Introduction

Nowadays, the need to achieve long-term stability and ease of use, in addition to greater functionality, means that many food products and liquid or solid ingredients are dehydrated or mechanically transformed into powder.

The manufacturing processes of food powders always require meeting a series of criteria such as process performance (energy efficiency, production cost, workplace safety, and environmental pollution) and product quality (composition, preservation, food safety, functionality, etc.).

Food dehydration is the oldest method of food preservation. According to the travel accounts of Marco Polo, the Mongols produced milk powder by drying milk in the sun. Dehydration makes it possible to partially or totally remove the water contained in food. Reducing the availability of water (water activity, called “aw”) in food inhibits microbial growth, stops enzymatic reactions, and also reduces the weight and volume of the product, which represents significant savings in packaging, transportation, and storage. However, the cost of the operation limits its widespread application.

Dehydration can be carried out by:

- **Concentration**: Partial dehydration of the product aimed at increasing the mass of a product per unit volume. This can be achieved through partial dehydration. The osmotic pressure of the food prevents any microbial growth.
- **Dehydration**: The action of partially or totally removing the water contained in food.
- **Drying**: Removal of excess moisture through intensive dehydration by water evaporation. This results in so-called dry products. A very low water activity ($aw < 0.4$) prevents any microbial growth and enzymatic activity.
- **Freeze-drying (Lyophilization)**: Total dehydration by sublimation at low temperature and under vacuum.

1. Importance of Dehydration

Food dehydration can be used to concentrate products, as a partial preservation method, or for full preservation. Dehydration is a physical and natural preservation method that is simple, relatively mild, and economical. It allows easier transportation and greater convenience of use (instant mashed potatoes, coffee, etc.).

Dehydration can be applied to several food products through:

- **Partial removal of water**, which can serve as a preservation method if other favorable factors are present, such as a high sugar or salt content that reduces water activity. Examples: dried fruits (less than 30% water and about 70% sugars), sweetened condensed milk (less than 30% water and about 55% sugars).

- **Total removal of water**, which allows long-term preservation (free water and most of the bound water are removed, aw ≤ 0.4).

2. Physico-Chemical Consequences of Intensive Food Dehydration

Microbial growth is stopped, enzymatic activity in the medium is greatly slowed, as well as hydrolysis reactions, enzymatic browning, and lipid oxidation.

Dehydrated products are not sterile; any accidental rehydration will be harmful (hence the need for airtight packaging).

2.1. Reactions During Dehydration

Certain reactions may occur during dehydration:

- Evaporation of flavoring substances (volatile compounds)
- Oxidation of pigments
- Protein denaturation
- Non-enzymatic browning
- Lipid oxidation and vitamin losses

These reactions result in deterioration of nutritional and organoleptic quality.

Reactions may also occur during storage, such as oxidation and non-enzymatic browning.

Preliminary blanching of products and the addition of additives (such as sulfur dioxide) are necessary to prevent these reactions.

3. Food Dehydration Methods

Food dehydration techniques are diverse:

- **Mechanical method**: Dehydration occurs through momentum transfer. It involves concentrating products by centrifugation (tomato juice), draining (cheese), pressing (pressed cheeses), and ultracentrifugation (milk protein concentrates and egg white concentrates).
  The mechanical method is characterized by its limited efficiency (about 60% residual moisture) and lack of selectivity.
- **Thermal method**: Dehydration occurs through heat transfer. Drying can be carried out in air (about 25%) or industrially by removing water using heat from:
  - Convection of hot, dry air (spray process)
  - Conduction through contact with a hot surface (Hatmaker process)
  - Sublimation of ice (freeze-drying / lyophilization)

4. Spray Drying

Spray-drying technology appeared at the end of the nineteenth century and is still considered the main technology for powder production in various fields such as the chemical, food-processing, pharmaceutical, and microbiological industries. Spray drying is also one of the most widely used techniques for encapsulating food ingredients, essential oils, and flavors.

The main advantage of spray drying is the ability to directly transform liquid raw materials into dry powder. It is widely applied in milk powder production. Depending on the intended use, different types of powders are obtained according to the drying method used (powders for reconstitution, used in bakery, pastry, chocolate manufacturing, ice cream production, animal feed, etc.).

New powder production technologies have emerged, such as Controlled Instantaneous Decompression (DIC), which allows the production of very high-quality finished products. However, the applications of DIC technologies in food powder manufacturing remain limited.

4.1. Processes Used for Spray Drying

The systems used in the food industry for preservation by spray drying are described below:

4.1.1. Drum or Cylinder Drying (Hatmaker Process)

Drum drying was introduced into industry about 100 years ago. Drum dryers consist of one or more hollow metallic cylindrical rollers mounted on horizontal shafts (Figure III.1). The diameter of a cylinder can reach up to 2 m and a length of 5 m. Their capacities range between 5–30 kg of product per square meter per hour. The rotational speed varies between 1 and 30 revolutions per minute, and the residence time can range from 2 seconds to 1 minute. The thickness of the product layer can be controlled by carefully adjusting the gap between the two cylinders, ranging from 0.05 to 0.5 mm.

Current applications of drum drying include drying animal feed, vegetable purées, applesauce, precooked cereals, and dry soup mixes.

In this process, also called thin-film drying or roller drying, the product—generally in the form of suspensions, pastes, or concentrated viscous solutions—is spread as a thin film over the external surface of metal cylinders heated internally, usually by steam. The water contained in the milk evaporates by boiling and is removed by an air stream when it comes into contact with the hot surfaces of the drums.

The dried product is scraped off by a knife after one revolution and is collected in the form of a thin sheet or flakes. These are then broken up by passing through a screw conveyor to obtain a coarse powder. A final grinding process is carried out to achieve the desired particle size.

The high temperature denatures proteins and degrades product quality; the powder becomes less soluble. It is also very difficult to dry products rich in sugar such as fruit juices or whey. This process is mainly limited to liquid or pasty products and is not suitable for products high in fat content.

Two types of dryers are distinguished:

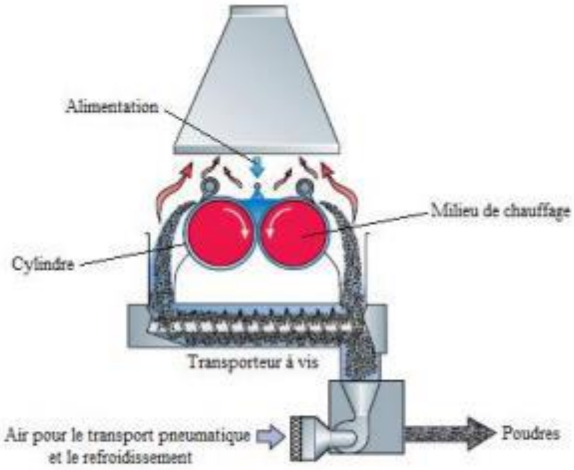


Figure 1: Principle of double-drum drying

a.1) Trough-fed dryer:
The pretreated product enters the trough. When the thin layer of product comes into contact with the hot surface, it is heated very rapidly. The water evaporates, and the layer of milk dries on the drum, forming a film that is regularly scraped off with blades. The dried milk falls onto a screw conveyor where it is reduced into flakes. Hard and burnt particles are separated by a sieve.

a.2) Spray-fed dryer:
Nozzles located above the drums spray a thin film of product onto the heated surfaces of the cylinders. The scraped powder undergoes the same treatment.

4.1.2) Spray Drying (Spray Process)

Spray-drying technology and equipment appeared at the end of the 19th century. Milk drying was the first application of spray-drying technology. Subsequently, spray-drying technology was continuously improved to better adapt to heat-sensitive products. The first recognized device was a spray dryer that used a pressure nozzle. The spray drying process Spray drying offers the advantage of directly transforming liquid raw materials into dry powder with very low moisture content.

Several spray drying systems or spray drying towers exist:

- **Open system:** Air is taken from the environment and heated in the heater. It performs heat transfer only once with the product in the drying chamber. It is then cleaned by the cyclone and the bag filter (Figure 2).

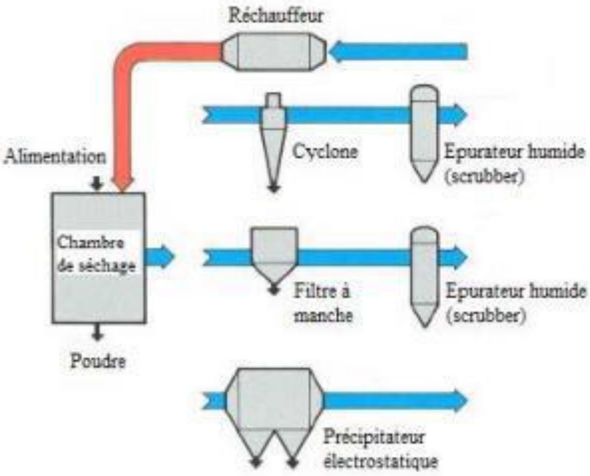
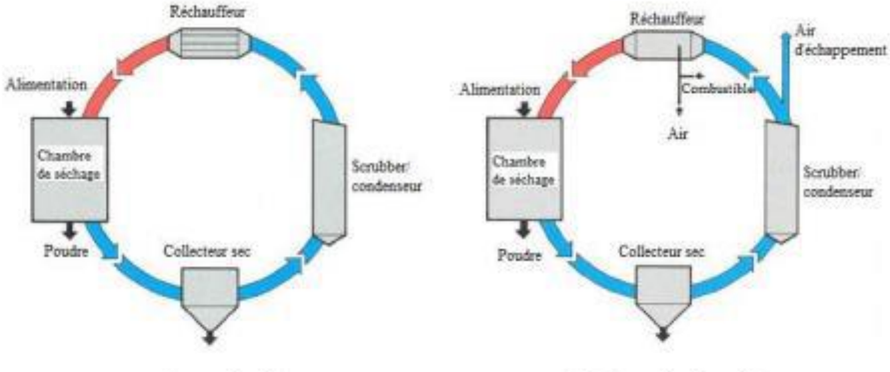


Figure 2: The open spray drying system

- **Closed system:** Also called a closed cycle, this system is used for products that are sensitive to oxygen. Drying is carried out in a sealed system using an inert gas (nitrogen).
- **Semi-closed cycle:** This system prevents fire and explosion risks by reducing the oxygen content in the drying chamber (Figure 3).
- **Aseptic cycle:** This system is characterized by the presence of air filters. It is installed close to the packaging chamber (Figure 4).



a. (Le cycle clos)

b. (Le cycle demi-clos)

Figure 3: Closed and semi-closed drying cycles

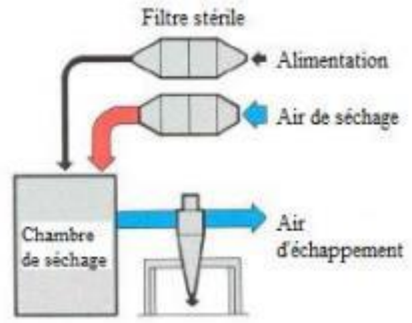


Figure 4: The aseptic drying cycle

Operating principle of the spray drying system

There are two typical types of spray dryers: the **vertical spray dryer** (or Cyclone type) and the **horizontal dryer** (or Rogers type). The design of the atomization equipment depends on the desired particle size and powder characteristics (texture, solubility, wettability, granular structure).

The operating principle of the two typical dryers is as follows:

a) Vertical spray dryer (Cyclone type)

This type of dryer (Figure 5) is very widely used. Its operating principle includes two main stages:

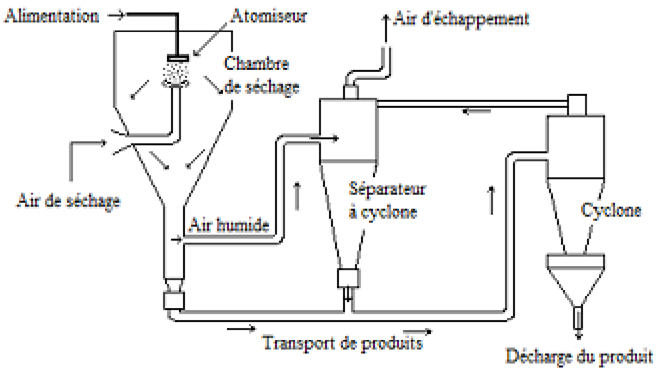


Figure 5: Vertical spray dryer

a.1) Product atomization:

The liquid raw material is pumped by high-pressure pumps to the atomizer (up to 200 bar) and sprayed into very fine droplets. The larger their effective surface area, the more efficient the drying process. An ideal atomizer is capable of producing individual fine droplets of uniform size. The rates of heat and mass transfer, as well as the drying time, are identical for all droplets, ensuring the uniformity of the dried product.

b) **Horizontal spray dryer (Rogers type)**

This configuration is less common and is used only for **sensitive powders** that cannot withstand the friction generated by the cyclone’s action. Powder dispersion is achieved using a **screw conveyor** (Figure 11).

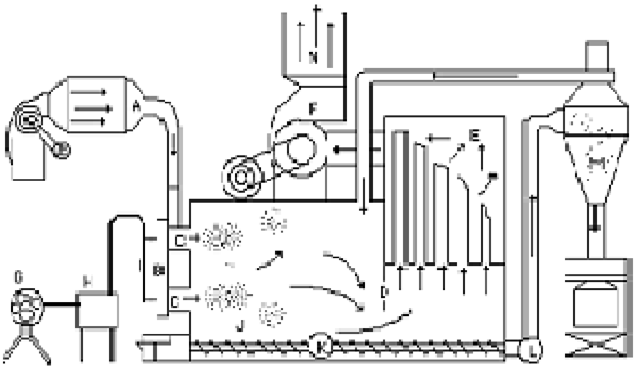


Figure 11: Horizontal spray dryer.
A: Pipe, B: Air distributor, C: Drying chamber, E: Filter, F: Fan, G: Heater, H: Pump, K: Screw conveyor.

- **Production of instant powder**

In the case of infant milk, it is important to obtain instant milk powders that allow very rapid dissolution. The milk particles must be processed to form larger, porous agglomerates (drying replaces water with air and particle humidification is done with water vapor).

The most effective instantization is achieved using a **fluidized bed** (Figure III.12).

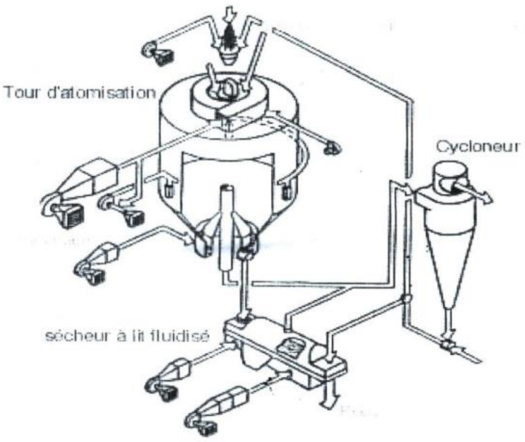


Figure III.12: Fluidized bed spray drying installation

4.2/ Factors Affecting Spray Drying Operation

Several factors can influence the efficiency of spray drying, such as product viscosity, atomizer orifice size, pressure, feed rate, solid content of the liquid, surface tension, moisture content, and more.

4.2.1/ Droplet Drying Time

The actual evaporation time of droplets at a fixed air temperature depends on the droplet shape, chemical composition, physical structure, and solids concentration. The actual drying time is the sum of the **constant rate period** and the **falling rate period** until the desired moisture content is reached. The general drying characteristics are illustrated by the **drying rate curve** (Figure III.13).

- **Phase AB:** The drying rate is established immediately when the droplets come into contact with the drying air. The droplet surface temperature increases slightly, and the drying rate rises within the milliseconds required for heat transfer across the droplet–air interface to reach equilibrium.
- **Phase BC:** Dynamic equilibrium conditions are represented. Drying progresses at a **constant rate**, which is the highest rate reached during the drying evolution of the droplets.

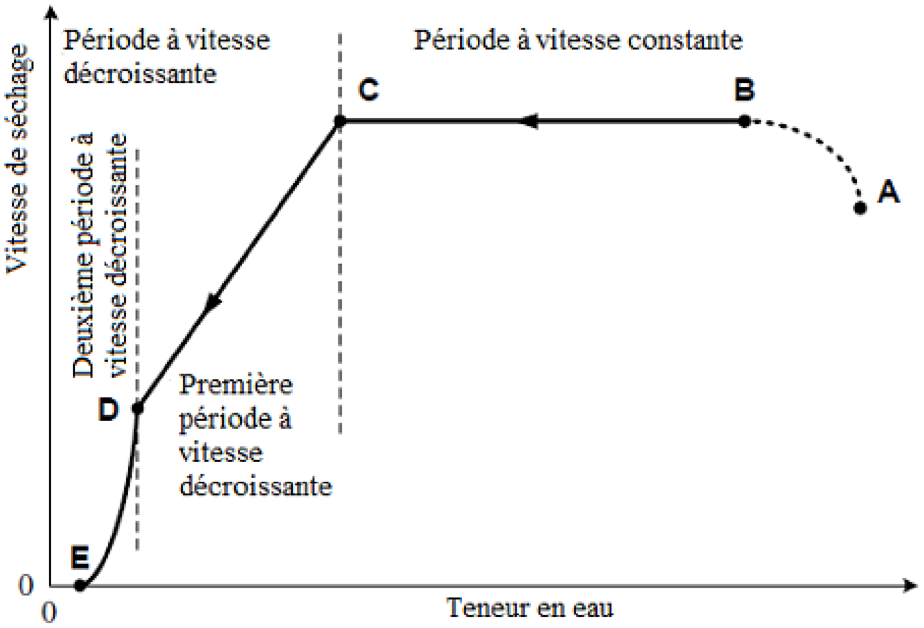


Figure III.13: Drying rate curve

5/ Advantages and Disadvantages of the Spray Drying Process

Compared to other powder drying techniques (roller or drum drying, pan drying, or mixer drying), spray drying has the following advantages and disadvantages:

5.1/ Advantages of Spray Drying Technology

- Converts liquid raw materials into dry powder with very low moisture content.
- Produces particles that are often regular, spherical, and have a narrow particle size distribution.
- Suitable for heat-sensitive materials.

- Short drying time.
- Capable of processing various products: solutions, emulsions, or pastes.

5.2/ Disadvantages of Spray Drying Technology

- Relatively high investment costs.
- Requires the evaporation of large amounts of solvent, which limits its use for low-value products.
- Requires highly skilled operators; otherwise, the product can easily degrade (loss of aroma and color).
- Difficulty in recovering 100% of the dried product.
- Regular hygiene and maintenance of the machines are essential.

All drying methods, whether drum drying or spray drying, rely on supplying heat to the product, but the characteristics of the resulting powder differ (Table 1).

Table 1: Properties of milk powder obtained by drum drying and spray drying (Maximum values)

Properties	Spray Drying	Drum Drying
Fat content	1,25%	1,25%
Moisture	4%	4%
Titratable acidity	0,15%	0,15%
Solubility index	1,25	15
Bacterial count	50000/g	50000/g
Burnt particles	15 mg	22,5mg

5- Freeze Drying (Lyophilization / Cryodesiccation)

Etymologically, the word **“lyophile”**, from the Greek roots *lyo-* and *-phile*, means “solvent-loving.” Indeed, a freeze-dried product appears solid, friable, and porous, and is characterized by a strong affinity for water.

Freeze drying, or cryodesiccation, is a **low-temperature dehydration process** that removes the majority of water from a product, reducing its weight to a minimum. This process allows **long-term preservation** by lowering water activity. Freeze drying produces **high-quality products**: the shape, appearance, and aromatic quality are well preserved. Additionally, freeze-dried products offer the advantage of **instant rehydration**, as the water is removed without disturbing the structure or composition of the cells.

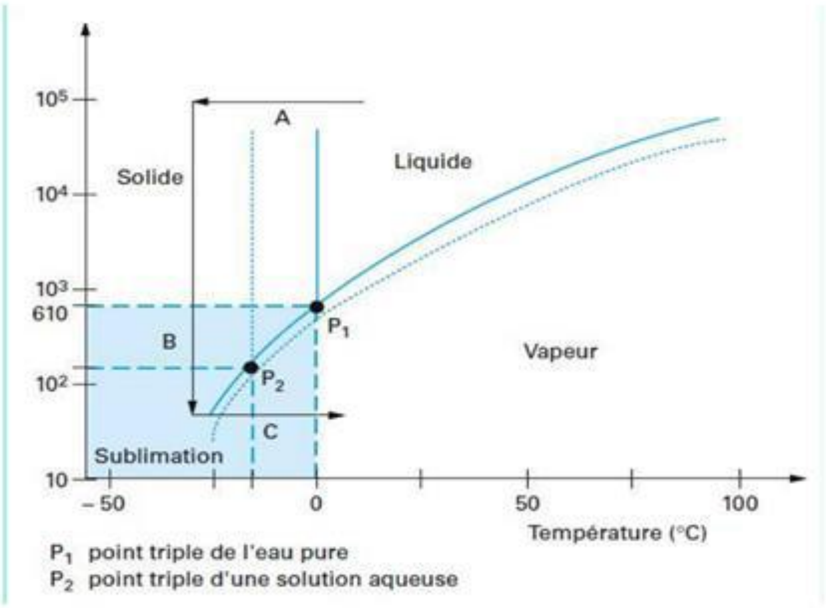
The **Incas of Peru**, around 1100 AD, were the first to use this method to dry meat. They took advantage of the particular climatic conditions of the high Andes: at high altitude, the air is cold and the pressure is low, so the meat dried by **sublimation**—a natural form of freeze drying at high pressure.

In 1906, the French physicists **Bordas** and **d’Arsonval** described a device capable of performing sublimation, based on the principle of **Wollaston and Leslie**, who had shown that ice could sublimate into vapor under very low pressure. Freeze drying was initially applied in **medicine and pharmacy** (vaccines, serums, etc.), and it was not until 1960 that it was applied in the **food industry**.

Due to its high cost, freeze drying is only used for **high-value products**, such as coffee, herbs, spices, prepared meals, meats, and seafood.

6/ Principle of Freeze Drying

Freeze drying takes advantage of a property of water: **below its triple point** (temperature 0 °C, pressure 4.6 mmHg) (Figure III.14), ice **sublimates**, turning directly from solid to vapor. The phase changes (solid–liquid–vapor) depend on both **temperature** and **pressure**.



A typical **freeze dryer** (Figure 15) has three main components: a **vacuum pump**, a **drying chamber**, and a **condenser**.

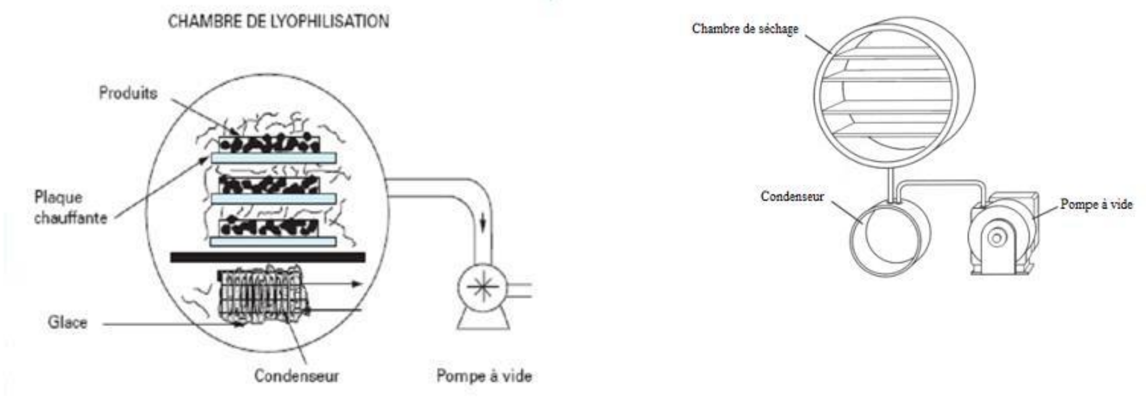


Figure III.15: Typical freeze-drying system

- **Drying chamber:** Contains the materials to be dried on trays and heating plates, which provide the energy required for the freeze-drying operation (sublimation and desorption) via heat transfer by conduction. The chamber is cooled by refrigeration systems.
- **Condenser:** When the ice begins to sublimate, the water vapor is transported through the drying chamber to the refrigerated condenser. Two types are available: a chamber cooled with a mechanical refrigeration system or a chamber cooled with methanol/dry ice or liquid nitrogen.
- **Vacuum pump:** Vacuum pumps remove air and water vapor from the drying chamber to create a vacuum. Pump capacity is important (the amount of air and water vapor the pump can remove per minute), as it controls the speed at which the system can be evacuated.

6.2/ Freeze-Drying Cycle

To freeze-dry a product, it must be placed under freeze-drying conditions. The steps of this cycle are:

a) Preparation and pretreatment
b) Rapid freezing
c) Application of vacuum
d) Sublimation or primary drying
e) Desorption or secondary drying
f) Return to atmospheric pressure

Process details:

a) Preparation and pretreatment

Since freeze-drying is very costly, it is important to **reduce drying time**. For liquid products, **pre-concentration** is possible. For solid products (fruits, vegetables, meats, fish), it is important to **reduce size** (cutting, grating, grinding, etc.) to increase the surface area for heat and mass transfer. Ideally, fragments should have **large surface area and small thickness**. This also helps to shorten the overall drying time.

Freeze-drying depends on the **thickness of the product**, which is why it is important to work with **thin layers**. Some vegetables are **pre-blanched with steam** to inactivate enzymes.

b) Rapid freezing

Before freeze-drying, the product must be frozen quickly to avoid the formation of **large ice crystals**. The typical freezing rate is between **0.5 – 3 cm/h**. This step also helps slow down product deterioration before freeze-drying. Five techniques are currently used industrially:

- **Vacuum freezing**
- **Air-blast freezing** (cold air circulation)
- **Contact freezing** (using nitrogen, cold plates, or plates heated and cooled successively)
- **Mixed freezing** (air-blast plus contact)
- **Immersion freezing**
- **Internal freezing** (freezing inside the freeze-drying chamber)

c) Application of vacuum

Vacuum is applied by lowering the total pressure in the chamber, removing the atmosphere from 760 mmHg to 1–0.3 mmHg (depending on the product and equipment). This is achieved using vacuum pumps or high-capacity vacuum pumping units. Applying vacuum also completes the freezing process.

d) Sublimation or primary drying

Sublimation is a **phase change** in which a substance passes directly from solid to gas without going through the liquid state. It is an **endothermic process**, so heat must be supplied to the frozen product. Heat is provided by **heating plates** (circulated with a heat-transfer fluid) located in the freeze-drying chamber.

The **water vapor** released during sublimation is captured by the **cold trap or condenser**, maintained at a temperature lower than the frozen product, where the vapor deposits as ice (“snow”). Since sublimation is continuous, the frozen product temperature is kept constant by the **heated shelves**.

The driving force for **sublimation** is essentially the **pressure difference** between the water vapor pressure at the ice front and the partial pressure of water vapor in the drying chamber. The initial drying rate is high due to **low resistance to heat and mass transfer** at the beginning. As drying continues, a **dry layer forms around the frozen material**, acting as an insulating barrier, which **reduces heat transfer to the ice front** and **mass transfer from the ice front**.

e) Desorption or secondary drying
This step begins when there is no more ice in the product, and the **bound water** in the dried material must be removed. This is called **secondary drying or desorption**, which involves **evaporation under vacuum at a positive temperature** (20–60 °C), taking into account the product's denaturation temperature. Drying continues until the product's moisture content reaches **2–10%**, depending on the product, packaging, storage conditions, and intended use of the freeze-dried product.

f) Vacuum breaking
To return the freeze-drying chamber to **atmospheric pressure**, a controlled leak is opened to gradually fill the vacuum. Since freeze-dried products have a **high absorption capacity**, it is preferable to break the vacuum under a **neutral gas** (nitrogen or CO_2). It is important to **defrost** the cold trap to remove all condensed water. The final defrosting can be performed simultaneously with the return to atmospheric pressure.

g) Packaging
Dry food products must be **properly packaged** to prevent moisture absorption and lipid oxidation.

6.3/ Other Freeze-Drying Techniques

a) Freeze-drying with adsorbents
Water vapor can be trapped by **adsorption or absorption** on various materials (P_2O_5, $CaCl_2$, zeolites, molecular sieves, starch, etc.). Since the adsorption reaction is **exothermic**, the adsorbent serves a **dual purpose**: trapping water (reducing the partial pressure of water) and providing heat. In all cases, the **adsorbent must be regenerated** for reuse.

(Zeolites) are **energy-intensive** and require heating to **high temperatures** (above 200 °C).

a.1) Under vacuum
The cold trap can be replaced by several **adsorbent columns** (zeolites); this is the principle of **zeodration**. It is also possible to **recover the heat released by adsorption** using a heat-transfer fluid to **heat the product during freeze-drying** (conduction heating), which tends to **reduce operating costs** compared to classical vacuum freeze-drying.

a.2) At atmospheric pressure
Freeze-drying can also be carried out **at atmospheric pressure** without additional energy input. In practice, **fluidization** is ensured by a flow of **cold, dry inert gas**. Direct immersion of frozen products into a **fluidized bed of adsorbents** maintained at low temperature is a solution for sublimation at atmospheric pressure (Annex 3). This principle allows **continuous processing**, eliminates the need for a vacuum pump and heating sources (reducing investment costs), but **mass transfer at low temperature is limited**. Another limitation of

freeze-drying is the **separation of the fluidized material from the freeze-dried product**. The use of an **appropriate food-grade adsorbent** is also an important criterion.

6.4/ Effects of Freeze-Drying on Food Quality

- Although freeze-drying is a **preservation method used in microbiology** for microbial strains, it also **destroys a significant portion of contaminating bacteria**, improving **hygienic quality**.
- Being a **cold treatment**, the changes caused by high-temperature drying are negligible in freeze-drying.
- The **ice structure** in the product during drying **minimizes shrinkage**, promoting **rapid and almost complete rehydration**.

Three key points of interest can be highlighted regarding the **freeze-dried product**:

- **Storage stability:** Freeze-dried products can be stored at **room temperature in airtight packaging for several years**.
- **Sensory quality:** Taste and aroma are well preserved.
- **Nutritional quality:** Nutrients and vitamins remain almost intact. Vitamins **A, B1, B2, and C** are exceptionally well preserved, at levels very close to the fresh product, even after many months of storage. **Carotenoids**, which are highly heat-sensitive, are not affected by freeze-drying. Total nutrient losses after freeze-drying **do not exceed 10%**, whereas with conventional dehydration they can reach 50%. Freeze-drying preserves almost all qualities of the fresh product (organoleptic and nutritional characteristics), while ordinary drying techniques—except for freezing—**degrade proteins, caramelize sugars, and decompose volatile compounds responsible for flavor**.

The **nutritional value** of a freeze-dried product is comparable to that of a fresh product. Preservation of flavor is also a major **marketing advantage** for freeze-dried products.

Two very important points should be emphasized:

1. **Freeze-drying is not a sterilization process**; it is essential to use fresh products with **good microbiological quality**.
2. Freeze-dried products, being **hygroscopic and porous**, must be packaged in **suitable containers** that protect them from light, heat, and moisture.

Finally, the **shelf life** of freeze-dried products depends largely on the **packaging method**, but most properly packaged freeze-dried foods—such as fruit juices, vegetables, milk, and coffee—have an **almost indefinite shelf life**.