Hardpack Frozen Dessert Storage – The Factors to Consider in the Age of Clean Label and Plant Based - Prototype Developers Perspective
Overview (Frozen Storage: Inherit Previous Process, Delay the Inevitable)

- Primary Events During Storage
- New Ingredient World – Lean on Process More
- Impact of Freezing Process
- Impact of Packaging and Filling
- Impact of Hardening Process
- Impact of Storage and Distribution Conditions
- Modes of Product Quality Loss
- Impact of Formulation
- Impact of Storage Conditions on Product Development
- Accelerated Shelf Life Testing
Good Frozen Storage Performance – Original product is thermodynamically unstable – 3 phase emulsion will eventually collapse – delay is the goal
“Clean Label Natural” Definition

**De facto eliminations**: mono and diglycerides, Polysorbate 60, 65 and 80, Lactic, Citric, Succinic and Acetic acid esters, DATEM, Lactylates, Propylene glycol and Polyglycerol esters, Propylene glycol and glycerol monostearates, chemically and enzymatically modified lecithins, cellulose derivatives, and most gelatins, most sugar alcohols, polydextrose, HFCS, some maltodextrins, artificial sweeteners and flavors, etc…

**De facto inclusions**: unmodified lecithins, whole eggs, egg yolks, egg whites, usual list of commodity proteins, some protein concentrates and isolates if substantially unhydrolyzed, natural sounding sweetener sources, occasional, grudging acceptance of corn syrup and some sucrose sources, most sources of gums derived from plants with occasional tolerance for xanthan gum and gellan gum and carrageenan and most plant based protein and fat sources (assuming unhydrolyzed or not chemically modified), solvent extracted plant fats not always accepted.
Practical Impact of going “Clean Label”

Taking away well known and tested ingredients means leaning on the process more until and if suitable “clean label” alternatives are found. So, presently and for a period of time in the near future:

- Lower overrun products (i.e. less than 90% overrun) will probably predominate in the plant based category and to a lesser extent in the dairy frozen dessert category (use of egg protein has helped but has not proven to be completely full proof yet)
- Extruded novelties (i.e. 80% overrun and higher) will be more challenging to formulate and produce – integrating the process with the formulation more directly may be necessary
- Quiescently frozen novelties and soft serve products might make for easier product launches
- Shorter shelf life declarations may be necessary in the short run
- Larger package sizes above a quart may require more effort to successfully deploy on the market
  - special emphasis on rigorous hardening/storage processes may be required
- Re-examination of mix making procedures (i.e. hydration procedures, thermal parameters, homogenization, aging practices) may be necessary for some products (see new ingredient functionality – next page)
Why Stabilizers are Hard to Replace

- Gram for gram gums generally exert the greatest influence on water mobility (i.e. ice formation raises the concentration of hydrocolloid in remaining liquid portion).

- Now water control will have to come from more ingredients than just hydrocolloids and emulsifiers.

- Careful balancing of formula freezing points relative to the freezing process and hardening - storage conditions will become more important.

Keeney, 1982
New Ingredient World – Emulsion behavior change dictates a Protein characterization focus for the future

- Protein - Fat interaction is key in finished product characteristics like melt rate, shelf stability, and sensory quality.
- Many plant protein ingredients are relatively new with unknown functionalities, while commonly used plant fats have well documented characteristics.

So, for frozen storage shelf life go back to basics of freezing to accommodate “clean label” ingredients until their functionality starts to improve.

LumiSizer Dispersion Analyzer data indicates significant differences in ability of new ingredients to bind water and form emulsions.

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References:
Clean Label” and/or Plant Based Development Decision Matrix
(conventional ingredients for conventional products have well established guidelines)

New Ingredients > New, undocumented emulsion dynamics > Which development path fits your situation:

1. **General To Specific**: general product concept with new ingredients > lot of time and money to spend on creating several prototypes to choose from > ability to dictate the processing and distribution environment needed for the product prototype after it has been created.

2. **Fit Product to Specific Processing and Distribution Conditions**: limited time and budget > first identify processing and distribution conditions > design the product formulation to fit the processing and distribution conditions > press the marketing people to identify the specific serving and packaging sizes and types ahead of time > press sales/marketing to identify specific tangible (nouns and numbers and competitors) organoleptic targets ahead of time

3. **Identify EOSL’s (end of shelf life parameters)**: sensory, chemical, functional, nutritional, microbiological, physical > many are possible > identify most likely problem(s) that occur soonest (key decision parameters). New products different from ice cream may need to specify different storage conditions.

4. **Decide approved test method for parameters**: collect base line data on competitor product or desired target (i.e. meltdown, ice crystal size, shrinkage, flavor color stability, etc.) > verify that tested mode of failure does occur in the temperature range selected for shelf life testing (accelerated and/or long term).

5. Make sure the testing criteria arrived at in the pilot plant/lab setting conforms to actual likely storage and distribution conditions.
Batch Freezer – Whipped Mix Method

- Batch Freezing: 5-10 minutes drawdown time (8-10 minutes typical).
- Draw Temperatures: 24-25 F (-4.4 C to -3.9 C)
- Overrun: 50-80% typical (100%+ possible)
Batch Freezer – Vertical Effe Freezer ("French Pot" method)

For 75 years the EFFE Vertical Batch Freezer by Cattabriga has set the world’s standard for artisan gelato and ice cream machines. This unique batch freezer allows you to add ingredients as the ice cream is being produced and provides a visible work area to consumers, something impossible to do with a horizontal batch freezer.

The freezer barrel spins while an automated beater and scraper assembly manipulate the product along the freezer wall freezing the mix and folding in the air.

Drawdown Time: 10 minutes (ave.)
Draw Temperature: -8 C to -10 C
Overrun: 20-35%
Batch Freezer – Supercritical Fluid Method (i.e. liquid nitrogen or carbon dioxide)

Pelletized Ice Cream:
Frozen into small pellet sized pieces using liquid nitrogen or carbon dioxide. Special low temperatures often used for the storage cabinets since piece dimensions tend to dictate a quick melt.
Overrun: negligible
Draw Temperature: -196 C (initial contact)
Drawdown time: <1 seconds

Rizvi Pattened Process:
: High-pressure CO₂
Overrun: 30-35%
Draw temperature: -70 C
Drawdown time: <5 seconds

Liquid Nitrogen Immersion:
Combine the two fluids and nitrogen boils off.
Overrun: 15% overrun
Draw Temperature: -196 C
Drawdown time: <30 seconds
Batch – Quiescent Mold (brine bath or blast freezer)

Products such as popsicles or ice cream or gelato with flat sides on sticks are manufactured by filling a mold with mix or soft frozen mix and then placing in a blast freezer.

*Drawdown Time: 10-30 minutes (static brine or blast freezer)*

*Draw Temperature: 25-26 F (-3 C to -4 C)*

*Overrun: 0-20%*
Continuous Freezer – Conventional Whipped Mix Method

**Drawdown Time:** 24 seconds
**Draw Temperature:** 21-22 F
**Overrun:** 30-110% (80-90% typical)
Conventional Freezer – Dasher Modifications

- Lower displacement volume
- Longer residence time in barrel
- More whipping time

- Higher displacement volume (up to 80%)
- Less residence time in barrel
- Higher shear rate
Conventional Freezer – “CREAM” Process (recirculated mix) Whipped Mix Method (patented)

Drawdown Time: 26-36 seconds
Draw Temperature: 22 F (-5 C) to 10 F (-12 C)
Overrun: 80 – 150%
Continuous Freezer – Supercritical Fluid Assisted (recirculated mix) Whipped Mix Method

*COLDFRONT™* ICE CREAM HARDENING

Pulls off about 10% of the originally soft frozen product and portions it into small bits that are deep frozen and then added back to the main product stream in a fruit feeder before total product filling.

*Note: consider for ripples and denser - warmer inclusions*

**Drawdown Time:** same as other continuous

**Draw Temperature:** same as other continuous

**Overrun:** 80-150%
Continuous Freezer – “Deep Blue” Low Temperature Extrusion Whipped Mix Method

ULTICE (ultra low temperature ice cream extrusion)

ULTICE (Swiss university project) evolved to Deep Blue two-stage freezer at Hoyer.
- First stage is similar to conventional whipped mix continuous freezer (exit 21-22 F with overrun)
- Second stage is a cold kneading extruder with product exiting at 10 – 5 F.
- Consider for low freezing point formulas

Drawdown Time: 48 seconds (estimate)

Draw Temperature: (-12 to -15 °C)
- 10 to 5 °F

Overrun: 80 – 120%
Continuous Freezer – Extrusion Freezing

Begins with a conventional whipped mix method then shaped, cut, deposited, decorated and hardened. Other variations possible (i.e. 2 mix co-extrusion).

Drawdown Time: 1-10 minutes

Draw Temperature: 19-21 F (-6 to -7 C)

Overrun: 80-110%
Continuous Quiescent Mold Freezing

Starts out as cold mix from a flavor tank or partially frozen mix from a continuous freezer that is filled into molds and partially frozen until stick insertion and extraction are completed before packaging and hardening. Many variations possible.

- **Drawdown Time**: 2-4 minutes (before extraction)
- **Draw Temperature**: 25-26 F (-3 to -4 C)
- **Overrun**: 0-50%

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Time vs. Temperature Chart – Setting Initial Ice Crystal Size By Type of Freezer

Typical Freezing Performance (technical literature, industry sources)

*Drawdown Time = initial mix temperature to draw temperature - assuming 40°F (5°C) mix temperature*

<table>
<thead>
<tr>
<th>Drawdown Time (seconds)</th>
<th>Draw Temperature (°F)</th>
<th>Type of Freezing</th>
</tr>
</thead>
<tbody>
<tr>
<td>480</td>
<td>24</td>
<td>Batch Horizontal</td>
</tr>
<tr>
<td>600</td>
<td>17.6</td>
<td>Batch Vertical (Effe gelato - &quot;French Pot&quot;)</td>
</tr>
<tr>
<td>4.5</td>
<td>-94</td>
<td>Batch Supercritical Fluid</td>
</tr>
<tr>
<td>1200</td>
<td>25</td>
<td>Batch Quiescent Mold</td>
</tr>
<tr>
<td>24</td>
<td>22</td>
<td>Conventional Continuous</td>
</tr>
<tr>
<td>30</td>
<td>10</td>
<td>&quot;CREAM&quot; Continuous (recirculation)</td>
</tr>
<tr>
<td>48</td>
<td>5</td>
<td>&quot;Deep Blue&quot; Continuous (2 stage)</td>
</tr>
<tr>
<td>180</td>
<td>25</td>
<td>Continuous Quiescent Mold</td>
</tr>
</tbody>
</table>
Critical Effect of Draw Temperature (freezer has more efficient heat transfer than the hardener)

***Draw Temp vs. Hardening Times***

- Package: 4 – 56 oz. SqRd
- Flavor: Pumpkin Crème
- Air Temp.: -23°F
- Air Velocity: 1000 FPM
- Core Temp In: +23.2°F
- Core Temp Out: -0.1°F
- Retention Time = 5 Hr 28 Min

- Package: 4 – 56 oz. SqRd
- Flavor: Pumpkin Crème
- Air Temp.: -23°F
- Air Velocity: 1000 FPM
- Core Temp In: +19.3°F
- Core Temp Out: -0.2°F
- Retention Time = 4 Hr 4 Min (26% less time)

At -10°C / 14°F 70%

At -8°C / 18°F 65%

At -5.5°C / 22°F 50%

Note: draw temperature should be lower with warmer inclusions (i.e. nuts, fudge, etc.)
Setting Initial Air Cell Size – Equipment Effects

- **Note:** Smaller well distributed air cells have been associated with hardpack products retaining smaller ice crystals above 50% overrun (air is good insulator)
- Pre-aeration can be used with any continuous freezer process and has been suggested to require less energy input than other processes
- Dasher design and speed with respect to barrel residence time can be used to partially substitute or supplement a pre-aeration step
- “Deep Blue” two stage freezing could potentially substitute for pre-aeration
Impact of Barrel/Back Pressure – Filling Line Pressure

Need viscosity and stiffness to support bubble formation:
- minimum mix viscosity necessary in beginning
- Sufficient ice formation necessary at exit of freezer
- Dependent on texture needed for particular package to be filled uniformly
- Use minimum barrel pressure necessary to generate needed texture (i.e. co-dependent with draw temperature, overrun, mix-air flowrate, valve settings)
- Can be affected by elevation of manufacturing plant (i.e. mountain vs. sea level) – significantly air volume expansion possible via Boyle’s Law

Ice Cream Viscosity at \(-5.5^\circ C\) = 1x
Ice Cream Viscosity at \(-8.0^\circ C\) = 5x
Ice Cream Viscosity at \(-10^\circ C\) = 14x

Note: “ice cream viscosity” refers to torque load on dasher – frozen foam is variably compressible. Flow curves for ULTICE process (i.e. smaller bubbles under high pressure)

Summary Notes:
- Larger pressure differentials need smaller air bubbles to survive filling operation and storage
- Use shortest practical line from freezing operations to filling operations – 3 phase emulsion is sensitive to temperature and shear stress at this point

The variation of pressure with altitude.

Create stress on air bubbles
Impact of Dasher/Agitator/Scraper Speed

- Interacts with previously mentioned variables (i.e. draw temperature, product viscosity, overrun, dasher design, formula composition)
- *Increased dasher speed tends to increase partial coalescence of fat and hence affects texture perception, product meltdown performance, stability of air cells, size of air cells, size of ice crystals and freezing efficiency*
- Finding an acceptable speed that sufficiently enhances heat transfer and reduces the ice crystal size and air cell size while not adding any more heat to the product (via viscous energy dissipation) than necessary since the refrigeration system must remove this excess heat
Post Freezer Effects – Filling, Packaging and Wrapping

What to avoid:
- long lines with lots of elbows to the filler from the freezer
- long lines between the filling operations and the boxing/wrapping equipment
- long lines between the boxing/wrapping equipment and the hardening equipment

Note: these only add additional time for initial hardening to be completed as well as additional heat to be removed; so maximum efficiency is critical especially for “clean label” or “natural” products” without hydrocolloids and emulsifiers.
Hardening – Description and Parameters to Target

- Most efficient, quick freezing and heat transfer has already taken place mostly by convection (largest number small ice crystal nuclei)
- Rest of the heat transfer takes place slowly by comparison mostly by conduction (quiescent). Ideal package shape has the smallest possible internal cross section in two or three dimensions depending on hardener design.
- Must do as quickly as practical to not allow small ice crystal nuclei to melt. The rest of the ice crystals form off of these small nuclei – fewer nuclei mean larger crystals during hardening and subsequent storage (see graph).

Targets:
- 45-50% of water is crystalline going to the hardener – keep it that way or push higher if possible and practical
- For ice cream try to get core temperature to 0°F (-18°C) in 2-4 hours; may need to change for some “clean label” and plant based formulas
- Reduce “skinning” and large ice crystals on container opening and top of product by maintaining a constant temperature drop in product with -25°F (-32°C) high velocity air movement; outside layer of product is most vulnerable
- Faster hardening reduces stress on air bubbles
Hardening Systems I – After the freezer and packaging

Note: partial hard freezing of novelties before packaging not considered here though still part of the total freezing process profile.

- Uses liquid N₂ or liquid CO₂
- Low residence time – very quick freezing
- Expensive
- Packaged product can be immersed in glycerol, propylene glycol, brine solutions

- Low operating cost
- High capital cost
- Size and shape limited (thin and flat only)
Hardening Systems II – After Freezer and Packaging

FUSION CELL™

- Uses conduction/convection – most efficient
- Multiple sizes and shapes simultaneously*
- Medium capital expense
- Minimum size 750 GPH

Static Manual Hardening Room

- No product movement
- Limited air movement
- Least expensive, easy retrofit, small plants

- Many configurations possible (i.e. spiral, oval, variable retention time, etc.)*
- More expensive, less efficient

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Example – Post Freezing Temperature Profile

**TRI-Tray Hardening Test (R.C. Greener)**

<table>
<thead>
<tr>
<th>Sample Type</th>
<th>Residence Time in Hardening Chamber (core temperature at 0°F)</th>
</tr>
</thead>
<tbody>
<tr>
<td><em>Individual Scround</em></td>
<td>2 hours, 13 minutes</td>
</tr>
<tr>
<td><em>6 plastic overwrapped Scrounds</em></td>
<td>4 hours, 33 minutes</td>
</tr>
</tbody>
</table>

Note: above is true if significant inclusions have been prechilled or frozen or if the draw temperature is dropped to compensate for warmer ingredients.
Storage (after hardening) – Packaging and Overrun

- Good size and shape for hardening may be more vulnerable in storage if there is too much temperature cycling (i.e. balance this dichotomy with choices of packaging material, formulation choices and selection of freezing/hardening processes)
- Higher overrun gives some protection due to the increased insulation value with conduction being the primary heat transfer mode
- Sensitive to the freezing point of the formula and mix viscosity of the melted product
Storage (after hardening) and Distribution Conditions

Ideal Design Parameters:

- **– 20°F (-28.9°C) air temperature** (colder temperatures for extended time can lead to freezer burn)
- low velocity air movement (3-4 air exchanges/hr)
- minimize temperature fluctuations – product at the edge of container suffers most
- distribution temperature never higher than retail cabinet temperature (i.e. max 10°F)
- organize packing and stacking eliminate dead air flow and high temperature zones
- product locations in storage organized around FIFO (first in first out) concept
- inventory pattern directed by production planning from an updated sales plan
- always harden product fully (core temperature reaches 0°F) before sending to storage
- use ice cream distribution system (i.e. target temperatures -20°F to -10°F) vs. frozen food distribution (i.e. target temperatures 0°F to 10°F); DSD (direct store delivery) is best when possible
- monitoring storage facility and distribution for temperature compliance (see pictures below)

> **GOOD STORAGE IS ABOUT PRESERVING WHAT IS GIVEN FROM THE FREEZING /PACKAGING PROCESSES** – So initial crystal size and air cell size and partial coalescence has to be set correctly
Either design your product and packaging for the above mentioned situation or move heaven and earth to avoid it. Good “clean label” product from the plant can easily be destroyed by distribution conditions like the above.

<table>
<thead>
<tr>
<th>Temperature Change</th>
<th>Time in hours</th>
<th>Concentrated juices and fruit pies</th>
<th>Other frozen foods</th>
</tr>
</thead>
<tbody>
<tr>
<td>30 to 5</td>
<td>-1 to -15</td>
<td>45</td>
<td>33</td>
</tr>
<tr>
<td>25 to 5</td>
<td>-3 to -15</td>
<td>43</td>
<td>23</td>
</tr>
<tr>
<td>20 to 5</td>
<td>-6 to -15</td>
<td>39</td>
<td>18</td>
</tr>
<tr>
<td>15 to 5</td>
<td>-9 to -15</td>
<td>30</td>
<td>13</td>
</tr>
<tr>
<td>10 to 5</td>
<td>-12 to -15</td>
<td>19</td>
<td>7</td>
</tr>
<tr>
<td>30 to 10</td>
<td>-1 to -12</td>
<td>26</td>
<td>26</td>
</tr>
<tr>
<td>25 to 10</td>
<td>-3 to -12</td>
<td>24</td>
<td>16</td>
</tr>
<tr>
<td>20 to 10</td>
<td>-6 to -12</td>
<td>20</td>
<td>11</td>
</tr>
<tr>
<td>15 to 10</td>
<td>-9 to -12</td>
<td>11</td>
<td>5</td>
</tr>
<tr>
<td>10 to 15</td>
<td>-10 to 0</td>
<td>15</td>
<td>20</td>
</tr>
<tr>
<td>25 to 15</td>
<td>-3 to -9</td>
<td>13</td>
<td>10</td>
</tr>
<tr>
<td>20 to 15</td>
<td>-6 to -9</td>
<td>9</td>
<td>5</td>
</tr>
<tr>
<td>30 to 20</td>
<td>-1 to -6</td>
<td>6</td>
<td>15</td>
</tr>
<tr>
<td>25 to 20</td>
<td>-3 to -6</td>
<td>4</td>
<td>5</td>
</tr>
<tr>
<td>30 to 25</td>
<td>-1 to -3</td>
<td>2</td>
<td>10</td>
</tr>
</tbody>
</table>
Ice Cream – Frozen Desserts are three phase (air, fat, water) emulsions that require freezing to remain stable for a limited period of time (i.e. shelf life). Collapse in one form or another is inevitable. Successful frozen storage is about increasing the delay of all failure mechanisms.
Oxidation Color Change (bulk portion example) – Oxidation Flavor Change (bulk portion example)

**Natural Red Color:**
- one source is red beet juice
- color largely results from the presence of betanin
- Not tolerant of most pasteurization temperatures
- stability is largely pH dependent (best in the 4-5 range)
- problematic in some dairy mixes
- overrun aggravates the color change

**Partial substitution of omega 3 oil sources:**
- oil source can make a difference
- form of the oil that is added to the mix can make a difference (encapsulation can keep out oxygen)
Inclusion Failure

Water Activity of Pure Water Ice

Possible Modes of Failure:
- Physical collapse (exposure to moisture or temperature gradient)
- Microbiological foodborne illness
- Color/flavor change

Typical Ice Cream $a_w$ Data: 0.90-0.93

Typical Nut Piece $a_w$ Data: 0.20-0.80
Ice Crystal Size Increase = Recrystallization

- Five types of generally known recrystallization (migratory, accretive, isomass, irruptive, pressure-induced).
- Migratory (also called Ostwald ripening) is the main mechanism in frozen desserts.
- Defined as increase in average size and reduction in average number of crystals owing to growth of larger crystals at expense of smaller ones due to fluctuations in storage temperature.

Why do these factors lead to rapid coarsening?
- Less increase is needed before crystals are detectable
- More liquid water, diluted stabilizer solutions
- Temperature fluctuation
- Small crystals melt more than large and the liquid water refreezes on the large

45 μm Sensory Threshold

Recrystallization Rate (μm/s)

Amplitude of Temperature Fluctuations, ΔT (°C)

Percent of Ice Crystals Less Than Size on the X-axis

Ice Crystal Size (μm)

Before Cycles
15 Cycles
50 Cycles
Air Cell Collapse - Shrinkage

Three observed stages for ice cream air cell increase and eventual release (shrinkage) during storage:

- **Coalescence** – two small air cells fuse to one
- **Disproportionation or Ostwald ripening** – large air cells grow at the expense of small ones due to pressure gradient between small air cells and large air cells
- **Drainage** – channeling/tunneling between large air cells to the extent that air escapes the matrix

Note: air cells are good insulators and hence give good heat shock resistance above 50% overrun and also correlate with creamy mouthfeel and good meltdown performance and good profitability; **but the most feared form of product failure**.
New Ingredient Categories:
Proteins and Solids Not Fat (SNF) – effects on formulation assumptions

Conventional dairy ice cream formula – maximum overrun estimation equation => (%Fat + %MSNF + %Total Solids) x 2 = % overrun -> no longer reasonably valid since composition assumptions for fat and Solids Not Fat have changed for plant based and mixed source and high protein products

- **Dairy** – Milk protein solids, Milk solids-not-fat (MSNF)
  > Condensed milk, NFDM, MPI, Whey, Buttermilk solids, etc.

- **Plant** – Plant protein solids, Plant solids-not-fat (PSNF)
  > Plant proteins:
    - Different make up of proteins/functional types (ex albumins, globulins) with different isoelectric points (pKi), no plant protein has the same characteristic as casein micelles, do not have the same digestibility or protein score as dairy protein (impacts nutritional %DV).
    - The degree of protein denaturation can vary depending on processing and this impacts solubility and water binding.
  > **PSNF** - Plant solids can contain a variety of functional carbohydrates (ex. Sugars (mono- and disaccharides), starches, fibers) that influence mix and frozen dessert characteristics, flavor and digestibility.
  > **Sources**: legumes, seeds, nuts and grains (ex Soy, Pea, Hemp, Canola, Chia, Peanut, Fava, Coconut, Cocoa, Almond, Cashew, Hazelnut, Oats)
  > **Dry** - Flour, defatted flour, meal, concentrates, isolates, hydrolysates
  > **Fluid** – “milks”
  > **Butter** – nut & seed creams
Critical ingredient differences:

5 examples

[note:sugars from plant sources tend to be monosaccharides (except maltose) vs. disaccharides like surose and lactose = 2x freezing point depression]

<table>
<thead>
<tr>
<th>COMPOSITION</th>
<th>Units</th>
<th>Defatted Soy Flour</th>
<th>Cashew Butter</th>
<th>Pea Protein Isolate 1</th>
<th>NFDM</th>
<th>MPC 50</th>
</tr>
</thead>
<tbody>
<tr>
<td>Protein</td>
<td>g/100 g</td>
<td>50</td>
<td>19</td>
<td>80</td>
<td>34</td>
<td>50</td>
</tr>
<tr>
<td>Total Fat</td>
<td>g/100 g</td>
<td>1</td>
<td>50</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Total Carbohydrates</td>
<td>g/100 g</td>
<td>34</td>
<td>28</td>
<td>3</td>
<td>51</td>
<td>37</td>
</tr>
<tr>
<td>Dietary Fiber</td>
<td>g/100 g</td>
<td>19</td>
<td>3</td>
<td>2</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Lactose</td>
<td>%</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>51</td>
<td>37</td>
</tr>
<tr>
<td>Sugars</td>
<td>g/100 g</td>
<td>15</td>
<td>3</td>
<td>0</td>
<td>51</td>
<td>37</td>
</tr>
<tr>
<td>Total Solids</td>
<td>%</td>
<td>92</td>
<td>95</td>
<td>95</td>
<td>96</td>
<td>96</td>
</tr>
<tr>
<td>Solids Not Fat</td>
<td>%</td>
<td>91</td>
<td>45</td>
<td>94</td>
<td>95</td>
<td>94</td>
</tr>
<tr>
<td>Relative Sweetness</td>
<td>g/100 g</td>
<td>7</td>
<td>6</td>
<td>0</td>
<td>7</td>
<td>5</td>
</tr>
<tr>
<td>Sucrose Equivalence</td>
<td>g/100 g</td>
<td>28</td>
<td>6</td>
<td>2</td>
<td>52</td>
<td>36</td>
</tr>
<tr>
<td>Ash</td>
<td>%</td>
<td>7</td>
<td>3</td>
<td>6</td>
<td>10</td>
<td>8</td>
</tr>
<tr>
<td>Sugar/Ash Ratio</td>
<td></td>
<td>2</td>
<td>1</td>
<td>0</td>
<td>5</td>
<td>5</td>
</tr>
</tbody>
</table>
Impact of ingredients on Freezing Point Depression: Minerals & buffering salts

- Plant based ingredients may contain highly variable amounts of minerals and buffering salts from processing (may not be labeled!)

- Minerals and buffering salts contribute to Sucrose Equivalence impacting freezing point depression.

- Example: Sodium citrate
  
  > 100g sodium citrate is equivalent to 466g sucrose in its ability to depress the freezing point

\[
\text{Citrate} \\
\text{Molecular Wt. 189.1}
\]
Impact of ingredients on Freezing Point Depression
Sugars & minerals calculation example

<table>
<thead>
<tr>
<th>Techwizard Formula Simulation</th>
<th>Salt - 3% Solution</th>
<th>Lactose - 3% Solution</th>
<th>Glucose - 3% Solution</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sucrose Equiv (%)</td>
<td>17.7</td>
<td>3.0</td>
<td>5.8</td>
</tr>
<tr>
<td>Total Solids (%)</td>
<td>3.0</td>
<td>3.0</td>
<td>3.0</td>
</tr>
<tr>
<td>Freezing Point (F)</td>
<td>30.0</td>
<td>31.7</td>
<td>31.4</td>
</tr>
<tr>
<td>Difference Reference</td>
<td>-1.6</td>
<td>-1.3</td>
<td></td>
</tr>
<tr>
<td>Freezer Exit Temperature (F)</td>
<td>25.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>% Product Frozen</td>
<td>67.5</td>
<td>91.9</td>
<td>87.4</td>
</tr>
<tr>
<td>Difference (%) Reference</td>
<td>36.2</td>
<td>29.5</td>
<td></td>
</tr>
</tbody>
</table>
**Lipid Ingredient Choices**

- **HOSO** = High Oleic Sunflower Oil
- **PKO** = Palm Kernel Oil

Affects texture, coldness perception, meltdown performance (heat shock resistance), flavor release, shelf life (oxidation sensitivity), mix preparation, overrun level

<table>
<thead>
<tr>
<th>Typical Data</th>
<th>Density (g/ml)</th>
<th>Crystallization Onset Temperature (°C)</th>
<th>Melting Point Temperature (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Milkfat</td>
<td>0.91</td>
<td>16-17</td>
<td>28-36</td>
</tr>
<tr>
<td>High Oleic Sunflower Oil</td>
<td>0.91-0.92</td>
<td>-45.8</td>
<td>4.4-7.2</td>
</tr>
<tr>
<td>Soybean Oil</td>
<td>0.92</td>
<td>-10.2</td>
<td>-22</td>
</tr>
<tr>
<td>Palm Kernel Oil</td>
<td>0.86-0.87</td>
<td>7.0</td>
<td>28.3</td>
</tr>
<tr>
<td>Coconut Oil</td>
<td>0.92</td>
<td>15.0</td>
<td>26.5</td>
</tr>
<tr>
<td>Canola Oil</td>
<td>0.92</td>
<td>-17.1</td>
<td>-9.0</td>
</tr>
</tbody>
</table>

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Impact of Emulsifiers

- Aids in emulsion formation with oil/fat in a mix and for aeration/texture control (adding the third phase of air) in the finished frozen product along with some water control functionality. Mechanism of function starts out with lowering surface tension but mostly seems to function by enhancing partial fat coalescence in carefully controlled amounts (i.e. too much leads to churning – fat chunks). This helps create more fine dispersed and more stable air cells.

<table>
<thead>
<tr>
<th>No PGMS</th>
<th>With PGMS</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="before.png" alt="Image" /></td>
<td><img src="after.png" alt="Image" /></td>
</tr>
</tbody>
</table>

“Clean Label” Emulsifiers:

- egg yolks, egg whites, whole eggs
- Lecithin – soy, sunflower, canola, other sources possible
- Buttermilk
- Denatured whey proteins
- Hydrolyzed proteins (casein, whey, soy, pea, other sources possible)
Impact of stabilizers: Controlling moisture flow during storage and distribution

- Hydrocolloids still required for “clean label” and plant based frozen desserts – new sources under development (endogenous proteins, starches and fibers)

- Improves shelf stability
  > Improves heat shock / freeze-thaw stability
  > Slows ice crystal formation
  > Supports air cell formation (viscosity)

- Affects eating properties
  > Texture (chew, icy, smoothness)
  > Coldness (eating temperature)
  > Flavor release
  > Slows melting (increased melt viscosity) [Schenz (1994)]

"skinning"

Heat Shock Resistance

Viscosity

Glassy, Leathery, Rubbery, Flow

Rubbery Viscous Flow

Temperature

Tg, Tm

10^15 poise

“Skinning”
Bench Level and Pilot Plant Evaluations

Building The Formula: Hardpack

- Starts as two phase oil in water emulsion
- Emulsion is frozen and often has a third phase (air) added to it
- What fat, total solids and overrun (i.e. final product bulk density)?
- What sweetness level?
- How is the product mix pasteurized and homogenized (i.e. temperatures, pressures and times)?
- How long and at what temperature is the product mix aged?
- What is freezing point of the mix relative to the drawing
- Temperature out of the freezer and the depositing temperature in the package?
- What inclusions (i.e. candy – fruit pieces, variegating syrups), if any, are involved with the finished product – level of inclusion, sizes, temperature?
- What is the size and shape of the package?
- What is the hardening temperature profile for the product?
- What does the temperature and time profile for the distribution of the product look like?
- What is the expected shelf life of the product?
Testing Considerations

Factors affecting emulsion stability and suitability for freezing/aeration step:
- oil/fat droplet concentration (fat level, total solids)
- water phase viscosity (amount and type of stabilizer)
- oil/fat droplet size (homogenizer pressures and stages)
- fat density difference with water (amount and type of emulsifier)
- solid fat content of the oil/fat used (preheat temperatures)
- presence of surface tension reducing/emulsifying ingredients

Generic Mix Procedure Notes:
- O/W emulsion - moderate refrigerated stability (i.e. susceptible to partial coalescence in freezer)
- Ensure some fat crystallization (i.e. aging) for higher overrun products (i.e. ≥ 50% overrun)
- Plant based – Mixed Source introduces hardfats (i.e. raw mix preheating) and liquid oils that are mostly unemulsified (i.e. homogenization changes).
- Many small batches for screening purposes

Helpful Tip: If it is pinholing out of the freezer, reformulate
Accelerated Heat Shock Methods

- Data from Slide # 31 can be used to identify practical upper and lower temperature limits and times.
- Can also be used to identify best and worse case scenarios for storage and distribution to set parameters for accelerated or extended shelf life testing.
- New products without a track record for comparison may need to rely on freezing point calculations and/or $T_g$ data to set initial accelerated test parameters.

Note: shelf life testing starting to be required by third party auditing organizations.

Some typical ice cream type methods from notes and industry contacts:
- Programmable freezer cycles - 0°F to 20°F two times in 24 hours
- Legacy procedure - nonfat/lowfat products: 5 days - 10-20 minute daily exposure - room temperature (22-25°C)
- 12 cycles of 0°F- 20°F for 10 days
- Potential possibilities are endless

Sometimes this information is held confidential. Industry has been moving to automated testing chambers.
Additional Considerations for Shelf Life Testing

- Cycling tests
- Steady state static tests
- Temperature and Humidity Control

Note: cup size = 3.5 fl. oz.
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Possible Ingredient Sources at Agropur:
- IsoChill 8000 and 9000 low temperature microfiltered (whey protein)
- BiPro series – ion exchange processed (whey protein)
- Cornerstone – filtered, standardized (casein and whey protein)
- Keystone – hydrocolloids and emulsifying ingredients
- Darigen – proteins combined with stabilizers and other bulk ingredients

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Thank you for your time today, do you have any questions?