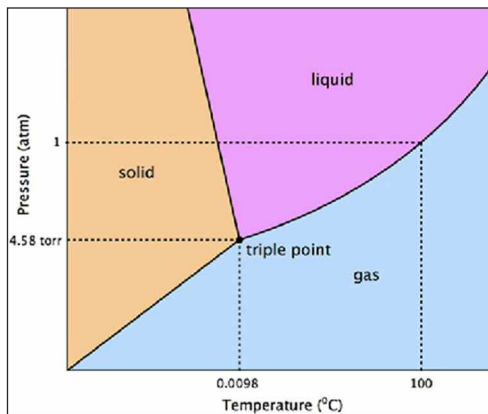


Freeze Drying

Freeze drying in general

The freeze drying process can be traced back to prehistoric times. Back in the 15th century, freeze drying was used by the Incas. They stored their crops on the mountain heights above Machu Picchu where the cold mountain temperatures froze the food and the water inside slowly vaporized under the low air pressure of the high altitudes.



The sublimation line is the line separating the solid phase from the gas phase.

Facts & Figures

- Maxwell House first introduced freeze dried instant coffee to the market in 1963.
- Secondary drying lasts 1/3 or 1/2 of the time that primary drying lasts.
- Some freeze dried products can be stored for up to 25 years without any issues at all!



How freeze drying works.

Freeze drying involves the removal of water or other solvent from a frozen product through a process called sublimation. Sublimation occurs when a frozen liquid transforms directly to the gaseous state without passing through the liquid phase.

The freeze drying process consists of three stages: prefreezing, primary drying and secondary drying.

Prefreezing:

Material intended to be freeze dried must first be prefrozen below its eutectic temperature. (read below to learn about eutectic temperature)

continued

Eutectic temperature

What is eutectic temperature?

The majority of products that are subjected to freeze drying consist primarily of water (the solvent) and the materials dissolved or suspended in the water (the solute). Most materials that are to be freeze dried are eutectics which are a mixture of substances that freeze at lower temperatures than the surrounding water. Although a product may appear to be frozen because of all the ice present, in actuality it is not completely frozen until all of the solute in the suspension is frozen. Only when all of the eutectic mixture is frozen is the suspension properly frozen. This is called the eutectic temperature.

How freeze drying works.

(continued)

Primary drying:

After prefreezing the product, conditions must be established in which ice can be removed from the product via sublimation. This requires very careful control of two parameters: temperature and pressure. The rate of sublimation of ice from a frozen product depends upon the difference in vapor pressure of the product compared to the vapor pressure of the ice collector. Molecules migrate from the higher pressure sample to the lower pressure sample. Since vapor pressure is related to temperature, it is also necessary that the product temperature is warmer than the ice trap temperature.

Secondary drying:

After primary drying is complete, all ice has sublimed but bound water is still present in the

product. Continued drying is necessary at warmer temperatures to remove the residual water. This process is called isothermal desorption as the bound water is desorbed from the sample. During

secondary drying, the product temperature is higher than ambient. All of the other conditions, such as pressure and ice trap temperature remain the same.

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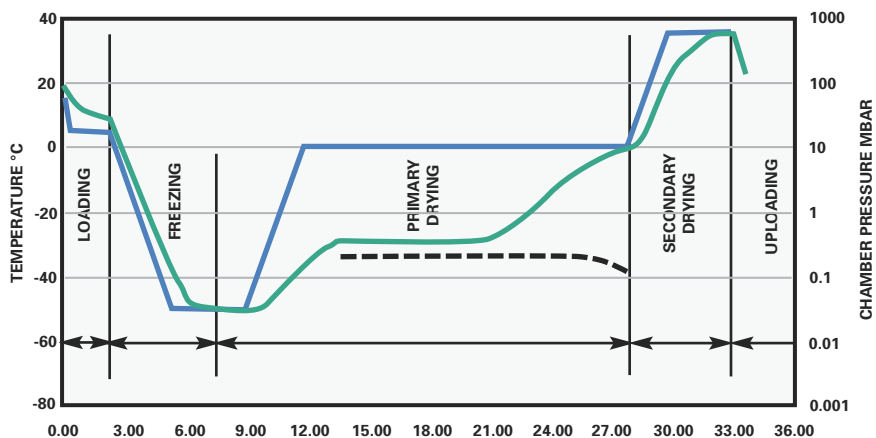


*Clockwise from above:
fresh raspberries;
frozen raspberries;
freeze dried raspberries.*

Freeze drying cycle

During the lyophilisation (freeze drying) process, the precise freezing process is very important. Rapid cooling results in small ice crystals which are useful in preserving structures to be examined microscopically, but resulting in a product that is more difficult to freeze dry. Slower cooling results in larger ice crystals and less restrictive channels in the matrix during the drying process.

- Environment temperature
- Product temperature



Why the need to measure the relative humidity?

Advantages and disadvantages

Freeze drying is commonly used in the production of pharmaceuticals to preserve ingredients. Many pharmaceuticals will degrade pretty quickly when exposed to water and air for the same basic reason that food degrades. Chemists can greatly extend pharmaceutical shelf life by freeze-drying the material and storing it in a container free of oxygen and water. Similarly, research scientists may use freeze drying to preserve biological samples for long periods of time. Freeze dried biological samples are also quite common in the florist world. The freeze drying process has also been used to restore water-damaged materials such as rare and valuable manuscripts.

Freeze dried material can be stored at warmer temperatures without degradation of the materials.

Freeze dried products can be stored without cryogenic or freezing facilities. The energy cost and the risk of product loss from power failure is greatly reduced.

As the product is dried out without excessive heating, proteins and other products that would be thermally denatured can be successfully preserved without loss of activity.

As products are much lighter when they are dry, freeze drying offers the benefit of reducing transportation costs.

The only disadvantage is that freeze drying is a very expensive



Freeze drying used in the production of pharmaceuticals . . . Freeze dried biological samples are also quite common in the florist world.

process in terms of high investment and maintenance costs of the equipment needed for the freeze drying process. Additionally, the production capacity is limited due to the lengthy process time.

Because freeze drying is an expensive process, after the product is freeze dried, it is important that it is monitored during the rest of the life span.

Optimizing the process

Air inlets: During the freeze drying process the air inlets must be monitored. The dryer the air entering the room, the better the freeze drying process.

By their nature, freeze dried products are highly hygroscopic. Exposure to water vapor during

storage can destabilize the product. This means the freeze dried products can never leave a controlled environment while in storage.

Tight control of the storage area where freeze dried products are placed is compulsory.

Residual humidity in elastomeric stoppers can cause degradation of freeze dried products. Prior to packaging the product, stoppers are typically washed, steam sterilized and dried. The steam sterilization process drives humidity into the stopper. If the drying conditions for the stopper are not optimised, residual humidity can transfer into the freeze dried product.

Packaging used for freeze dried materials must be impermeable to relative humidity.