Freeze Drying Foods

Curtis Braun, SDSU Extension Food Safety Field Specialist

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Introduction

There is an increase interest in freeze drying foods with food entrepreneurs and processors in South Dakota. Freeze-drying is a processing method to remove moisture from product to make it shelf-stable. This publication will describe the freeze-drying process to help food entrepreneurs understand the process, as well as watch outs with this process.

Credit given to Mary Grace Danao, Research Associate Professor at the University of Nebraska-Lincoln who gave this presentation at the AFDO - Advanced Inspector Boot Camp (https://www.afdo.org/event/advanced-inspector-boot-camp-01-23-2025/).

Freeze Drying Fundamentals

Freeze drying is also called lyophilization which is a stabilizing process in which a substance is first frozen, the solvent is reduced by sublimation and then desorption, to values that will no longer support biological activity or chemical reactions. This process allows the freeze-dried product to be stored without refrigeration, and thus extends the shelf life and stability of the product. This process can also preserve biological activity or heat-sensitive compounds.

Another way to state what freeze-drying is in a non-technical term would be as follows:

“Freeze-drying is a **shelf-stable** process in which the water in the **food product** is first frozen to produce **ice crystals** and then the **water** or **moisture** is reduced first by turning **solid ice to water vapor (gas)** followed by the r**elease of moisture from a surface** to values that will no longer support biological activity or chemical reactions.” We will discuss the steps in this process to understand how water is removed from the product.

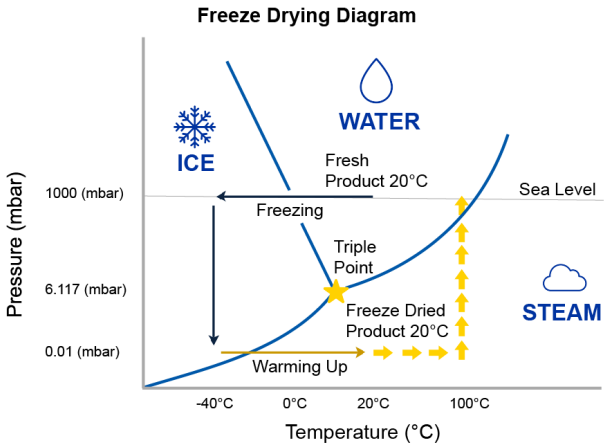

Figure 1. Freeze Drying Diagram

Freezing

First the moisture in the food is frozen to form ice crystals. However, the freezing step can sometimes be optional. For example, food can be put into a freezer first, rather than going directly into the freeze dryer. Some systems are made to accommodate this type of process.

The sublimation rate can vary by the size of the ice crystals. Smaller ice crystals can lead to longer sublimation rates and less moisture loss, but result in less structural damage to the food. The faster the freezing process, the smaller the ice crystals will

become. Conversely, larger ice crystals can lead to the product being more crunchy or porous and can also potentially damage the texture, flavor, and nutrients through the moisture loss. The slower the freezing process, the larger the ice crystals will become. It is typically recommended that the product be converted to bite sized pieces to make the freeze-drying process efficient. Products that are larger in size will take longer to freeze dry.

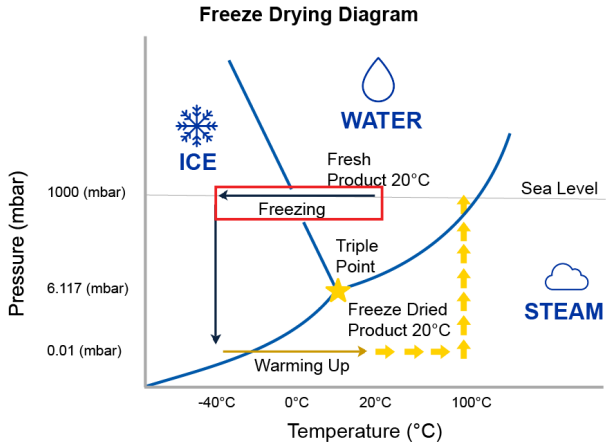

Figure 2. Freeze Drying Diagram highlighting freezing.

Vacuum Freezing

Next, the pressure is dropped by pulling a vacuum. The vacuum pump will remove air and gases in the freeze drying chamber and will maintain the vacuum in the chamber.

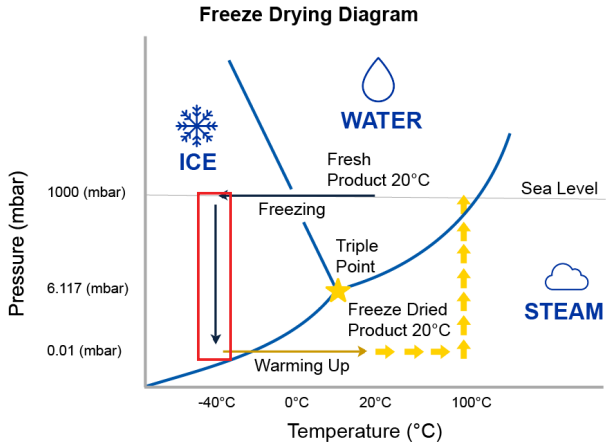

Figure 3. Freeze Drying Diagram highlighting vacuum freezing.

Sublimation

Next, the temperature is increased. This is accomplished by warming up the trays where the food is placed by conduction, the food is heated up, and the water is sublimated (solid to gas state). Without the heating step, there is no sublimation of the ice crystals into gaseous/vapor phase.

Knowing the triple point of the product is important. The triple point of the product is the combined temperature and pressure where all three phases of water are present (solid, liquid, gas). At a given temperature and pressure, water will quickly go from one phase to the next which means it will go from being ice, to water, and then to gas or vapor. The triple point is important to know because if the product never reaches the triple point, the water remains in or around the product. Once the product moves past the triple point, there is sublimation of the water into the gas or vapor phase. When we view the graph, we notice that when temperature increases, there is also an increase in pressure. The increase in pressure is the result of water going to gas.

Water begins to sublimate from the exterior of the product to the interior of the product as the heat transfers from the exterior to the interior of the product. For example, it may be possible that the exterior of the product may be dry, but the interior of the product may still have water that has not sublimated. For this reason, it is important to take measurements or cut open the product to see if it's dry at the center. It should be mentioned that ensuring that the product is thinly sliced and evenly sliced is important. For example, if the product is not evenly sliced, there is a potential that the thinner portion of the product will be properly freeze-dried, but the thicker portion may not be properly freeze-dried. Additionally, double-stacking product is not advised because the heat transfer from the tray to the product through conduction is what allows for the product to be properly heated, rather than conduction from product sitting on top of product.

One of the indications of when sublimation is complete is when there is no more increase in pressure. The reason we can know that the sublimation process is complete when there is no more pressure increase is because a majority of the water in the product has been sublimated into vapor. Thus, when there are no more pressure increases in the chamber, this means there is no more sublimation occurring.

After completion of the sublimation step, the moisture in the product is brought down to levels that no longer support biological activity or chemical reactions. If the process and equipment are well designed, the water activity can likely get below 0.2 which prevents biological growth. There is often confusion between moisture content of food and water activity of food. The moisture content of food is the total amount of water in a material whereas the water activity is the water vapor pressure in a product to the water vapor pressure of pure water and measures the water available for chemical reactions, enzymatic reactions, and microbial growth.

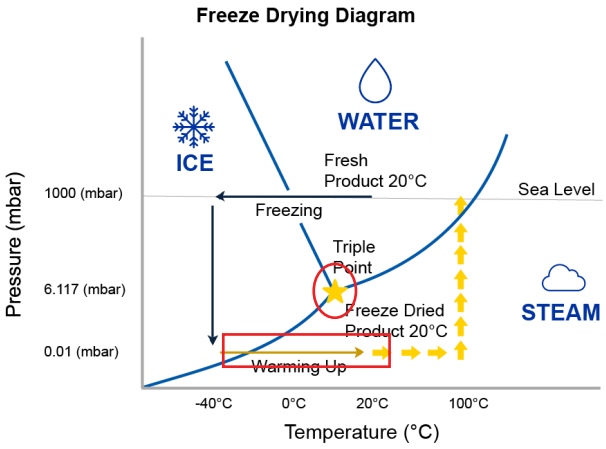

Figure 4. Freeze Drying Diagram highlighting sublimation.

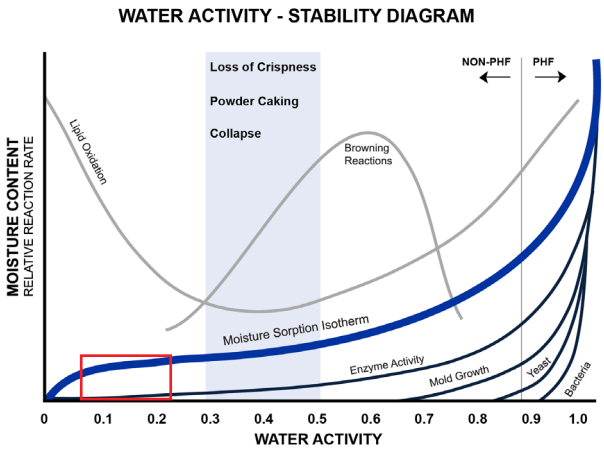

Figure 5. Water Activity - Stability Diagram

The table below highlights the role water activity plays in controlling microbiological growth. As we see below, microbiological growth does not occur with a water activity <0.60.

aw	Bacteria	Mold	Yeast	Typical Products
0.97	*Clostridium botulinum E*	-	-	fresh meat, fruits
	Pseudomonas fluorescens			vegetables, canned fruit, canned vegetables
0.95	*Escherichia coli*	-	-	low-salt bacon, cooked sausages,
	Clostridium perfringens			nasal spray, eye drops
	Salmonella spp.			-
	Vibrio cholerae			-
0.94	*Clostridium botulinum A, B*	*Stachybotrys atra*	-	-
	Vibrio parahaemolyticus			-
0.93	*Bacillus cereus*	*Rhizopus nigricans*	-	some cheeses, cured meat (ham), bakery goods, evaporated milk, ral liquid, suspensions, topical lotions
0.92	*Listeria monocytogenes*	-	-	-
0.91	*Bacillus subtilis*	-	-	-
0.9	*Staphylococcus aureus*	*Trichothecium roseum*	*Saccharomyces*	-
	(anaerobic)		*cerevisiae*	-
0.88		-	*Candida*	-
0.87	*Staphylococcus aureus*	-	-	-
	(aerobic)	-	-	-
0.85	-	*Aspergillus clavatus*	-	sweetened condensed milk, aged cheeses (cheddar), fermented sausage (salami), dried meats (jerky), bacon, most fruit juice concentrates, chocolate syrup, fruit cake, fondants, cough syrup, oral analgesic suspensions
0.84	-	*Byssochlamys nivea*	-	-
0.83	-	*Penicillium expansum*	*Deharymoces hansenii*	-
		Penicillium islandicum		-
		Penicillium viridicatum		-
0.82	-	*Aspergillus fumigatus*	-	-
		Aspergillus parasiticus		-
0.81	-	*Penicillium Penicillium cyclopium*	-	-
		Penicillium patulum		-

Source: Aqualab Site (https://aqualab.com/en/knowledge-base/expertise-library/how-water-activity-and-ph-work-together-control-microbial)

aw	Bacteria	Mold	Yeast	Typical Products
0.8	-	-	*Saccharomyces bailii*	-
0.79	-	*Penicillium martensii*	-	-
0.78	-	*Aspergillus flavus*	-	jam, marmalade, marzipan, glace fruits, molasses, dried figs, heavily salted fish
0.77	-	*Aspergillus niger*	-	-
		Aspergillus ochraceous		-
0.75	-	*Aspergillus restrictus*	-	-
		Aspergillus candidus		-
0.71	-	*Eurotium chevalieri*	-	-
0.7	-	*Eurotium amstelodami*	-	-
0.62	-	-	*Saccharomyces rouxii*	dried fruits, corn syrup, licorice, marshmallows, chewing gums, dried pet foods
0.61	-	*Monascus bisporus*	-	-
0.6	*No microbial proliferation*	-	-	-
0.5	*No microbial proliferation*	-	-	caramels, toffees, honey, noodles, topical ointments
0.4	*No microbial proliferation*	-	-	whole egg powder, cocoa, liquid center cough drop
0.3	*No microbial proliferation*	-	-	crackers, starch-based snack foods, cake mixes, vitamin tablets, suppositories
0.2	*No microbial proliferation*	-	-	boiled sweets, milk powder, infant formula

Source: Aqualab Site (https://aqualab.com/en/knowledge-base/expertise-library/how-water-activity-and-ph-work-together-control-microbial)

Packaging

Once the material has been dried, it's important to package quickly as the material is hygroscopic (readily absorbs moisture from the air). It becomes even more crucial to quickly package the material when working in a very humid environment. Additionally, because the product is more porous and has a higher surface area, it may be more prone to lipid oxidation and going rancid. The packaging material which should be used is material with high moisture, oxygen, and light barrier properties. This includes aluminum mylar bags. Food grade oxygen absorbers may also be used to absorb the oxygen that is trapped in the package. Oxygen absorbers would be more crucial for products that are high in fat.

Food Safety Risks

- Poor GMPs and Sanitation Practices
  - » **Freeze-drying does not kill pathogenic bacteria. If bacteria is present on the food when it's freeze-dried, it can remain there. If the freeze-dried product is re-hydrated, there is a risk that any pathogen that was on the product will now have an opportunity to grow, especially if the product is hydrated to where the water activity is >0.85.**
- Inconsistent Processing
  - » **Product that has inconsistent thickness, double-stacking product, variation in freezing/drying and other factors can contribute to varying results.**

SDSU Extension Services

SDSU has the capability to test for the water activity of products. If you would like to have your product tested for water activity, please reach out to the SDSU Food Safety Extension Specialist who can help coordinate the testing for your freeze-dried product.

South Dakota Food Regulations for Freeze-Dried Products

Products that are freeze dried would fall under the cottage food law since they are considered non-temperature controlled. However, depending on the product, some freeze-dried products would not be allowed to be sold under the cottage food laws without licensing or approval from the regulating agency. Check with SD Department of Health, SD Department of Agriculture and Natural Resources, or the SD Animal Industry Board if you have questions regarding the product you're interested in freeze drying. Below are some examples of freeze-dried products that cannot be sold under the cottage food law without licensing.

Product	Regulating Agency
Freeze-dried Meats	SD Animal Industry Board and/or SD Department of Health
Freeze-dried Eggs	SD Department of Agriculture and Natural Resources
Freeze-dried Fish	SD Department of Health
Freeze-dried Ice Cream	SD Department of Agriculture and Natural Resources and/or SD Department of Health

Conclusion

Freeze drying is a process that makes products shelf-stable, but does not kill pathogens that may be present in or on the food. The freeze-drying process makes food shelf stable by sublimating the water in or on the food into a gas which decreases the water activity. Understanding the freeze-drying process is important to understand the steps in which the product is dried, appropriately prepping the product prior to freeze-drying, understanding how to determine when freeze drying is complete, how to protect the product from picking up moisture after freeze drying, and how this process is regulated in South Dakota.

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