Ice Cream at a Structural Level

- Ice crystals
  - Provide cooling effect and hardness
- Air cells
  - Reduce density
- Partially-coalesced fat globule network
  - Affects melt-down rate and hardness of ice cream
- Proteins and hydrocolloids
  - Network in serum phase
- Serum phase
  - Dissolved sugars, minerals, proteins, etc.
  - Some liquid even at very low temperature

Van Wees et al., 2021
Ice Cream Processing

**Freezer**
- refrigerant

**Hardening**
- cold air
- -30 °C

**Storage and Distribution**
- 75-80% frozen
- -10 to -20 °C

- 45-50 µm
- 50% frozen
- 10-70% fat destabilization

- 30-35 µm
- 80-100% overrun
- 10-70% fat destabilization

**Ice**
- nucleation
- growth

**Air**
- incorporation
- breakdown

**Lipid**
- growth
- partial coalescence

**Ice**
- growth

**Air**
- coalescence

**Lipid**
- growth

**Ice**
- melting
- growth
- ripening

**Air**
- coalescence

**Lactose**
- crystallization

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**Scraped Surface Freezer (SSF) Development of Structures**

- Formation of ice crystals
  - Scraping of slush off wall of freezer; mixing of slush in center of barrel; ripening and growth to form ice crystal size distribution

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Cook & Hartel, 2010
Scraped Surface Freezer (SSF) Development of Structures

• Continued crystallization of lipid during freezing
• Fat destabilization
  – Breakdown of emulsion due to shearing forces in freezer; partial coalescence due to liquid fat

50% OR, 500 RPM

0:0 ER, 5.9%  
100:0 ER, 19.6%  
90:10 ER, 28.3%  
80:20 ER, 56.2%

Warren & Hartel (2017)

Scraped Surface Freezer (SSF) Development of Structures

• Aeration
  – Increase in overrun; breakdown of air cells into tiny bubbles; development of air cell distribution; stabilization of air cells by proteins, destabilized fat globules and viscous unfrozen matrix

Chang (2002)
Scraped Surface Freezers

- Exactly what goes on within the barrel of the freezer with all of these structures being developed at the same time is still uncertain
- Recent attempts at modeling the processes within the freezer may provide better understanding

Residence Time Distribution (RTD)

- The path of an element of fluid from inlet to outlet of a scraped surface heat exchanger is complicated
  - Scraping at wall and distribution of cooler fluid into the center of the barrel
- This complicated flow pattern results in a distribution of times for any element to dwell within the heat exchanger

Yataghene et al., 2008
Residence Time Distribution (RTD)

- Some fluid elements exit earlier than others
- Not all fluid elements see the same conditions within the freezer barrel
  - Some ice crystals remain in the barrel longer and can grow to larger size than those that exit much quicker
  - Similar for air bubbles and partially-coalesced fat globules
  - This behavior explains, in part, the distribution in sizes of these structural elements

Fayolle et al., 2013

Measuring RTD in a Scraped Surface Freezer

Measure RTD for 5 different dasher designs at different operating conditions to correlate against development of structures

Arellano et al., 2013
New/Recent Directions
Structures/Melt Down

- “No melt” ice cream based on addition of polyphenols
  – CJ Wicks
- Rheological properties of continuous phase
- Phase separation of protein/hydrocolloids
  – Dr. Jasmine Wu
No-Melt Ice Cream?

• Japanese “no-melt” ice cream
  – Strawberry extract
• After 2 hours, all the ice is melted, these ice creams just don’t collapse
  “no-collapse” ice cream
• Must be related to the structures
  – Fat globules, protein

“Polyphenol liquid has properties to make it difficult for water and oil to separate so that a popsicle containing it will be able to retain the original shape of the cream for a longer time than usual and be hard to melt”

Tomihisa Ota
Professor Emeritus of Pharmacy at Kanazawa University,
Co-Developer of Ice Cream

Ice Cream Melting

- Not all ice creams are created equal – or melt in the same way
- Drip-through test – slabs on mesh, measure drip through weight and height change

Which is better? That’s up to you and what the manufacturer wants
High Fat Destabilization
Minimal Collapse

- Ice crystals
- Free water
- Serum phase
- Fat/destabilized fat
- Air cells

t = 0 minutes
t = 60 minutes
t = 120 minutes

Objective 1
Do polyphenols affect partial coalescence of fat or is the primary mechanism protein mediated?

Tannic Acid
Microstructure
Gelation
Melting Rate
Complex Viscosity

Increase in complex viscosity and particle size as TA% increases

Mean Particle Size

• SDS releases fat crystals to disrupt partially-coalesced fat
  - but also breaks non-covalent bonds
• EDTA sequesters Ca
  - disrupts casein micelle structure

Looks like no partial coalescence, just protein-mediated aggregation
  - confirmed since skim milk showed no aggregates
Objective 3

Evaluate logical target PPs and/or extracts for further study in frozen dessert systems.

Experimental Design:

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>Fat %</th>
<th>Protein %</th>
<th>PP %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cream</td>
<td>10</td>
<td>2</td>
<td>0</td>
</tr>
<tr>
<td>Non-Fat Dry Milk</td>
<td>13</td>
<td>3.5</td>
<td>3</td>
</tr>
<tr>
<td>Milk Protein Concentrate (80%)</td>
<td>16</td>
<td>5</td>
<td>0.2%</td>
</tr>
<tr>
<td>Sugar</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tannic Acid</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Water</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mono and Diglycerides (0.12%)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Stabilizers (0.2%)</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Methods:

- Mix Preparation with polyphenol
- Particle Size Distribution
- Microscope Images
- pH of mix
- Overrun
- Rheology
- Melting Rate
- Ice Crystals

Mix and Ice Cream Preparation
Microscope Images

Control

2.5% TA

Drip Weight

LOW:
10% f & 2% p

MEDIUM:
13% f & 3.5% p

HIGH:
16% f & 5% p

2.5% Tannic Acid
Future Work

• Evaluate TA level on melt properties
  – Correlate to structure development through microscopy and rheology
• Evaluate various extracts and other delivery formats as developed from Objective 2
• Can extracts modulate melting properties of frozen desserts?
  – Non-dairy products?

Funding acknowledgment: USDA NIFA (WIS03038 GRANT 12905866)

Rheological Effects

• Previous work has shown that viscosity of the mix had the most important effect on melt-down
  – Overrun and partial coalescence were only important at the lowest level of stabilizer addition

Wu et al., 2019
Rheological Effects

• Phase 2. The effect of rheological properties on meltdown behavior of non-aerated frozen sucrose system
• Phase 3. The effect of rheological properties on meltdown behavior of aerated frozen sucrose system
• Phase 4. The effect of protein-polysaccharides interaction on meltdown behavior of aerated frozen sucrose system

Wu J., Understanding the meltdown behavior of frozen dessert: from ice cream to model system, PhD Dissertation, UW-Madison (2023)

Phase 2. Rheology on non-aerated system

**Hypothesis:** The effect of rheological properties on melting and dripping is caused by either apparent viscosity or shear-thinning behavior in the non-aerated frozen sucrose system.

- Apparent mix viscosity (at 5 s⁻¹ shear rate)
- Shear-thinning behavior
  - Flow rate index (power law model)  \[ \sigma = \eta \dot{\gamma}^n \]

**Experimental design**

<table>
<thead>
<tr>
<th>Same flow index (0.74)</th>
<th>Apparent viscosity at 5 s⁻¹</th>
<th>Same viscosity at 5 s⁻¹ (0.20)</th>
<th>Flow index</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.22% guar gum (GG)</td>
<td>0.10±0.00ᵃ</td>
<td>0.11% xanthan</td>
<td>0.47±0.01ᵃ</td>
</tr>
<tr>
<td>0.3% locust bean gum (LBG)</td>
<td>0.15±0.00ᵇ</td>
<td>0.28% guar gum (GG)</td>
<td>0.66±0.00ᵇ</td>
</tr>
<tr>
<td>0.3% sodium alginate (SA)</td>
<td>0.26±0.00ᶜ</td>
<td>0.25% sodium alginate (SA)</td>
<td>0.76±0.00ᶜ</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.7% pectin</td>
<td>0.86±0.01ᵈ</td>
</tr>
</tbody>
</table>
Phase 2. Rheology on non-aerated system

**Surface tension**

- Polysaccharides reduce surface tension
- The surface tension is related to the natures of polysaccharide
- Surface-active property results in air incorporation

<table>
<thead>
<tr>
<th>Same flow rate index*</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>0.22% GG</td>
<td>58.4 ± 0.8b</td>
</tr>
<tr>
<td>0.3% LBG</td>
<td>54.0 ± 0.6c</td>
</tr>
<tr>
<td>0.3% SA</td>
<td>63.2 ± 0.9a</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Same apparent viscosity*</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>0.11% XAN</td>
<td>69.0 ± 1.0a</td>
</tr>
<tr>
<td>0.28% GG</td>
<td>56.4 ± 1.0c</td>
</tr>
<tr>
<td>0.25% SA</td>
<td>64.9 ± 1.0b</td>
</tr>
<tr>
<td>0.7% PEC</td>
<td>56.8 ± 1.2c</td>
</tr>
</tbody>
</table>

*Filled: same apparent viscosity; hollow: same flow rate index

**Overrun (%)**

<table>
<thead>
<tr>
<th>Same flow rate index*</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>0.22% GG</td>
<td>17.5 ± 1.4a</td>
</tr>
<tr>
<td>0.3% LBG</td>
<td>13.7 ± 1.0b</td>
</tr>
<tr>
<td>0.3% SA</td>
<td>11.9 ± 2.9b</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Same apparent viscosity*</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>0.11% XAN</td>
<td>12.4 ± 0.7b</td>
</tr>
<tr>
<td>0.28% GG</td>
<td>16.1 ± 0.8a</td>
</tr>
<tr>
<td>0.25% SA</td>
<td>9.2 ± 1.5c</td>
</tr>
<tr>
<td>0.7% PEC</td>
<td>9.8 ± 1.4c</td>
</tr>
</tbody>
</table>

Phase 2. Rheology on non-aerated system

**Meltdown**

- The nature of polysaccharide affected the melting rate.
- Anionic polysaccharide showed a faster melting rate than galactomannan

**Key conclusions:**

- No significant difference was found in induction time
- The nature of polysaccharide affected the melting rate.
- Anionic polysaccharide showed a faster melting rate than galactomannan
**Phase 3. Rheology on aerated system**

**Hypothesis:** The effect of rheological properties on melting and dripping is caused by either apparent viscosity or shear-thinning behavior in the aerated frozen sucrose system.

<table>
<thead>
<tr>
<th>Polysorbate 80</th>
<th>Overrun</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.04%</td>
<td>45%</td>
</tr>
<tr>
<td>0.15%</td>
<td>75%</td>
</tr>
</tbody>
</table>

**Experimental design**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Target overrun</th>
<th>Flow rate index</th>
<th>Apparent viscosity at 5 s⁻¹ shear rate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Same flow rate index</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.014% xanthan</td>
<td>45%</td>
<td>0.76 ± 0.01</td>
<td>0.02 ± 0.00</td>
</tr>
<tr>
<td>0.22% guar gum</td>
<td>75%</td>
<td>0.74 ± 0.00</td>
<td>0.10 ± 0.00</td>
</tr>
<tr>
<td>Same apparent viscosity</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.11% xanthan</td>
<td>45%</td>
<td>0.47 ± 0.00</td>
<td>0.20 ± 0.00</td>
</tr>
<tr>
<td>0.28% guar gum</td>
<td>75%</td>
<td>0.69 ± 0.00</td>
<td>0.19 ± 0.00</td>
</tr>
</tbody>
</table>

**Phase 3. Rheology on aerated system**

**Meltdown**

- A strong correlation was found between apparent viscosity and induction time, but not between the flow rate index and induction time.
- The effect of overrun was only seen in xanthan, where increase in overrun decreased melting rate.
Phase 4. Phase separation on meltdown

**Hypothesis:** The protein-polysaccharide phase separation in serum results in a slow meltdown behavior due to the interaction between two immiscible phases.

**Experimental design**

<table>
<thead>
<tr>
<th>Protein (NFDM*)</th>
<th>Locust bean gum</th>
<th>Guar gum</th>
<th>κ-carrageenan</th>
</tr>
</thead>
<tbody>
<tr>
<td>4%</td>
<td>0.05%</td>
<td>0.05%</td>
<td>0%</td>
</tr>
<tr>
<td>6%</td>
<td>0.15%</td>
<td>0.15%</td>
<td>0.015%</td>
</tr>
<tr>
<td>8%</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*NFDM: non-fat dry milk

**Phase separation**

- CLSM provided additional information on phase separation
- Freezing prevented phase separation on LBG system
Phase 4. Phase separation on meltdown

**Meltdown behavior**

NFDM+LBG/GG

Guar

Locust bean

Melted samples observed under microscope.

Key conclusions:
- Correlation between rheology and induction time only seen in LBG.
- Protein affected meltdown by achieving different overrun
- The more phase separation in the drip-through solution, the slower the melting rate (carrageenan+GG).
**Conclusions**

- Connection between melt-down and rheological properties still remains unclear
- Locust bean gum in general slows down the meltdown process through cryo-gel formation
- Freezing prevented phase separation in the locust bean gum system

**Future recommendations**

- The types of polysaccharide influence meltdown in the ice cream system
- Local viscosity vs. bulk viscosity in phase separation system
- The structure in the serum phase changes during freezing-melting process

Ice cream is complex and there is still so much we don’t understand

Questions?

*Thanks to all the students who have contributed to these studies*