterino et al.*, 2024; *Zudana et al.*, 2025).

Banana cubes dried with pulsed microwave vacuum drying showed that their vitamin C content was similar to that of the original material and significantly higher than that obtained with simple microwave treatment. The FD technology combined with infrared radiation reduces energy usage and drying time (*Kar et al.*, 2003; *Wang et al.*, 2013; *Jin et al.*, 2017; *Wu & Zhang*, 2019). The infrared-heating-coupled FD of pears results in the considerable decrease of drying time (between 14% and 43%), with higher rehydration capacity and better chemical profile of the final product (*Antal et al.*, 2017).

## 3.1. Vegetable drying

### *3.1.1. Potatoes*

The FD system is excellent for the sublimation drying of frozen materials, including potatoes, under vacuum. The combination of vacuum and microwave FD can produce a product comparable in quality to vacuum FD using conductive heating. Different drying techniques affect the properties of potato powder and the quality of fresh pasta made from it. FD creates numerous pores and channels within the starch granules, disrupting the ordered structure of the starch, which increases its digestibility and bioavailability in the human body (*Kuan et al.*, 2016; *Bao et al.*, 2021; *Lin et al.*, 1998; *Liu et al.*, 2018; *Bhatta et al.*, 2020).

### *3.1.2. Mushrooms*

Biologically, mushrooms are the fruiting body of the fungi. They are neither plant nor animal raw material, but in the food industry (and gastronomy), given their numerous – mainly textural, nutritional, and processing – similarities, they are included in the vegetable category (*Feeney et al.*, 2014). Freshly harvested mushrooms are highly perishable due to their high-water content, so they can only be stored for a short period. Drying is one of the best preservation techniques for the long-term storage of mushrooms. During FD of shiitake mushrooms, significant colour changes were observed in microwave-treated samples, whereas only minimal colour changes occurred in untreated samples. Freeze-dried mushrooms showed maximum rehydration rates and protein content, with a better preservation of minerals and improved biochemical properties compared to air-dried or microwave-dried samples (*Pei et al.*, 2013; *Rodriguez et al.*, 2005; *Wang et al.*, 2015; *Salehi et al.*, 2019; *Jia et al.*, 2024).

### *3.1.3. Tomato*

Examining the flavour profile of freeze-dried tomatoes, it was found that the citric acid concentration was 25-30% higher in freeze-dried samples compared to sun-dried and oven-dried samples. Studies on drying and rehydration revealed that during freeze-drying, the total ash content, sugar, reducing sugar, ascorbic acid, lycopene, and β-carotene levels in tomato powder were higher, and the brightness, redness, and yellowness indices were also greater compared to hot-air-dried tomato powder (*Chang et al.*, 2006).

### *3.1.4. Carrot*

When applying freeze-drying (FD) to carrots, the functional food components are fully preserved, and nutrient loss is negligible. Ultrasonic osmotic dehydration was investigated, and it was found that pre-treatment improved the retention of carotenoids and β-carotene. A control system was developed, and during the microwave FD treatment of carrots, it was determined that dynamic microwave FD could improve drying efficiency compared to the original FD process (*Cui et al.*, 2008).

Research on the effects of osmotic dehydration and hot-air pre-drying on the quality of freeze-dried carrots showed that pretreatment with osmotic solutions and hot air significantly improved the red colour index, yellowness index, and radical scavenging activity of the samples. Freeze-dried carrot sticks pretreated with sucrose had higher β-carotene levels (*Lin et al.*, 1998), the retention of carotene during FD being quite high in general (*Ma et al.*, 2023), which is why this method is recommended for carotene-containing vegetables.

### *3.1.5. Cabbage*

In the case of cabbage, microwave-assisted FD can significantly shorten drying time compared to conventional FD, resulting in a product of better quality than when treated with traditional methods. Cabbage dried via FD showed minimal shrinkage, colour retention, and preservation of flavour compounds in the final product (*Duan et al.*, 2007).

### *3.1.6. Spinach*

For spinach, FD is an appropriate drying method for preserving heat-sensitive components and vitamins. Studies on the effect of FD on spinach's nutrient concentration, bioavailability of lipophilic compounds, and hydrophilic flavonoids indicated that freeze-dried or spray-dried spinach, in spinach juice, fruit juice, and spinach pieces, best retains lipophilic and total flavonoid content (*Grace et al.*, 2022).

### *3.1.7. Okra*

The physical and chemical properties of okra powder produced by hot-air oven drying, microwave vacuum treatment, and FD were examined. It was found that

FD-produced okra powder exhibited the highest viscosity, total phenolic content, total flavonoid content, and antioxidant capacity. For okra drying, vacuum FD was used to measure drying rate, efficiency, and physicochemical quality. The rehydration curve for vacuum-FD-processed okra was higher than that of okra dried at 25 °C, and similar results were obtained with ultrasound-assisted vacuum drying at 100 °C for 12 minutes (*Wang et al.*, 2019; *Al Faruq et al.*, 2025).

## 3.2. Fruit drying

### *3.2.1. Blueberry*

FD (freeze-drying) is considered the optimal method for fruit and vegetable drying because it ensures that products remain functional, as the phytochemical components of the foods do not undergo significant changes during the process (*Csapó et al.*, 2016). In the case of red raspberry and blueberry, compared to hot-air drying, FD improved the preservation of phytochemical compounds during drying, and in several cases even resulted in higher phytochemical levels, mainly polyphenol content, increasing simultaneously the total antioxidant capacity (*Akcicek et al.*, 2025). For ultrasound-pretreated blueberries, examining the effect of chitosan coating, it was found that both ultrasonic treatment and soaking in sodium bicarbonate solution had positive effects on the properties of blueberries produced by FD (*Yu et al.*, 2017; *Bhatta et al.*, 2020).

When the blueberry skin was perforated with a carbon dioxide laser before the FD process, the drying time for blueberries decreased from 17 hours (untreated samples) to 13 hours. The $CO_2$ laser perforation improved both product quality and mass transfer rate. When ultrasonically pretreated and frozen blueberries dried using vacuum infrared FD, with $CO_2$ laser perforation, ultrasound waves, and freeze-thaw preconditioning, these pretreatments reduced drying time, enhanced rehydration capacity, and decreased shrinkage values from 57% to 25%. In frozen-dried blueberries, $CO_2$ laser perforation and ultrasonic pre-treatment had positive effects on all phenolic and anthocyanin contents (*Jin et al.*, 2017). For berry fruits, a comprehensive study of different drying methods showed that the freeze-drying combined with innovative heating methods (infrared, microwave) and various pretreatments were the most efficient for obtaining high-quality products with acceptable costs. Furthermore, for process optimization and further cost reduction, an improvement of mathematical models of FD is needed (*Sun et al.*, 2019). This requirement is yet to be met, but a new study (*Juckers et al.*, 2025) has shown a significant improvement in terms of prediction accuracy (deviation between experimental data and model prediction was under 4%) regarding FD modelling.

Moreover, another recent publication proposes and validates a detailed mathematical model just for blueberry freeze-drying, showing that the blueberry skin is the most relevant mass transfer parameter (*Schenck et al.*, 2025). This is in good agreement with the experimental results, namely that any pretreatment or processing method that disrupts skin (peel) integrity enhances the mass transfer considerably. The mechanical puncture pretreatment of blueberry reduces the drying time from 5 000 to 2 000 minutes, decreasing the processing cost without product quality degradation (*Jakubczyk et al.*, 2025); also, ultrasound-assisted vacuum drying shortened the drying time with about 6% (*Akcicek et al.*, 2025).

### *3.2.2. Strawberry*

Studies on colour and volume changes, as well as drying kinetics of whole and sliced strawberries, have shown that FD treatment reduces the volume of whole strawberries by 8% and sliced strawberries by 2%. However, the extent of shrinkage is independent of the dehydration temperature. The combination of ultrasound and osmotic dehydration as pre-treatments, followed by freezing in a pulsed fluidized bed microwave, resulted in dried strawberries with increased moisture loss, improved drying rate, and shorter drying time (*Ciurzyńska & Lenart*, 2010; *Hammami et al.*, 1997; *Holzwarth et al.*, 2012; *Huang et al.*, 2009).

Pretreating strawberries with PEF (pulsed electric field) improved the microstructure and crispness of dried strawberries, preserved the shape and colour of the final products, and enhanced homogeneity and pore thickness. It was also found that PEF pre-treatment can be an effective low-energy process for improving quality, increasing rehydration capacity, and enhancing the mechanical properties of freeze-dried strawberries (*Jiang et al.*, 2021; *Lammerskitten et al.*, 2010, 2020; *Theplib et al.*, 2008; *Shih et al.*, 2008; *Shishehgarha et al.*, 2002; *Zhang et al.*, 2020; *Giancaterino et al.*, 2024).

### *3.2.3. Apple*

For apples, FD is suitable for water removal, resulting in higher-quality characteristics in the final product compared to other dehydration methods. The combination of microwave drying with vacuum and freeze-drying was highly effective for drying apple slices. The atmospheric FD method was also extended to apple cubes, where using temperature-programmed drying and slicing, the drying time was reduced by nearly half while maintaining product quality (*Cui et al.*, 2008; *Lammerskitten et al.*, 2019, 2020; *Parniakov et al.*, 2016; *Naliyadhara et al.*, 2025).

PEF pretreatment non-thermally destroys or inactivates microorganisms in fresh products, improving shelf life, preserving nutrients, vitamins, flavours, and the excellent sensory properties of the products (*Lammerskitten et al.*, 2019). Research has shown that PEF pretreatment can significantly increase mass transfer during FD, improve dried product quality, increase rehydration capacity, reduce shrinkage, and lower energy consumption and processing costs. Using vacuum FD, pretreating apples with PEF enhanced pore size and rehydration ratio in dried apple slices, while the final product maintained its shape due to low shrinkage and large tissue pores. PEF pretreatment of apple tissues had a significant impact on FD efficiency, reducing drying time by 57%, increasing crystallinity by 35%, improving moisture diffusion coefficient by 44%, and boosting rehydration capacity (*Wang et al.*, 2018; *Wu & Zhang*, 2019; *Zudana et al.*, 2025).

### *3.2.4. Peach*

When examining the use of ultrasound waves and cross-linking agents during osmotic dehydration, it was found that increasing the ultrasound treatment time reduced the drying time and shrinkage of yellow peaches. The pretreated samples had better rehydration properties, retained their colour and nutrient content more effectively, and the treatment further improved texture characteristics compared to directly dried pieces (*Chu et al.*, 2021).

### *3.2.5. Banana*

Studies on the microwave freeze-drying (MFD) properties of banana slices revealed that the drying time decreased with banana ripening. A microwave power of 2.0 $W \cdot g^{-1}$ was optimal for preserving the quality and sensory attributes of the final products. Sequential infrared irradiation combined with FD produced very crispy banana slices, and extra acidic soaking improved the product's colour and reduced the required drying time (*Kar et al.*, 2003; *Khampakool et al.*, 2019; *Wang et al.*, 2007).

Vacuum belt drying, FD, and air drying caused significant differences in the volatile compounds of banana powders. The volatile components were similar across the three products, but some compounds were only present in the freeze-dried product, while others were found only in the vacuum belt or air-dried products. The relatively high drying temperature of vacuum belt drying and hot air drying could damage key aroma components and lead to the transformation of aroma compounds. FD was the best technology, followed by vacuum belt drying, with hot air drying being the least suitable for producing good-tasting banana powder (*Jiang et al.*, 2013; *Khampakool et al.*, 2018).

Comparing the dehydration properties and uniformity of dried bananas obtained by pulse microwave vacuum drying, FD, and microwave FD, it was found that pulse microwave vacuum drying provided better uniformity than microwave FD. Compared to conventional microwave FD, pulse microwave treatment resulted in better sensory properties and higher ascorbic acid content, indicating better nutrient retention (*Kar et al.*, 2003).

The effectiveness of FD combined with infrared radiation for producing banana chips was studied, and it was found that the banana snacks were of excellent quality, with significant savings in drying time and energy consumption. This combined technique allowed the production of the crispiest banana chips (*Wang et al.*, 2007; *Khampakool*, 2018).

### *3.2.6. Orange*

Oranges contain large amounts of bioactive compounds, vitamins, carotenoids, flavonoids, and other polyphenols. Studies on the effect of FD at temperatures between 30 °C and 50 °C on bioactive compounds, physical characteristics, and sensory properties of orange-based products concluded that 50 °C is the optimal drying temperature for oranges. This temperature reduced drying time by 64%, preserved vitamin C content, and did not affect total phenol and carotenoid levels. Similarly, as in traditional drying, the vitamin C-loss is in close correlation with processing time and temperature (*Ma et al.*, 2023). In general, probably the antioxidants are well protected by the low pressure, given by consequent decrease in oxygen concentration. Additionally, it improved mechanical properties without impacting the flowability or rehydration rate of powdered products (*Kuan et al.*, 2016; *Uscanga et al.*, 2021; *Coşkun et al.,* 2024).

## 4. Conclusions

FD technology involves sublimation, removing water at low temperature and pressure. This method ensures the highest quality and consumer acceptance of dried foods. For preserving functional components, bioactive compounds (total antioxidant activity, total phenolic compounds, and total anthocyanins), FD is demonstrably the best method for fruits and vegetables. For many agricultural products, FD is among the top techniques for moisture removal and producing the highest-quality final products.

Compared to other drying methods, FD outperforms them, as evidenced by the more favourable composition, colour, and aroma of freeze-dried fruit and vegetable pieces. Despite its numerous advantages, FD is not widely used in the food industry due to higher costs compared to other technologies. However, pretreatments such as microwave, ultrasound, and infrared treatments, combined with vacuum, PEF, and $CO_2$ laser perforation methods, when used together with freeze dryers, can accelerate dehydration and significantly reduce costs compared to traditional FD technology.

The combination of freeze-drying with ultrasound-assisted freezing, microwave or infrared heating, also pretreatment of raw material with pulsed electric fields, or carbon dioxide laser perforation are new, innovative methods that could be applied in some special cases at industrial scale. Another important innovation, atmospheric freeze-drying (AFD), could have an important role in future technologies. Our final conclusion is that although the gentle drying of fruits and vegetables requires significantly more time, energy, and costs compared to classic drying methods, freeze-drying remains the optimal technology given the high product quality. The future development in energy recovery and optimized industrial scale-up may reduce the main drawbacks (long duration and high energy consumption) of this processing technology.

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