



Indiana
Department
of
Health

FREEZE-DRYING FUNDAMENTALS & SAFETY GUIDANCE

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FOOD PROTECTION DIVISION

01/17/2023

OUR MISSION:

To promote, protect, and improve the health and safety of all Hoosiers.

OUR VISION:

Every Hoosier reaches optimal health regardless of where they live, learn, work, or play.



Webinar agenda

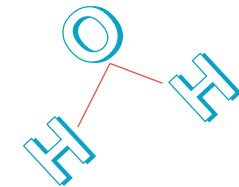
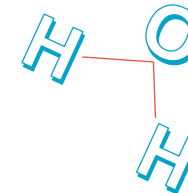
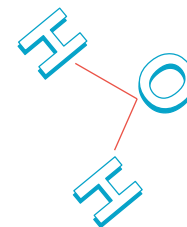
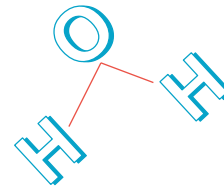
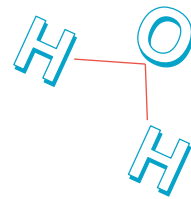
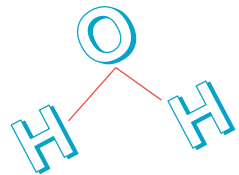
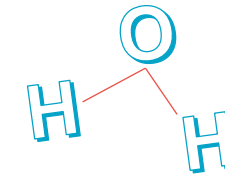
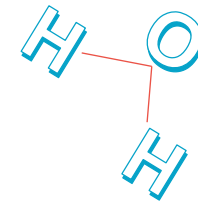
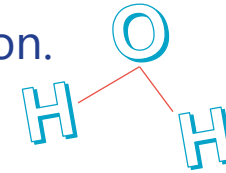
- Brief introduction
- Share guidance document
- Food freezing
- Food drying
- Freeze-drying
- Food safety risks
- Equipment considerations
- Packaging considerations (including ROP)
- IDOH freeze-drying guidance
- Brief Q & A session

About Erik - I'm a Boilermaker. I'm here to help.

- Food scientist trained by Purdue University and the Swedish University of Agricultural Sciences
- More than 20 years of experience with food manufacturing operations, food safety, and food science instruction
- IDOH special processing technology consultant
- Currently Director of Quality Control for Wick's Pies, Inc., home of Indiana's State Pie
- Served on professional staff of Purdue University for 17 years
- Designed Purdue's central production kitchen
- Managed food processing and warehousing operations for Purdue University food stores
- Multi-unit manager of retail restaurants for Purdue Dining & Catering
- Managed and updated Purdue University's Food Science Pilot Plant
 - Instructor for FD152, six years
- Designed the Purdue Food Science Pilot Brewery
- Designed and managed Skidmore Food Product Development Laboratory
- Served executive board for the Indiana Section of Institute of Food Technologists (IFT)
- Provided mentoring for Mandela Washington Fellowship (YALI)
- Owned and operated private consultancy Food Manufacturing Solutions, LLC
- Featured speaker for numerous regional and national food industry events

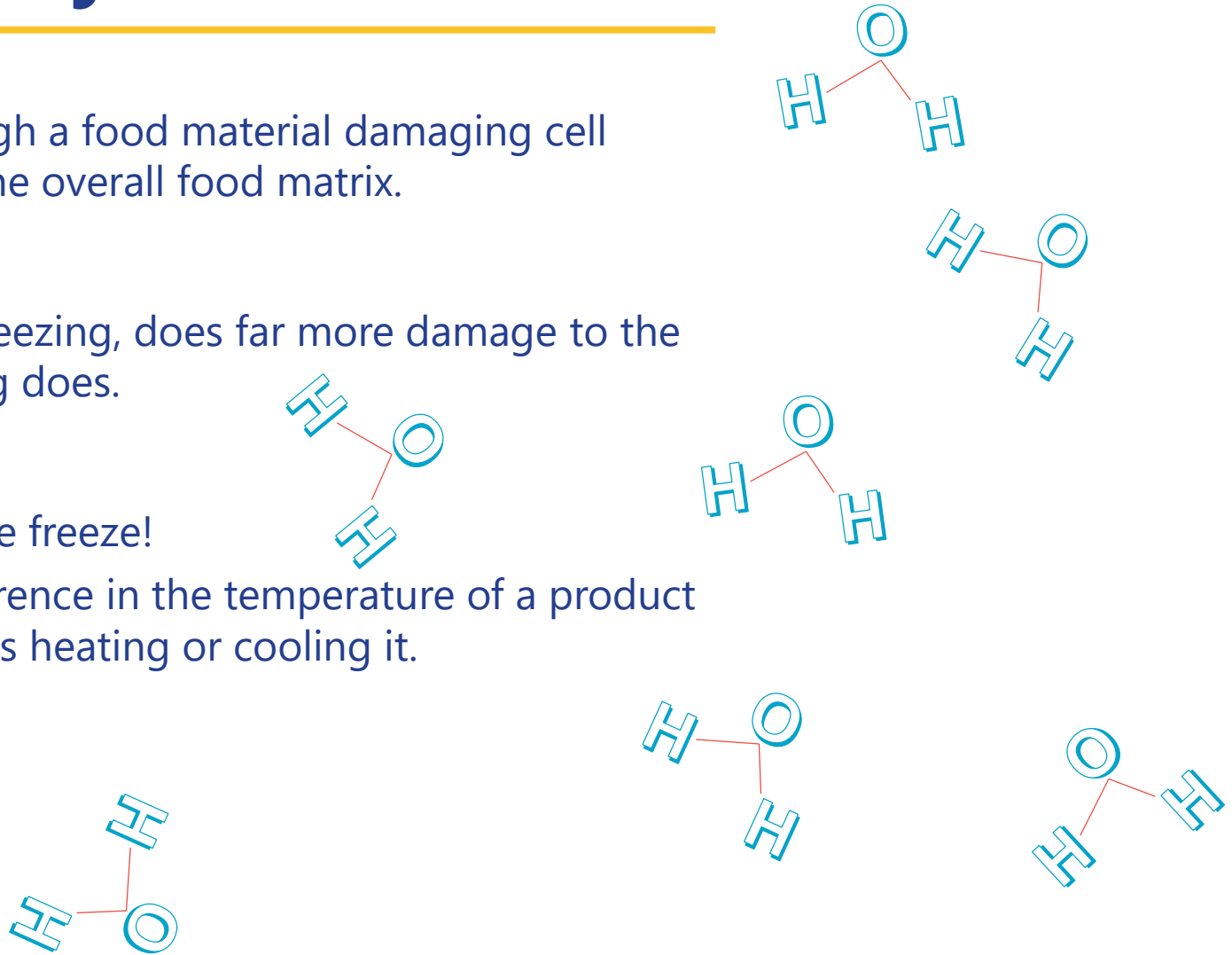
Food freezing- water migration

- Whenever food is in the process of freezing, water is put into motion.
- Water molecules are sharp little triangles - H-O-H.
- When water changes from liquid to solid QUICKLY, it results in SMALLER more uniform, smoother ice crystals.
- When water changes from liquid to solid SLOWLY, it stacks upon itself resulting in LARGER non-uniform, jagged ice crystals.



Food freezing - crystals and concentration

- As crystals form, they migrate through a food material damaging cell membranes, macromolecules, and the overall food matrix.
- Slow freezing, often called SHARP freezing, does far more damage to the product structure than rapid freezing does.
- The larger the delta T, the quicker the freeze!
 - **Delta T** (written as ΔT) is the difference in the temperature of a product and the temperature of whatever is heating or cooling it.



Food freezing - crystals and concentration

Tomato example:

Here's something fun to consider. It's a tomato that I froze at -1F for 14 hours. It's just incredible how much water ran out of it without even breaking the skin when I thawed it on that plate at room temp for 10 hours.

This is a product compromised by SHARP freezing.



Food freezing - crystals and concentration

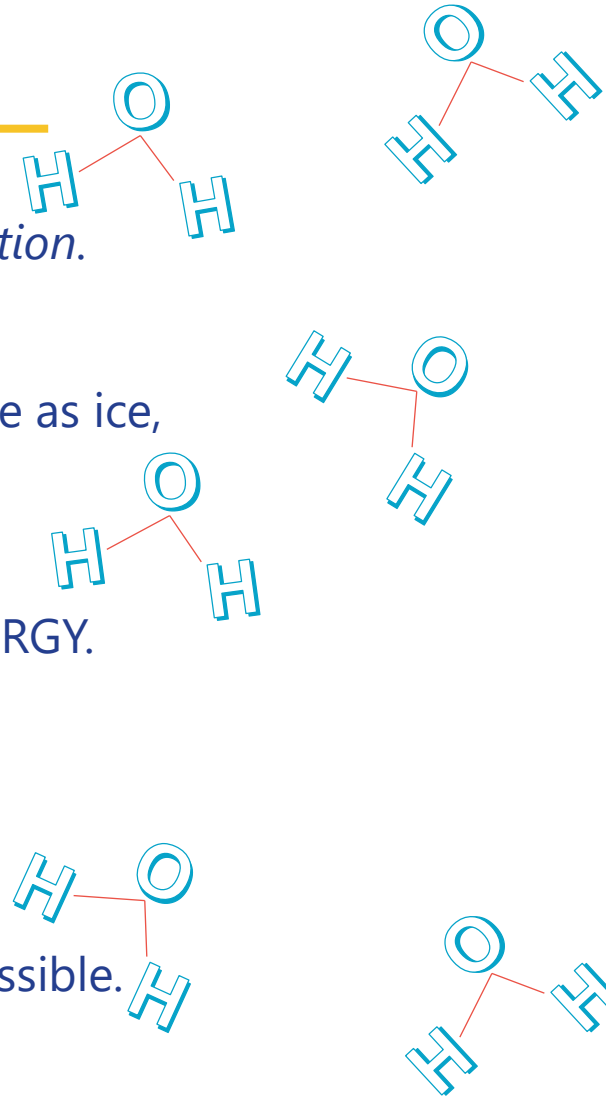
Side-by-side of fresh tomato vs freeze-thaw tomato: NOTE the wrinkled skin AND the



NOTE the cell structure and the quantity of free water in both the fresh and the freeze-thaw tomato.

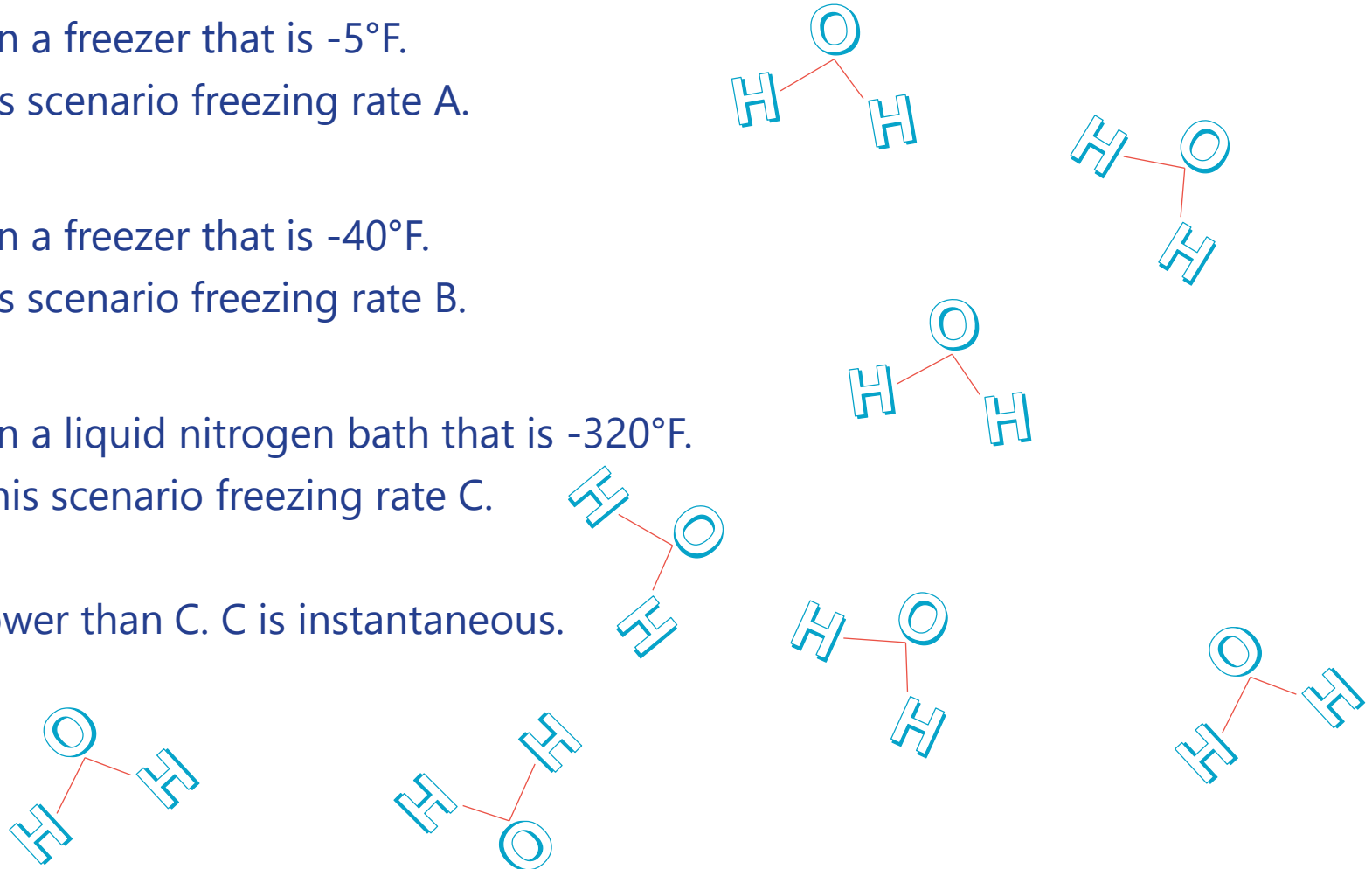
Food freezing – eutectic point

- All phase changes require some ENERGY called *latent heat of transformation*.
- When **all** of the water (100%) in a given system exists in a crystalline state as ice, that system has reached its **eutectic point**.
- In a eutectic state, ice crystals are incredibly effective **conductors** of ENERGY.
- Large delta T helps a sample effectively reach eutectic point.
- We cannot freeze dry without reaching eutectic point. It is simply not possible.



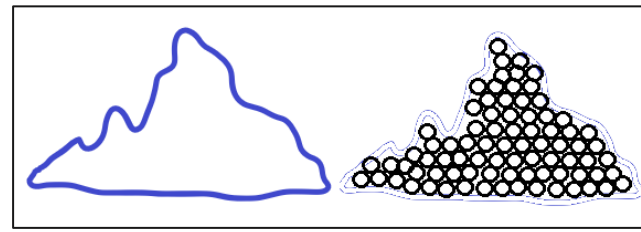
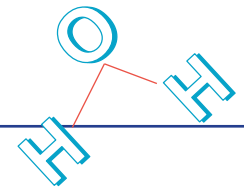
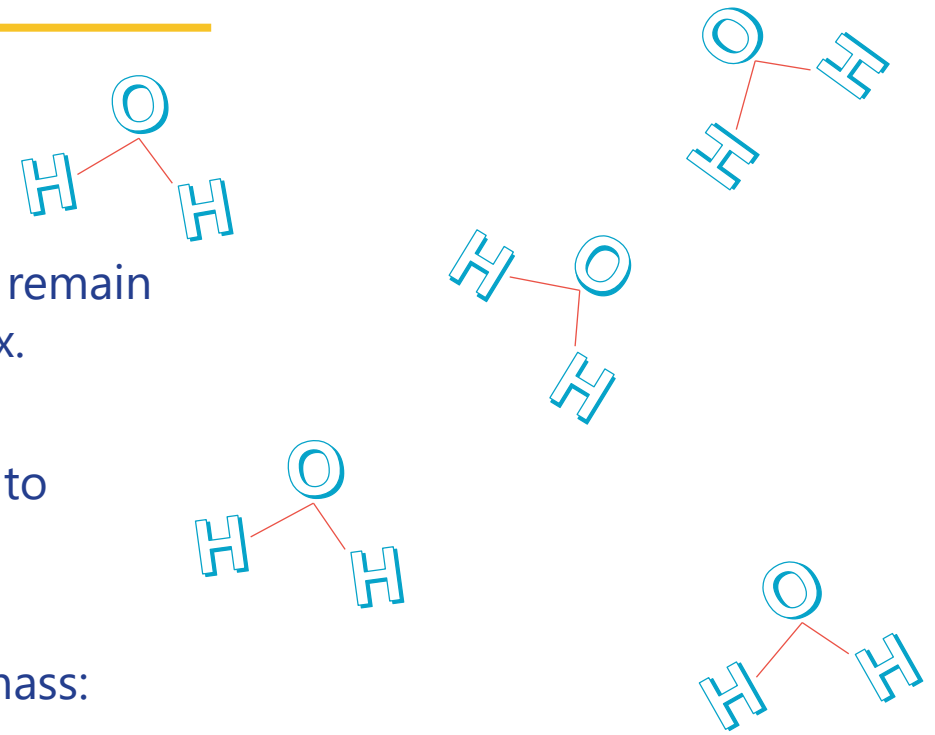
Food freezing – delta T examples

- 8 oz of 40°F juice is placed in a freezer that is -5°F.
 - $\Delta T=45^{\circ}\text{F}$ We will call this scenario freezing rate A.
- 8 oz of 40°F juice is placed in a freezer that is -40°F.
 - $\Delta T=80^{\circ}\text{F}$ We will call this scenario freezing rate B.
- 8 oz of 40°F juice is placed in a liquid nitrogen bath that is -320°F.
 - $\Delta T=360^{\circ}\text{F}$ We will call this scenario freezing rate C.
- A is slower than B. B is slower than C. C is instantaneous.



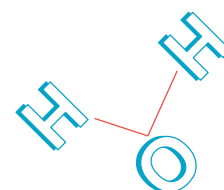
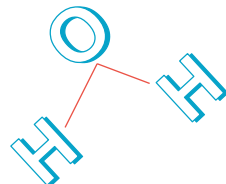
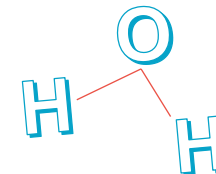
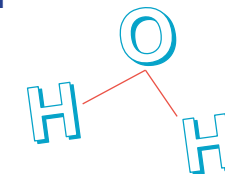
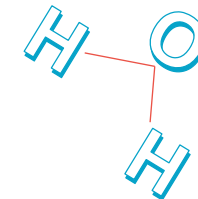
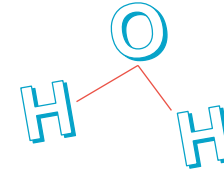
Food freezing – time and distance consideration

- Ice crystal movement through the food matrix requires **TIME**.
- If crystals form instantaneously, time is not a factor, so crystals remain very small and have no opportunity to damage the food matrix.
- SMALLER ice formations have a LARGER surface area available to interact with the ATMOSPHERE during **sublimation**.
- Fun example: Huge stack of snowballs vs an iceberg of equal mass:
 - Same amount of ice, very different surface area.
 - In snowballs there is far less physical distance for any given water molecule to move while migrating towards the surface.
 - Which would you prefer for FD?



Food freezing – freeze-drying settings

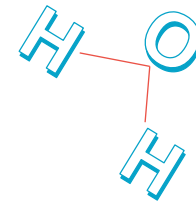
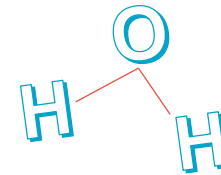
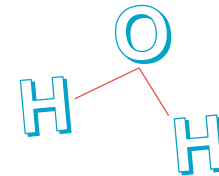
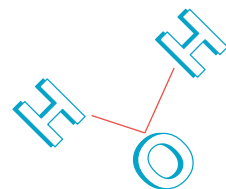
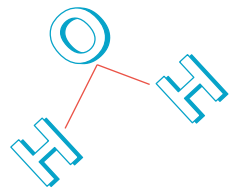
- In most HBV freeze dryers, freezer setpoints will be around -40°F .
- If product is placed in FD at 70°F , then the typical HBV $\Delta T=110^{\circ}\text{F}$.
- Scientific and/or industrial FD will have temperature setpoints far lower than the general HBV freeze dryers.
 - Not unusual to see -80°F or lower as setpoints.
- Some operations will pre-freeze products to be certain they achieve eutectic point.
- Eutectic point settings are often treated as confidential trade secrets.



Food freezing – pause for chat questions

- Brief pause, field chat questions and clarify any confusing points

- Moving on to food-drying...



Food drying – dehydration principles

Drying of foods is driven by interactions between the atmosphere and the surface of the product.

- Not a targeted process specific to H-O-H and will draw out many molecules along with the water that leaves the product. We always SMELL a drying operation before we SEE it.
- Product is subjected to dramatic physical changes as drying takes place.
 - Case hardening, encapsulation
- Water is moving from a liquid state to a vapor state – evaporation.
- Subject to thermal degradation of the product (heat damage)

Food-drying – dehydration principles

- Dehydration fundamentally changes the physical properties of the solids involved.
 - Dehydrated foods do NOT rehydrate well
 - Raisins vs grapes
 - Prunes vs plums
 - Jerky products
- Case hardening
 - Cell structure collapses at the surface during onset of drying process.
 - Collapsed cell structures are further damaged by heat and by interactions with moving crystals.
 - Salts, sugars, complex carbohydrates, and proteins can all create a 'case' on the surface of a product trapping moisture inside.

Food-drying – dehydration principles

- **All drying relies upon gradients.**
- Gradients: Two dissimilar conditions adjacent to each other will seek to reach an equilibrium condition.
 - Pressure gradient (particularly vapor pressure)
 - Moisture gradient
 - Temperature gradient
 - Salinity
 - Acidity

Food-drying – conduction or convection

We use multiple types of energy to dry product so there are different types of drying.

- **Conductive** - Energy moves in the product via **direct physical contact** (electric burner to a pan to a pancake)
 - Think 'all your ducks in a row' to remember conduction.
- **Convective** - Process uses the energy of motion to help establish gradients that facilitate drying.
 - **Air currents are constantly moving over the surface** of the product

Food-drying – small scale dryers

- Home dehydrator, convection oven drying, “air fryers”
 - Use low temperature (around 150 degrees F) and forced air
- Traditional home ovens
 - Use conduction and slightly higher temperature (often 200 degrees F to 275 degrees F)
- Solar dryers
 - Use greenhouse effect to create heating chamber
 - Exhausting the moist air from the heating chamber with solar powered fans creates low-level convective currents.

Food-drying – large-scale dryers

- Grain dryers
- Drum dryers
- Spray dryers
- Tunnel dryers
- Cabinet dryers
- Kiln dryers
- Air lift dryers
- Vacuum dryers
- And many others

Far too many
options to list
here!

Food-drying – pre-process

- Increasing surface area will increase ease of drying
 - Smaller distance for water to travel to reach the atmosphere.
 - More direct product contact with the atmosphere.

Consider these apples:



Food-drying – preparation

- Both are placed on a rack uncovered and held in oven for three hours at 275 degrees F.

Consider these apples:



Non-uniform slices, notable water escaping during preparation



Prior to thermal process of conductive drying

Food-drying – post-process

- Intact outer skin holds substantial moisture in the product.
 - Trapped moisture has boiled the internal structure of apple during heating.
 - Moisture could not escape the product.



Only outer skin contacts the atmosphere.



Note the leathery character of the exterior creating a jacket or “case” that is trapping the moisture.



Food-drying – post-process

- Both are placed on a rack uncovered and held in oven for three hours at 275 degrees F.

Consider these apples:



Thin slices became dark and brittle.
Thicker slices remain slightly flexible.



Can you identify the major factor
leading to the difference?

Moisture content!

Darkening of the starch and fiber indicates that so much water has been removed from the product that the energy available for vaporization has now been used for roasting these thin slices.

Food-drying – dehydration principles

- The exact same process and conditions will have very different results determined by the product preparation.
- No slicing vs thicker slice vs thinner slice
 - All require different amounts of distance for the water to move
 - IF the prep is not uniform, then different energy and time requirements will be needed to reach the same level of drying.

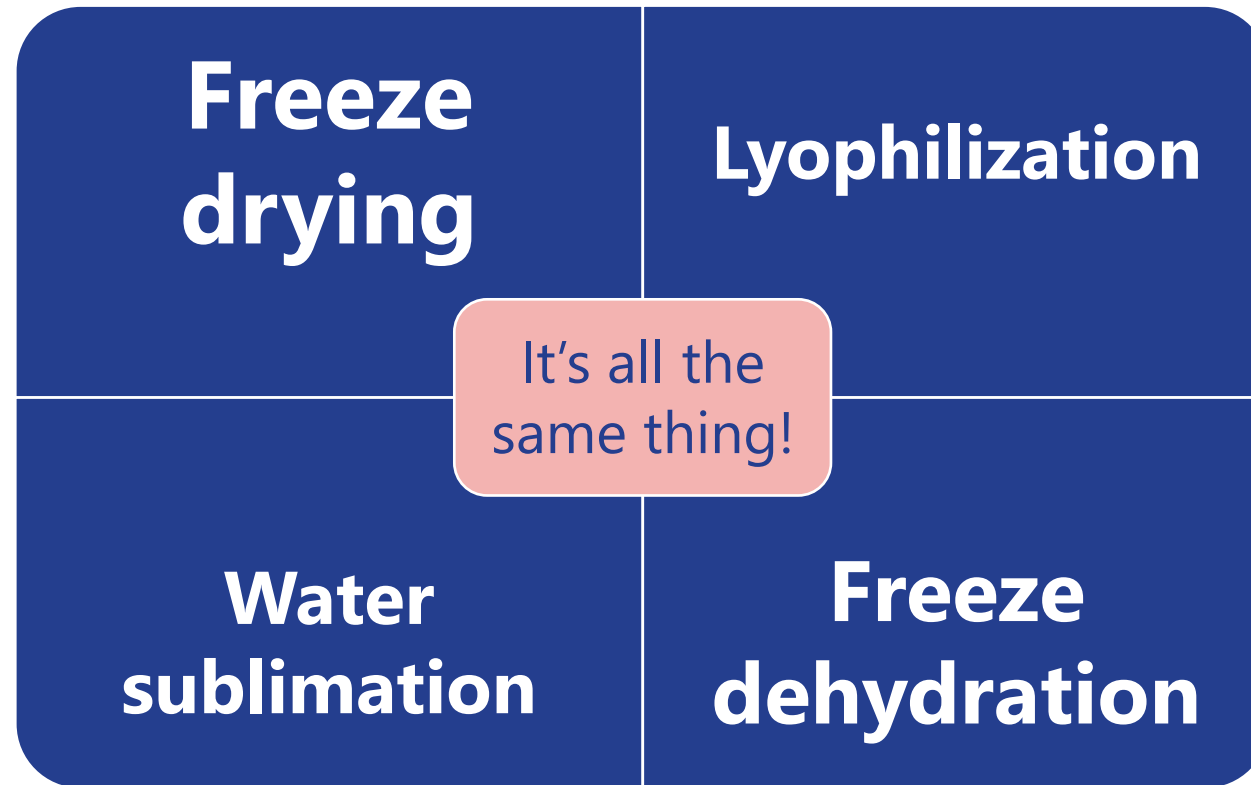


Food-drying – pause for chat questions

- Questions in chat about drying?

- Moving on to freeze drying

Freeze-drying: one process, many names



The fundamentals

- Does not kill most microorganisms
- Targeted process removing H-O-H and leaving other compounds intact
- Water changes from solid to gas without entering the liquid phase – sublimation.
- Product structure is protected, so rehydration works well

Four required steps

- 1. Properly prepare the product**
- 2. Freeze the product**
- 3. Apply a strong vacuum**
- 4. Add energy for phase change**

Properly prepare the product

- Maximize surface area
- Expose product to atmosphere in the chamber
- Enzyme inactivation
 - Often via blanching or acids
- Preparation is similar to other drying methods.

Freeze the product

- Freeze product as quickly as possible
- Product must reach eutectic point for this process
- Common to pre-freeze products



Freeze the product

- Blended products like trayed meals or vegetable stew need special consideration
- Eutectic point requirements for potatoes will be different than requirements for celery, carrots, or sauce
- Must reach eutectic point for most difficult component

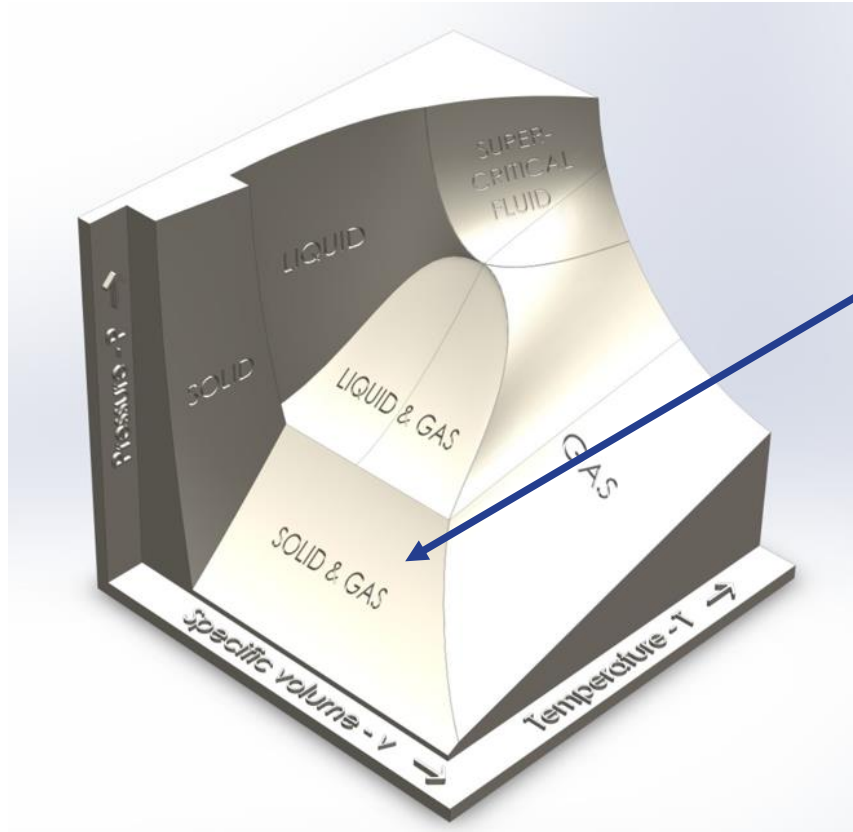
Apply a strong vacuum

- Space is considered a 'perfect vacuum'
- Space is measured at 30 inHg
- Effective freeze-drying vacuum levels need to reach at least 28 inHg
- Quality vacuum pumps and chamber seals are critical to freeze-drying success

Apply a strong vacuum

- When does water boil?
 - Nope! It isn't always 212 degrees F...
- Consider Death Valley - more temp required to boil.
- Consider Denver – less temp required to boil
- Lower the pressure of the atmosphere, and we lower the temperature needed for phase change.

Apply a strong vacuum



- We are using the solid and gas section of the phase change model

Phase model credit:
Dr. Natan Zawadzki,
Oxford Thermofluids Institute

<https://grabcad.com/library/p-v-t-phase-change-diagram-1>

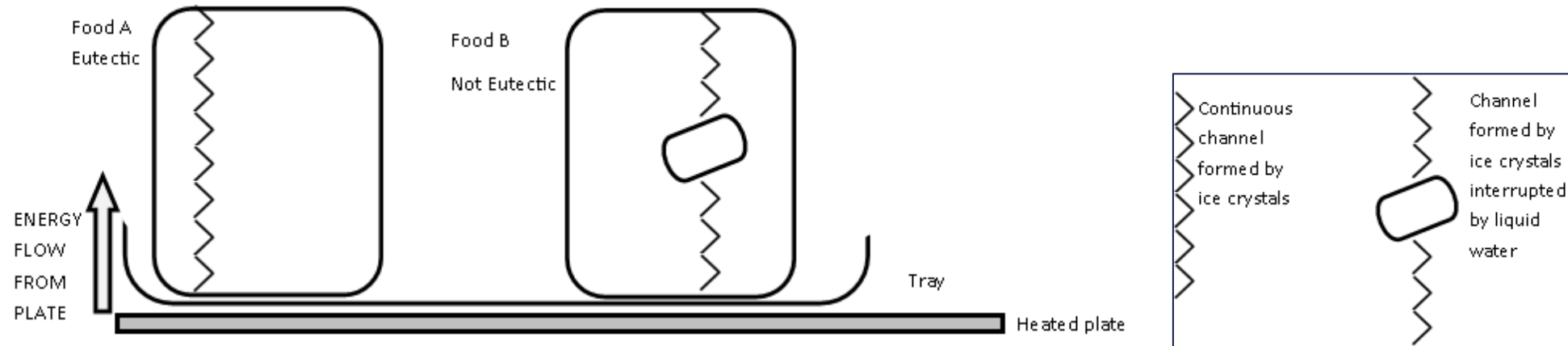
Add energy for phase change

- Turn on the HEAT - seems counterintuitive
- Heat energy from the plate channels through the eutectic ice and triggers sublimation when energy reaches the atmosphere.
- Product is only a conduit if it is in eutectic state
- Ice undergoes sublimation and leaves product as water vapor

Add energy for phase change

- A solid block of pure water in the form of eutectic ice can rest on a plate that is 150 degrees F while undergoing sublimation without ever melting.
- THAT'S SCIENCE!

Add energy for phase change



Pause for chat questions...

- It's a lot of information. There are going to be some questions, and that's a good thing.
- Let's just be sure we all grasp these concepts.

Food safety risks

- Products are contaminated during prep, equipment loading, or packaging
- Products do not reach eutectic point, so water is not properly removed
- Packaging process traps moisture enabling microorganism activity during storage
- Inadequate vacuum levels stop the process from removing water from product
- Rehydrated PHF Product is not treated as PHF

Corrective actions

- ❑ When a freeze-dry process fails:
 - ❑ It IS possible to re-freeze the product, reach eutectic point, and re-run the freeze-drying process.
 - ❑ It IS possible to achieve a safe product even when initial FD fails.
 - ❑ A failed freeze-dry process seriously damages the product quality.
 - ❑ Re-worked freeze-dried products are often not of a suitable quality to be saleable even though they can technically be safe products.

Equipment considerations

- Closed system so water collects in vacuum chamber
- Correct defrosting of coils during extended freezing cycles
- Vacuum oil efficacy and oil life
- Vacuum pump maintenance
- Seal integrity and use of vacuum grease
- Equipment programming
 - For example - ramping temps, holding temps, plate heat vs process time, post heat
- Data capture
- Cleaning process for chamber interior and for trays

Packaging considerations

- ❑ Package quality is primary limiting factor in shelf life of freeze-dried products
- ❑ Needs to provide barriers to moisture, oxygen, and light
- ❑ Often packed in pouches of multi-layer laminates with foil
- ❑ Windows are a bad idea – changes OTR and many other factors.
- ❑ Correctly packaged freeze-dried product can be safely stored at room temperature.
 - ❑ Ideally kept in cool, dark, dry, storage areas
- ❑ Pouch packs can leak at side seals and end seals
- ❑ ROP - Facultative anaerobes will remain in spore state if moisture is absent.
- ❑ If stored in clear packages, product will experience light oxidation over time.

Thank you!

- Thank you for participating in the Indiana Department of Health Food Protection Division's Freeze-Drying Fundamentals & Safety Guidance session!
- The conversation and attentive interest is very much appreciated!
- Please feel free to reach out to me as needed. I'm here to help!
- Final questions and contact information.

Questions?

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Indiana
Department
of
Health

FREEZE-DRIED GUIDANCE (RETAIL)

BRIAN SHORTRIDGE

RETAIL FOOD PROGRAM MANAGER

1/17/2023

Purpose

- Assist LHDs
- Address legal requirements
- Acknowledge best practices

Background

- Common food preservation method for many years
- “Freeze-drying is a process in which water is removed from a product after it is frozen and placed under a vacuum, allowing the ice to change directly from solid to vapor without passing through a liquid phase.”
- Results in a lightweight, dehydrated product

Food safety concerns

- Not a “kill step”
- Many microorganisms can survive freeze-drying.
- Pathogenic organisms can create serious health hazards.
- Although an A_w of less than .85 can be achieved through freeze-drying, according to the FDA it is a specialized process that requires additional precautions.

Freeze-drying best practices to mitigate illnesses

- Proper handwashing
- Approved food sources
- Cleaned and sanitized tools and equipment
- Manufacturer-validated freeze-drying machines
- Product testing re: *Aw*/record-keeping

Freeze-drying best practices to mitigate illnesses

- Shelf stability testing/record-keeping
- ROP variance
- BOAH or USDA inspection of meat products
- USDA inspection of egg products

IDOH expectations

- Perform subsequent Aw value tests
- Maintain records (one year)
- Display “best-by dates”
- Prepare HACCP plan
- Have a corrective action plan

Home-based vendors

- Guidance forthcoming
- Candies
- Chocolates
- Small, intact, uncut fruit



In conclusion...

IDOH leadership intends to encourage local health officials to promote the use of best practices in the freeze-drying process and to educate them of their responsibilities in producing safe foods.

Questions?

Brian Shortridge

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