## ICE CREAM MICROSTRUCTURE



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## 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



## Ice Cream Processing



Ice

- nucleation
- growth

Air

- incorporation
- breakdown

Lipid

- growth
- partial coalescence

Ice

- growth

Air

- coalescence
$\underline{\text { Lipid }}$
- growth

Ice

- melting
- growth
- ripening

Air

- coalescence

Lactose

- crystallization


## 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



## 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


Warren \& Hartel (2017)


0:0 ER, $5.9 \%$


90:10 ER, 28.3\%

100:0 ER, 19.6\%


## 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




## 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



## 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



## Measuring RTD in a Scraped Surface Freezer



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

## Dasher Speed




## Overrun



## Throughput Rate





## 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

https://youtu.be/GFE91TTJfN8
- 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


After 30 mins


## 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



## High Fat Destabilization Minimal Collapse



## Objective 1

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



## Complex Viscosity



Wicks et al., 2023

## Mean Particle Size

## Dispersion Method



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


## Objective 3

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

Experimental Design:

| Fat \% | 10 | 13 | 16 |
| :---: | :---: | :---: | :---: |
| Protein \% | 2 | 3.5 | 5 |
| PP \% | 0 | 3 |  |

## Methods:

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


## Mix and Ice Cream Preparation

| Ingredient |
| :---: |
| Cream |
| Non-Fat Dry Milk |
| Milk Protein Concentrate $(80 \%)$ |
| Sugar |
| Tannic Acid |
| Water |
| Mono and Diglycerides $(0.12 \%)$ |
| Stabilizers $(0.2 \%)$ |



## Microscope Images



## Drip Weight



## 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?

https://youtu.be/sA-lc6ZnWLo


## 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



## 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 $5 \mathrm{~s}^{-1}$ shear rate)
$>$ Shear-thinning behavior

- Flow rate index (power law model) $\boldsymbol{\sigma}=\boldsymbol{\eta} \dot{\boldsymbol{\gamma}}^{\boldsymbol{n}}$


## Experimental design

| Same flow index (0.74) | Apparent viscosity at $5 \mathrm{~s}^{-1}$ | Same viscosity at $5 \mathrm{~s}^{-1}$ (0.20) | Flow index |
| :---: | :---: | :---: | :---: |
| $\begin{aligned} & 0.22 \% \text { guar gum } \\ & \text { (GG) } \end{aligned}$ | $0.10 \pm 0.00^{\text {a }}$ | $0.11 \%$ xanthan | $0.47 \pm 0.01^{\text {a }}$ |
|  |  | 0.28\% guar gum (GG) | $0.66 \pm 0.00^{\text {b }}$ |
| $0.3 \%$ locust bean gum (LBG) | $0.15 \pm 0.00^{\text {b }}$ |  |  |
|  |  | $0.25 \%$ sodium alginate | $0.76 \pm 0.00^{\text {c }}$ |
| $0.3 \%$ sodium alginate (SA) | $0.26 \pm 0.00^{\text {c }}$ | (SA) | $0.76 \pm 0.00^{\text {c }}$ |
|  |  | 0.7\% pectin | $0.86 \pm 0.01^{\text {d }}$ |

## Phase 2. Rheology on non-aerated system

## Surface tension


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

| Same flow rate index* |  |
| :---: | :---: |
| $0.22 \% \mathrm{GG}$ | $58.4 \pm 0.8^{\mathrm{b}}$ |
| $0.3 \% \mathrm{LBG}$ | $54.0 \pm 0.6^{\mathrm{c}}$ |
| $0.3 \%$ SA | $63.2 \pm 0.9^{\mathrm{a}}$ |
| Same apparent viscosity* |  |
| $0.11 \%$ XAN | $69.0 \pm 1.0^{\mathrm{a}}$ |
| $0.28 \% \mathrm{GG}$ | $56.4 \pm 1.0^{\mathrm{c}}$ |
| $0.25 \%$ SA | $64.9 \pm 1.0^{\mathrm{b}}$ |
| $0.7 \%$ PEC | $56.8 \pm 1.2^{\mathrm{c}}$ |

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


## Overrun (\%)

| Same flow rate index* |  |
| :---: | :---: |
| $0.22 \%$ GG | $17.5 \pm 1.4^{\mathrm{a}}$ |
| $0.3 \% \mathrm{LBG}$ | $13.7 \pm 1.0^{\mathrm{b}}$ |
| $0.3 \%$ SA | $11.9 \pm 2.9^{\mathrm{b}}$ |
| Same apparent viscosity* |  |
| $0.11 \%$ XAN | $12.4 \pm 0.7^{\mathrm{b}}$ |
| $0.28 \%$ GG | $16.1 \pm 0.8^{\mathrm{a}}$ |
| $0.25 \%$ SA | $9.2 \pm 1.5^{\mathrm{c}}$ |
| $0.7 \%$ PEC | $9.8 \pm 1.4^{\mathrm{c}}$ |

## Phase 2. Rheology on non-aerated system

## Meltdown




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.

| Polysorbate 80 |  | Overrun |
| :---: | :---: | :---: |
| $0.04 \%$ |  | $45 \%$ |
| $0.15 \%$ |  | $75 \%$ |
|  |  |  |

## Experimental design

|  | Sample | Target overrun | Flow rate index | Apparent viscosity at $5 \mathrm{~s}^{-1}$ shear rate |
| :---: | :---: | :---: | :---: | :---: |
| Same flow rate index | 0.014\% xanthan | 45\% | $0.76 \pm 0.01$ | $0.02 \pm 0.00$ |
|  |  | 75\% |  |  |
|  | 0.22\% guar gum | 45\% | $0.74 \pm 0.00$ | $0.10 \pm 0.00$ |
|  |  | 75\% |  |  |
| Same apparent viscosity | 0.11\% xanthan | 45\% | $0.47 \pm 0.00$ | $0.20 \pm 0.00$ |
|  |  | 75\% |  |  |
|  | 0.28\% guar gum | 45\% | $0.69 \pm 0.00$ | $0.19 \pm 0.00$ |
|  |  | 75\% |  |  |

## 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.

| Background |  |  | I Experimental design |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  |  |  |  |  |  |
| me |  |  | $\begin{gathered} \text { Protein } \\ \left(\mathrm{NFDM}^{*}\right. \end{gathered}$ | $\begin{array}{\|c} \text { Locust } \\ \text { bean } \\ \text { gum } \end{array}$ | $\begin{aligned} & \text { Guar } \\ & \text { gum } \end{aligned}$ | $\begin{gathered} \mathrm{K}- \\ \text { carrageenan } \end{gathered}$ |
| - | n |  | 4\% | 0.05\% | 0.05\% | 0\% |
| teil |  |  | 6\% | 0.15\% | 0.15\% | 0.015\% |
| (20) |  |  | 8\% |  |  |  |
| $\begin{array}{\|c} \text { Phase } \\ \text { separation } \end{array}$ |  | No phase separation | *NFDM: non-fat dry milk |  |  |  |

## Phase 4. Phase separation on meltdown

## Phase separation

- CLSM provided additional information on phase separation
- Freezing prevented phase separation on LBG system



## Phase 4. Phase separation on meltdown

## Meltdown



6\% protein | Mix | $0.05 \% \mathrm{LBG}$ |
| :--- | :--- |



6\% protein


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 dripthrough solution, the slower the melting rate (carrageenan+GG).



## Phase 4. Phase separation on meltdown

## Meltdown behavior

NFDM + LBG/GG



Locust bean



## 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


