Freeze Drying and Potential Food Safety Challenges

23 Jan 2025

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University of Nebraska-Lincoln

THE FOOD PROCESSING CENTER

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The Food Processing Center is a multi-disciplinary resource for the food industry, providing a combination of consulting, educational, technical, and business development services.

- Analytical Food Chemistry Laboratory
- Applied Research and Engineering
  - High Pressure Processing & Engineering Services Laboratory
- Labeling and Regulatory Compliance
- Microbial Testing Laboratory
- Pilot Plants
- Product and Process Development
- Sensory Analysis Laboratory
- National Food Entrepreneur Program

The Food Innovation Center on the Nebraska Innovation Campus houses the Food Processing Center.

About Us

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Research Associate Professor

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M.S. Student

Consulting • Education/Training • Process Optimization & Validation
Microbial Challenge Studies • Shelf-Life Studies • Engineering Analysis

Table of Contents

Freeze Drying Fundamentals

Definition

- Also called “lyophilization”
- A stabilizing process in which the substance is first frozen and then the solvent is reduced – first by sublimation followed by desorption – to values that will no longer support biological activity or chemical reactions.

Objectives

- Preserve the biological activity or heat-sensitive compounds in a food product.
- Store the freeze-dried substance without refrigeration, thereby extending the shelf-life and stability of the product.

Freeze Drying Fundamentals

shelf-stable *food product*

A stabilizing process in which the substance is

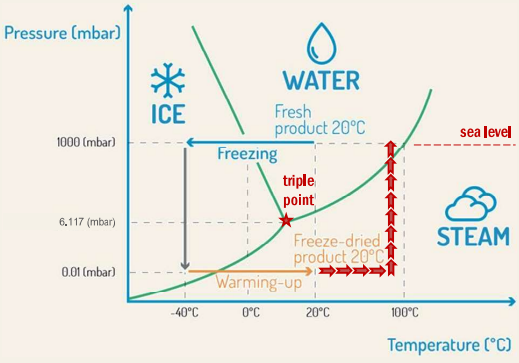
ice crystals *water or moisture*

first frozen and then the solvent is reduced –

solid ice to water vapor (gas) *release moisture from a surface*

first by sublimation followed by desorption to

values that will no longer support biological activity or chemical reactions.



https://www.barnalab.com/en/what-about-freeze-drying/
Note: This diagram is not to scale. The triple point (or eutectic point) of water is at 0.01 C and 6.117 mbar.

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low water activity

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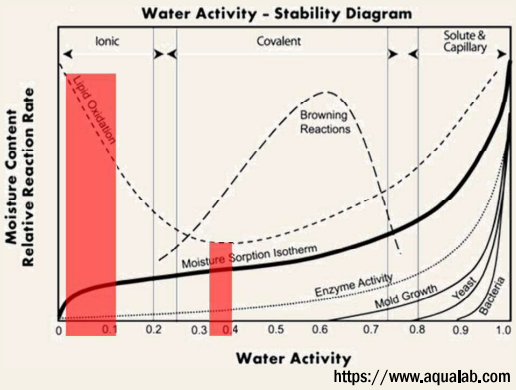


https://www.aqualab.com

FD Equipment

- For "home" use (6-10 lbs "wet")
- Popular with hobbyists and entrepreneurs

- Pilot scale (20-50 lbs "wet")
- R&D testing

- Industrial, batch (250-4.000 lbs "wet")
- Food manufacturing

FD Equipment

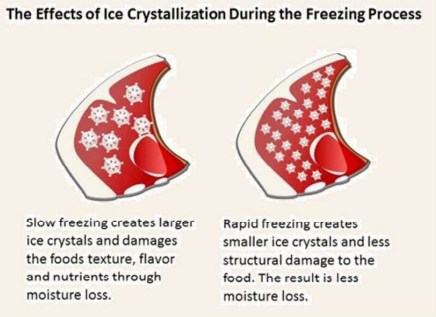
- Industrial, continuous systems
- Food manufacturing, co-manufacturing

FD Operations

Freezing → Vacuum freezing → Sublimation (Primary drying) → Desorption (Secondary drying) → Packaging

Freezing (optional)

- Can occur inside the freeze dryer or prior to loading product in the freeze dryer
- The smaller the ice crystals, the longer the sublimation rates.
- The larger the ice crystals, the more porous (or "airy") the freeze-dried product.

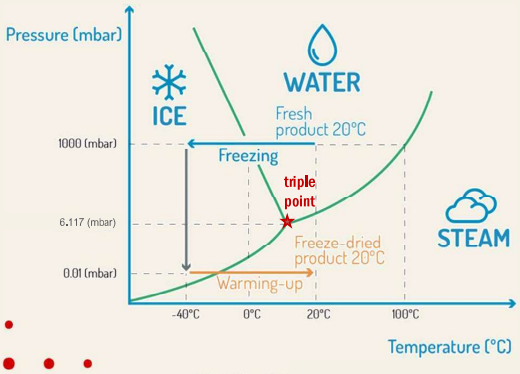


FD Operations

Freezing → Vacuum freezing → Sublimation (Primary drying) → Desorption (Secondary drying) → Packaging

Vacuum freezing and sublimation

- Vacuum freezing is typically a quick step.
- Ice crystals in the product sublimate, vaporizing without passing through the liquid phase of water.
- The fastest evaporation rates occur at 0.200 mbar.
  - Below this threshold, there are not enough molecules present in the system for **good heat transfer.** >> FD will take longer.
  - Above this threshold, there are too many molecules present, which slow down the sublimation rate.



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FD Operations

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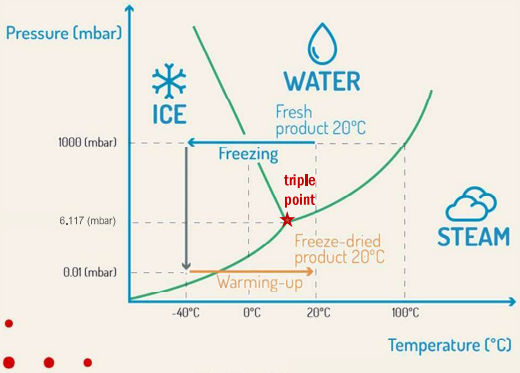
Sublimation

- Heat is applied to the shelves and, by conduction, heats the frozen product, to accelerate sublimation.
- Know the triple point of your product and monitor product temperature. You want the product to warm up past this triple point. Otherwise, the product may “melt back”.



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FD Operations

Freezing → Vacuum freezing → **Sublimation** (Primary drying) → Desorption (Secondary drying) → Packaging

Sublimation

- FD is a simultaneous heat and mass transfer process!
- Heat flux to the surface of the material occurs by convection and, in the dry solid, by conduction to the sublimation surface (i.e., an ice front moves through the sample).
- The sublimating water vapor must pass through the dried portion of the sample. The less sample it must pass through, the faster the drying process.

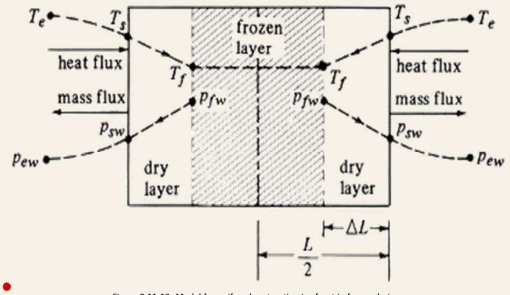


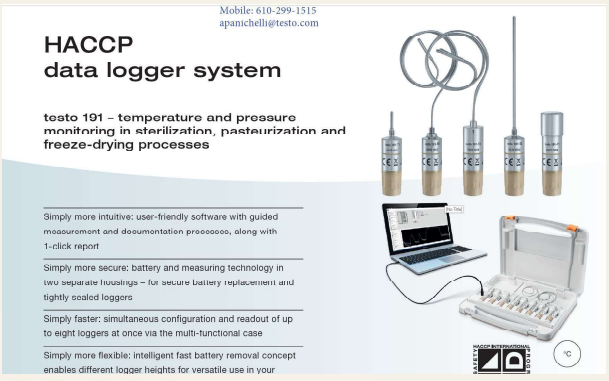
Figure 9.11-12. Model for uniformly retreating ice front in freeze drying.

FD Operations

Freezing → Vacuum freezing → **Sublimation** (Primary drying) → Desorption (Secondary drying) → Packaging

Sublimation

- During this stage, the product undergoes evaporative cooling...
- ...which means the product temperature will be lower than the shelf temperature.
- When the product and shelf are at the same temperature, sublimation is complete!
- A properly designed sublimation step can remove over 90% of the moisture from the sample.

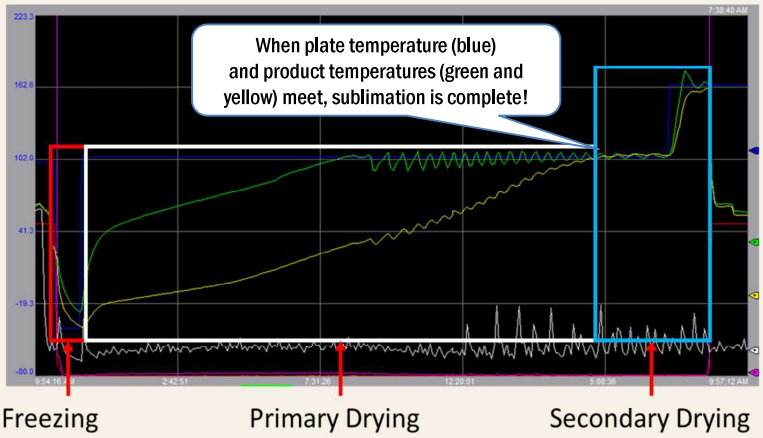


FD Operations

Freezing → Vacuum freezing → **Sublimation** (Primary drying) → Desorption (Secondary drying) → Packaging

PARKER FREEZE DRY
A DIVISION OF PROFORM FABRICATION

For more info, contact:
Matt Graunke
Sales & Marketing Manager
ProForm Fabrication LLC/
Parker Freeze Dry



FD Operations

Freezing → Vacuum freezing → Sublimation (Primary drying) → **Desorption** (Secondary drying) → Packaging

Desorption

- Shelf temperatures can be raised to 50 to 60 °C and vacuum lowered to 67 millibars.
- Drying rate will be slower than that of sublimation, and will increasingly slow down as most water molecules remaining are strongly bound to the product structure.
- An extra 1-2% reduction in moisture content may add hours of processing time, which drastically reduces production throughput.

FD Operations

Freezing → Vacuum freezing → Sublimation (Primary drying) → Desorption (Secondary drying) → Packaging

Packaging

- At the end of drying, vacuum inside the dryer may be replaced by filtered dry air or nitrogen to establish atmospheric pressure.
- The dried material is extremely hygroscopic and must be packaged quickly.
- Because the dried material is porous and has a higher surface area than before, it is more prone to oxidation (i.e., going rancid).
- Use packaging material with high moisture, oxygen, and light barrier properties.

Food Safety Risks/Challenges

Scenario #1. Hobbyist or food entrepreneur using a freeze dryer for "home" use.

Higher likelihood for...

- Lack of experience and good GMPs >> Products to be contaminated during preparation, equipment loading, or packaging.
- Lack of experience and monitoring tools (temperature, initial/final water activity, initial/final moisture content) >> Inadequate drying. Product appears dry on the outside, but is wet on the inside.
- Lack of equipment and/or process controls. >> Inadequate drying and batch-to-batch variations.

Food Safety Risks/Challenges

Scenario #1. Hobbyist or food entrepreneur using a freeze dryer for “home” use.

FOOD

Mung bean omelet, anyone? Sky high egg prices crack open market for alternatives

JANUARY 26, 2023 · 11:25 AM ET

HEARD ON MORNING EDITION

By Stacey Vanek Smith

5-Minute Listen + PLAYLIST

The proof is in the profits

Kern charges about $20 a dozen for his freeze dried eggs. He tells me this is a good deal: the eggs weigh almost nothing, keep for decades, don't lose any nutritional value and come in a little mylar envelope, which stores easily.

And, mostly, it gives customers peace of mind: whatever supply chain disasters, deadly flus, price spikes and shortages the economy might throw at us, they will still have their beloved breakfast dish.

The proof is in the profits. The monent Kern started selling his eggs online, orders poured in from all across the country.

"The demand went nuts," he recalls. "Every single package that we put on our online store was sold within 30 seconds. They just ... fly off the shelves," He adds: "I'm not even a pun person, but there you go."

Food Safety Risks/Challenges

Scenario #1. Hobbyist or food entrepreneur using a freeze dryer for “home” use.

EASY

If you can press a button, you can freeze dry! Simply press start on the touch screen, and the patented Smart Freeze© technology senses when it's done. Everything is automatic.

Download Our Free Guide >

Food Safety Risks/Challenges

Scenario #1. Hobbyist or food entrepreneur using a freeze dryer for "home" use.

Hello – We purchased a scientific model freeze dryer a few years ago. Do you still sell them? Also, does a "standard" (not customized recipe) run in a scientific model exactly the same as a "standard" run in a home freeze dryer model?

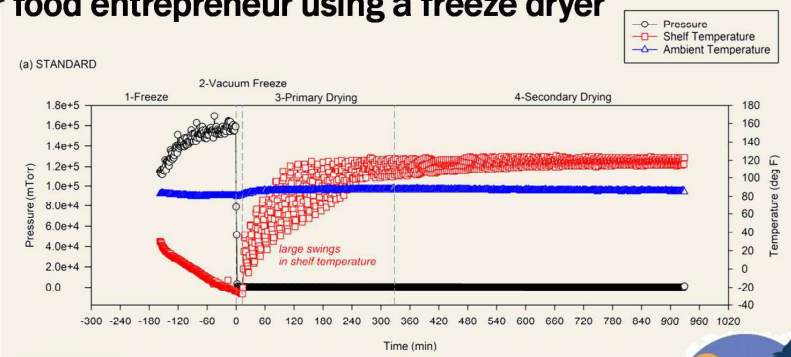
Yes, we do. Here is the link. The in-home freeze dryer has a standard profile that gets a product dry (you do have some temperature control), but you cannot create your own profiles.

Thank you! I wanted to see a typical profile from the in-home freeze dryers.

A 9-12 hour freeze. Heat at 125 F. mTorr to trigger the heat is 500 mTorr.

Food Safety Risks/Challenges

Scenario #1. Hobbyist or food entrepreneur using a freeze dryer for "home" use.



PRELIMINARY RESULTS…after freeze-drying…

- Average moisture content = 0.52%
- Average water activity = 0.04

Reductions (log CFU/g_{dm}):

- Generic *E. coli,* 1.36
- *E. faecium,* 0.08
- *L. innocua*, 1.67

IAFP 2024 Long Beach California July 14-17

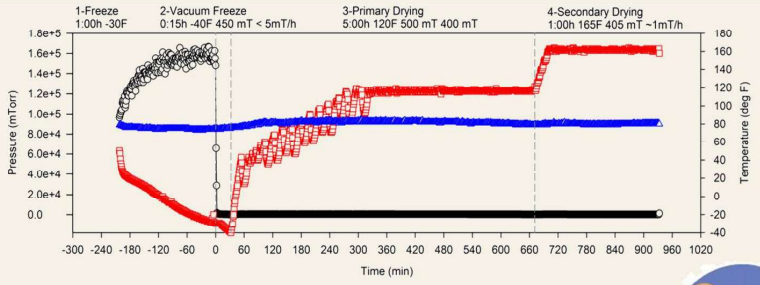
Food Safety Risks/Challenges

Scenario #1. Hobbyist or food entrepreneur using a freeze dryer for "home" use.

- No means of monitoring product temperature.
- Lack of consumer education on how FD process works and how to develop process controls or critical control points (CCP) – e.g., target final water activity or moisture content.
- Temperature-time combination in home FD equipment is not a suitable "kill step", which is okay. But, consumers are led to believe FD products are safe and can last for years!
- Lack of consumer education that a rehydrated potentially hazardous food (PHF) product is not treated as PHF.

Food Safety Risks/Challenges

Scenario #2. Food manufacturers do not necessarily understand the FD process either and believe it can be a "kill step".



PRELIMINARY RESULTS…after freeze-drying…
- Average moisture content = 0.44%
- Average water activity = 0.05

Reductions (log CFU/g_{dm}):
- Generic *E. coli,* 1.75
- *E. faecium,* 0.29
- *L. innocua*, 1.75

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Food Safety Risks/Challenges

Scenario #2. Can freeze drying be a "kill step"?

Cuddon FREEZE DRY

Lethality validation study of *L. monocytogenes* and *Salmonella* spp. in ground beef patties in freeze dried pet food

Descriptive purposes only. Not the actual burgers used in the study.

For more info, contact:
Lloyd Parks
VP Sales
Cuddon Freeze Dry

Hypothesis: Using USDA Appendix A as a guide, programming a FD to meet an appropriate internal temperature-time combination in beef patties can result in a 7-log reduction in Lm and *Salmonella*.

Processing Conditions:

- At a vacuum < 2.66 millibar.
- Internal product temperature held at or above 156.2 °F for 120 min.
- Temperature data were collected from the centers of inoculated patties.

Results:

- Per temperature-time logs, samples were held above 156.2 °F for an average of 163 min.
- Average log reductions:
  - Lm, 3.81 and *Salmonella*, 5.41

Food Safety Risks/Challenges

Scenario #3. What corrective actions can be used when FD process fails?

- Can I re-freeze the product, reach the triple point, and re-run the FD process again?
- How well does a failed FD process affect product quality?
- Can a failed FD product be re-worked?
- For in-home FD, how do we capture processing data from the equipment?
- Packaging failures:
  - If drying has not been completed, packaging traps residual moisture, enabling microbial activity during long-term storage without refrigeration or freezing.
  - It is possible facultative anaerobes remain in spore form in a FD product stored long-term without refrigeration or freezing.

Next Steps: NebGuide

Strawberries (sliced)

Date: 19 July 2024

Non-TCS food? ☒ Y ☐ N

Freeze dryer mode: ☒ Standard or Normal ☐ Fast ☐ Candy ☐ Programmed

Yield = Final (dry) weight / Initial (wet) weight x 100% = 9.5 / 142 = 6.69 %

Final (dry) moisture content = 10% Final (dry) water activity = 0.05

Note: Took 15 h. No extra drying time needed.

Green beans (whole)

Date: 20 July 2024

Non-TCS food? ☐ Y ☒ N

Freeze dryer mode: ☒ Standard or Normal ☐ Fast ☐ Candy ☐ Programmed

Yield = Final (dry) weight / Initial (wet) weight x 100% = 29 / 138.5 = 20.9 %

Final (dry) moisture content = 45% Final (dry) water activity = 0.67

Note: 18 h cycle. Still wet! Water was unable to escape the "skin" of the whole green beans. Not suitable for freeze drying.

apples, bananas, basil, blackberries, carrots, cantaloupe, cherries, cilantro, green beans, lemons, mint, oranges, parsley, raspberries, strawberries, tomatoes, watermelon

edible cookie dough (no egg), Gouda cheese slices, heavy whipping cream, skim milk, ice cream with strawberries, whipped cream cheese, whole milk

gummy bears and worms, marshmallows, Skittles

chicken noodle soup, chili mac

Thank you for your attention!

Freeze drying is a highly sophisticated food processing technology that can deliver a high quality, premium product.

But, we have much to learn on how to use it appropriately to deliver high quality, safe foods.

in CONNECT

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