Freeze drying in food processing

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Freeze-Drying Of Foods

What is freeze drying/Lyophilization and why????

Freeze drying is a water removal process typically used to preserve perishable materials, to extend shelf life or make the material more convenient for transport. Reduced pressure for this is normally 27-133Pa. It works by freezing the material, then reducing the pressure and adding heat to allow the frozen water in the material to sublimate.

This technique is used for preserving a wide variety of heat-sensitive materials such as proteins, microbes, pharmaceuticals, tissues & plasma.

(Freeze-drying: The origins of freeze drying-America pink)
History

The method can be trace back to prehistoric times and was used by the Aztecs and Eskimo for preserving foodstuffs. Toward the end of the 1880s the process was used in laboratory scale and the basic principles understood at that time. In 1890: Freeze drying was first carried out by Altmann, who freeze-dried organ pieces. In 1932: Gersh designed an effective vacuum plant for freeze drying of histological preparations. During World War II, freeze drying was for the preservation of biological samples such plasma. In the starting 1950: freeze drying was applied to foods and gained momentum in the food industry. In 1964: Nestle developed an improved method of producing instant coffee by freeze drying.

Today, several other coffee producers use similar process of preservation.

Triple point

In thermodynamics, the triple point of a substance is the temperature and pressure at which the three phases (gas, liquid, and solid) of that substance can exist in thermodynamic equilibrium.

(from; Basic Principles of Freeze Drying-John)
Sublimation????

Sublimation is when a solid (ice) changes directly to a vapor without first going through a liquid (water) phase. As shown on the phase diagram for water, low pressures are required for sublimation to take place.

Sublimation is a phase change and heat energy must be added to the frozen product for it to occur.

Components of Freeze Dryer

Main components;

- Refrigeration System
- Vacuum System
- Control System
- Product Chamber or Manifold
- Condenser

- **The refrigeration system**- It cools the (ice) condenser located inside the freeze dryer. The refrigeration system can also be employed to cool shelves in the product chamber for the freezing of the product.
- **The vacuum system**- It consists of a separate vacuum pump connected to an airtight condenser and attached product chamber.
- **Control systems** vary in complexity and usually include temperature and pressure sensing ability.
• **Product chambers** are typically either a manifold with attached flasks, or, a larger chamber with a system of shelves on which to place the product.

• **Condenser**—the purpose of the condenser is to attract the vapors being sublimed off of the product. Because the condenser is maintained at a lower energy level relative to the product ice, the vapors condense and turn back into solid form (ice) in the condenser. The sublimated ice accumulates in the condenser and is manually removed at the end of the freeze drying cycle (defrost step). The condenser temperature required is dictated by the freezing point and collapse temperature of the product. The refrigeration system must be able to maintain the temperature of the condenser substantially below the temperature of the product.

**Freeze Drying Mechanism**

Batch process can be summarized as follows:

1. **Pretreatment**
2. Loading /container (bulk, flask, vials)
3. Freezing at atmospheric pressure
4. Primary drying (sublimation)
5. Secondary drying (desorption)
6. Backfill & stoppering under partial vacuum
7. Removal of freeze dried product from the dryer
There are four main stages in the complete drying process:

- Pretreatment
- freezing
- primary drying
- secondary drying

**Pretreatment**

Pretreatment includes any method of treating the product prior to freezing. This may include concentrating the product, formulation revision (for example; addition of components to increase stability, preserve appearance, and/or improve processing), decreasing a high-vapor-pressure solvent, or increasing the surface area.

**Freezing**

Here the product temperature is lowered below the freezing point and, thus, most of the solvent freezes, forming ice crystals. It is extremely important that the sample be fully and completely frozen prior to pulling a vacuum and starting the drying process. Unfrozen product may expand outside of the container when placed under a vacuum.

**Primary drying**

The drying portion of freeze drying is actually a two part process consisting of Primary Drying and Secondary Drying. Primary drying (sublimation) is a slow process conducted at lower pressure & cooler temperatures (-40°C to -10°C) safely below the product’s critical collapse temperature. The bulk of water is removed from the product during freeze drying. At the end of primary drying when all of the free ice crystals have been sublimed, the product will appear to be dried. Organic solvents are also removed during primary drying.
However, the moisture content can still be in the 5-10% range due to the presence of “sorbed” water molecules attached to the product.

**What is COLLAPSE / EUTECTIC Temperature??**

Determination of the critical collapse temperature of a product is an important step in establishing and optimizing a freeze drying process. This critical temperature determines the maximum temperature that the product can withstand during primary drying without it melting or collapsing.

Thermal analysis (Differential Scanning Calorimetry & Freeze Dry Microscopy) and Dielectric Resistance analysis and are common methods used to determine this critical temperature of the product.

It is very important to keep the Temperature below the collapse Temperature. Otherwise collapse will happen. The occurrence of collapse can increase the residual water content in the final product and the reconstitution time, beside decreasing the activity of the pharmaceutical principle, moreover, a collapsed product is often rejected because of the unattractive physical appearance

**Determination end of primary drying**

Determination of end point of primary drying is very important. Because if the temperature increased too early to the value required by the last phase of the cycle, product Temperature may exceed the maximum allowed value and melting or collapse. Several analytical methods are available for determining that primary drying is complete.

~ **The most basic method is to monitor the product temperature with a thermocouple probe.**

| product temperature = shelf temperature |⇒| primary drying is complete |

~ comparison of parallel pressure readings between a Pirani gauge and a capacitance manometer.

A capacitance manometer always gives a true pressure reading in the product chamber. The Pirani gauge, however, will give a false high reading in the presence of water vapor. When the Pirani pressure reading
decreases and approaches the true pressure reading of the capacitance manometer, little or no water vapor is present and it can be concluded that primary drying is complete.

By measuring the pressure of the product chamber after closing the isolation valve at the vapor port.

This valve can be closed for a short period of time and the subsequent rise in pressure in the product chamber can be measured. When this pressure rise approaches zero, no more water vapor is being generated via sublimation.

**Secondary drying**

When the sublimation of the ice has been completed, shelf temperature is raised (e.g. to 20-40°C) and chamber pressure is further decreased. It allows desorption of the water bounded to the product, thus getting the target moisture in the product. And the secondary drying rates are dependent on the product temperature.

**Backfilling**

For many freeze dried products, the most ideal system of closure is while under vacuum. This provides an environment in which moisture and oxygen, both detrimental to the freeze dried material, are prevented from coming in contact with the product. In some cases, vacuum in a container may be less than ideal.

In these cases, backfilling the product container with an inert gas such as argon or nitrogen is often beneficial. The inert gas must be ultrapure, containing no oxygen or moisture. Backfilling of the product container is generally useful in a batch tray dryer type system. The backfilling should also be carried out through a bacteriological filter.
Storage and Stability of Dried Product

Lyophilized products are extremely hydroscopic and they must be sealed in air tight containers following freeze drying to prevent rehydration from atmospheric exposure.

Main factors affect the stability;

• Moisture
• Oxygen

The detrimental effect of oxygen and moisture are temperature dependent. If higher the storage Temperature, higher the degradation of the product. Because of that, at lower Temperatures it extends the shelf life.

Freeze Dryers

Choosing a freeze dryer depends on….

• the product characteristics
• the total volume of ice to be condensed
• as well as many other application-based variables including the container that the product will be dried in, the shelf area or number of ports required to accommodate the quantity to be dried in each batch
• Whether organic solvents are present- Because, Lower temperatures are required to freeze and condense organic solvents and they can easily bypass the condenser and end up causing damage to the vacuum pump.
• the type and shape of product being dried
• Cost and its end-use
Freeze-dryers can be grouped by size & use:

(1) laboratory bench-top units for R&D

(2) pilot units for process development and scale-up

(3) larger production-sized units/Industrial freeze dryers

- Industrial freeze dryers;
  - Tray and pharmaceutical freeze dryers
  - Multibatch freeze dryers
  - Continuous freeze dryers
  - Tunnel freeze dryers
  - Vacuum-spray freeze dryers

( from; Basic Principles of Freeze Drying-John Barley, SP Scientific)
Freeze dryers can be informally classified by the type of product chamber:

1. Manifold dryers where the product is typically pre-frozen & in fl/asks
2. Shelf dryers where the product is placed in a tray or directly on a shelf
3. Combination units with both drying options

Freeze Dried Vs. Dehydrated Foods

These are two food preservation processes used for removing the moisture from foods, such as fruits, vegetables, meat, and almost any other ingredient, food item, or meal. Freeze-dried foods may be stored 2 years or longer at room temperatures.
APPLICATIONS IN FOOD INDUSTRY

• The largest application is freeze dried food.
  ✓ to make instant coffee, pet foods
  ✓ To overcome seasonal variations….
  ✓ Freeze dried spices and vegetables are used in instant vegetable noodles, soups, snacks and in different kind of fat foods
  ✓ Fruit granules are used in products where food content is, texture and crunchiness is required
  ✓ To freeze dry Herbs, food-flavorings, fruits, and meats
  ✓ Freeze dried fruit slices and dices are often used;
  ✓ for making many delicious food products including ice creams, thick shakes, yoghurt, fruit drinks, and other desserts when fresh fruits are not available.
  ✓ Used in rich cream fillings, chocolate products and other baked goods
  ✓ Used to make soft candy, toffee and hard candy
  ✓ Used for flavoring baby foods and preparing food premixes

• In the pharmaceutical industry

• In the agriculture based industry Freeze dried microorganisms- for fermentation reactions, in bioconversion reactions and stored for research purposes

Freeze dried:

- Banana
- Cabbage
- Carrot
- Apple
- Garlic
- Papaya
Applicability in Sri Lanka

This is a highly cost involve method. As a developing country we can’t preserve variety of foods as other countries do. But we can apply it for some foods like processed fish, meats, and rare herbs and for pharmaceutical industry.

If enough capital /investors are there, even we can go for powdered products making process. Because there are lot of imported freeze dried foods in the market. So if we can produce our own it will be better.

Advantages and Disadvantages

Advantages;

✔ Easier to carry or transport
✔ all the nutrients and flavors are retained
✔ does not shrink or minimum structural change
✔ it takes only 10 minutes to rehydrate
✔ It has a very long shelf life
✔ Freeze-dried foods can stay edible for up to 25 years if unopened

Disadvantages;

✘ extremely costly, and may not be cost-effective for certain foods and it is time consuming
✘ Not all foods can be freeze dried
✘ Additives may be used when freeze-drying food
✘ Canning or dehydration is cost-efficient compared to freeze-drying
✘ Along with the water, other liquids like vinegar can sublimate
Airtight and moisture-control storage is absolutely necessary to ensure that the food does not spoil.

Freeze-dried food may not be easily available everywhere. The other disadvantages are minor, only the cost factor can be a problem for most people.

Quality changes in Freeze dried meat:
- Changes in protein structure and water binding
- Non-enzymatic browning reaction
- Degradation of myoglobin pigment
- Degraded home pigments may catalyse lipid oxidation

The future of freeze drying technology in the food industry:

- Newly developed active freeze drying technology…..

  A quicker and less labor intensive freeze drying process and the possibility of producing loose and free-flowing powder at low temperatures and low pressures, all in one vessel. Finally the freeze drying process is simplified because all process steps can be done in a single processing unit instead of handling trays filled with product between freezing units, drying chambers and crushers. This results in easy handling of the product especially when compared to the traditional tray dryer equipment.

  Active Freeze Dryer batch volumes can range from a few liters to hundreds of liters.

  “Recent developments are aiming to convert this unique technology into a continuous production process, allowing a further increase of capacity”
• **Spray-freeze-drying (SFD)**

It is an unconventional freeze drying technique that produces uniquely powdered products whilst still including the benefits of conventionally freeze dried products. SFD has potential applications in high value products due to its edge over other drying techniques in terms of product structure, quality, and the retention of volatiles and bioactive compounds. Here a solution being atomized, solidified and sublimed at low temperature and pressure.

Recent developments in this technique are reviewed including ultrasonic spray-freeze-drying the application of computational fluid dynamics and mathematical modeling, and the incorporation of new technologies to improve product quality. In addition, the advantages, limitations and future scope for research in the field of SFD are discussed.

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**Future in other sections…….**

- In bacteriology freeze-drying is used to conserve special strains
- Advanced ceramics processes sometimes use freeze-drying to create a formable powder from a sprayed slurry mist
- Freeze-drying is also used for floral preservation
- A new form of burial which previously freeze-dries the body with liquid nitrogen has been developed by the Swedish company

**Conclusion**

Freeze drying is a science which deals with the methods of prevention of decay or spoilage of food, thus allowing it to be stored in a fit condition for future use.
REFERENCES


