

# Freezing, Melting and Shrinkage of Ice Cream

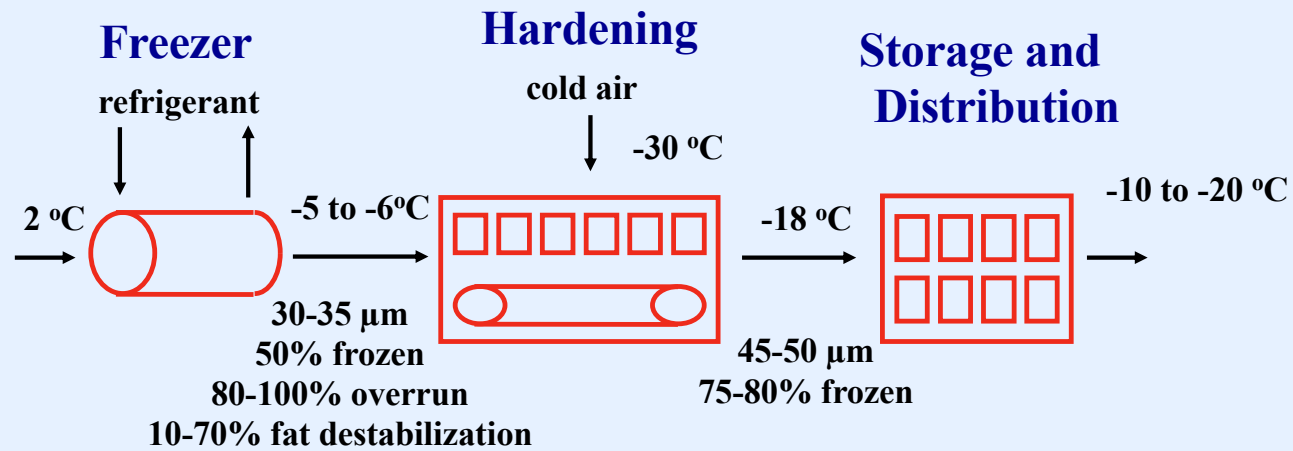
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**L Gallagher**  
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University of Wisconsin  
Madison, WI USA



Funding: USDA, NDC



# Ice Cream Processing



## Ice

- nucleation
- growth

## Air

- incorporation
- breakdown

## Lipid

- growth
- partial coalescence

## Ice

- growth

## Air

- coalescence

## Lipid

- growth

## Ice

- melting
- growth
- ripening

## Air

- coalescence

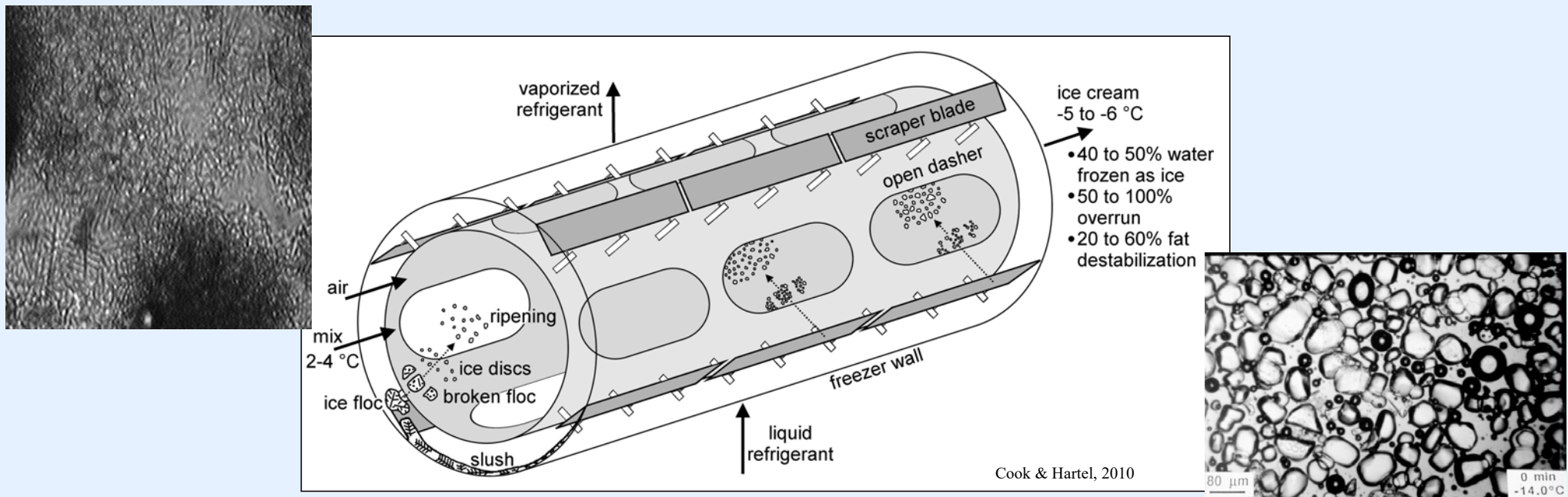
## Lactose

- crystallization

# Scraped Surface Freezer (SSF)

## Development of Ice Phase

- 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



# Experimental Design

**How long after start-up does it take the freezing process to stabilize?**

## Theoretical Residence Times (s)

Overrun		50% OR	75% OR			100% OR
Throughput Rate		300 L/h	200 L/h	300 L/h	400 L/h	300 L/h
Dasher Assembly	Solid	81	141	94	71	108
	Multi + Solid	104	181	121	91	138
	Standard + Solid	110	193	129	96	147
	Multi + Wing	160	281	187	140	214
	Standard + Wing	167	291	194	146	222

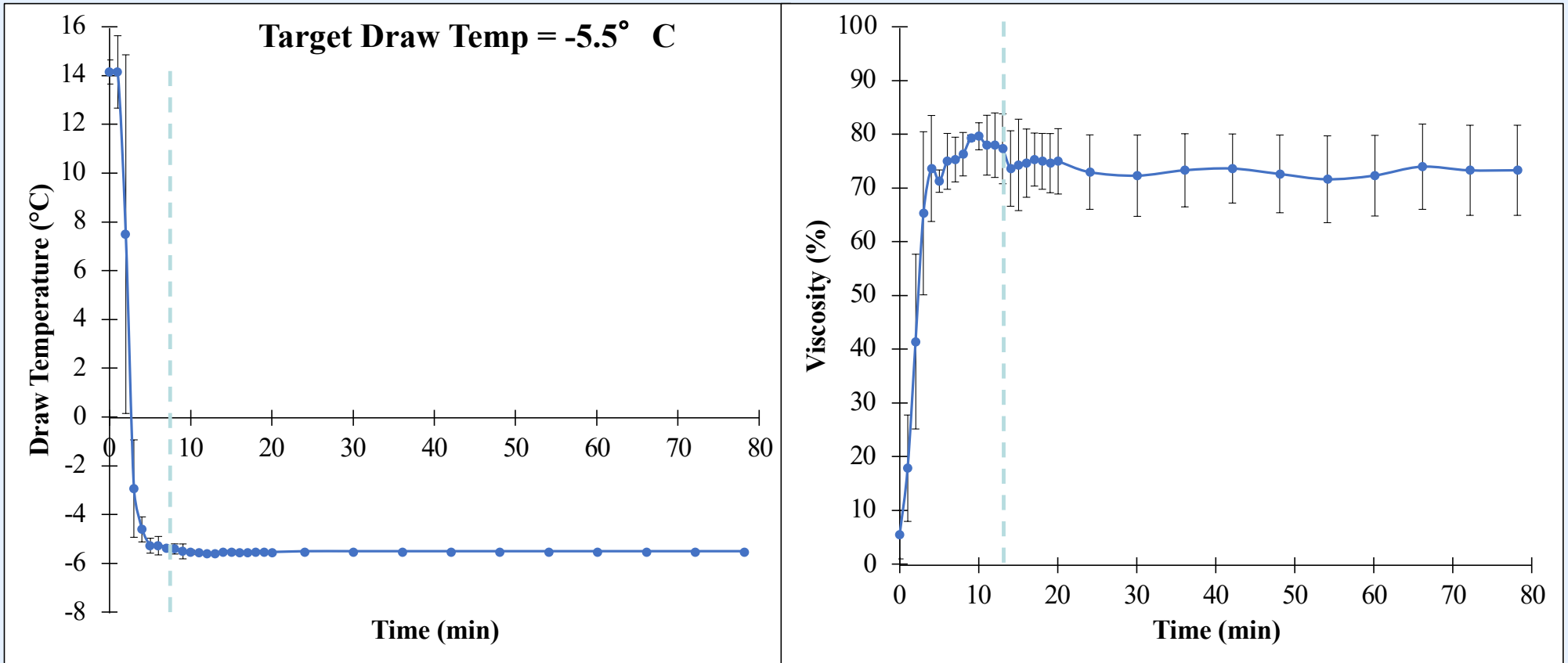
## Sampling Frequency

- Every 1 min for the first 20 min
  - Hardened only
- Every 6 min for 78 min
  - Draw and hardened

## Measurements

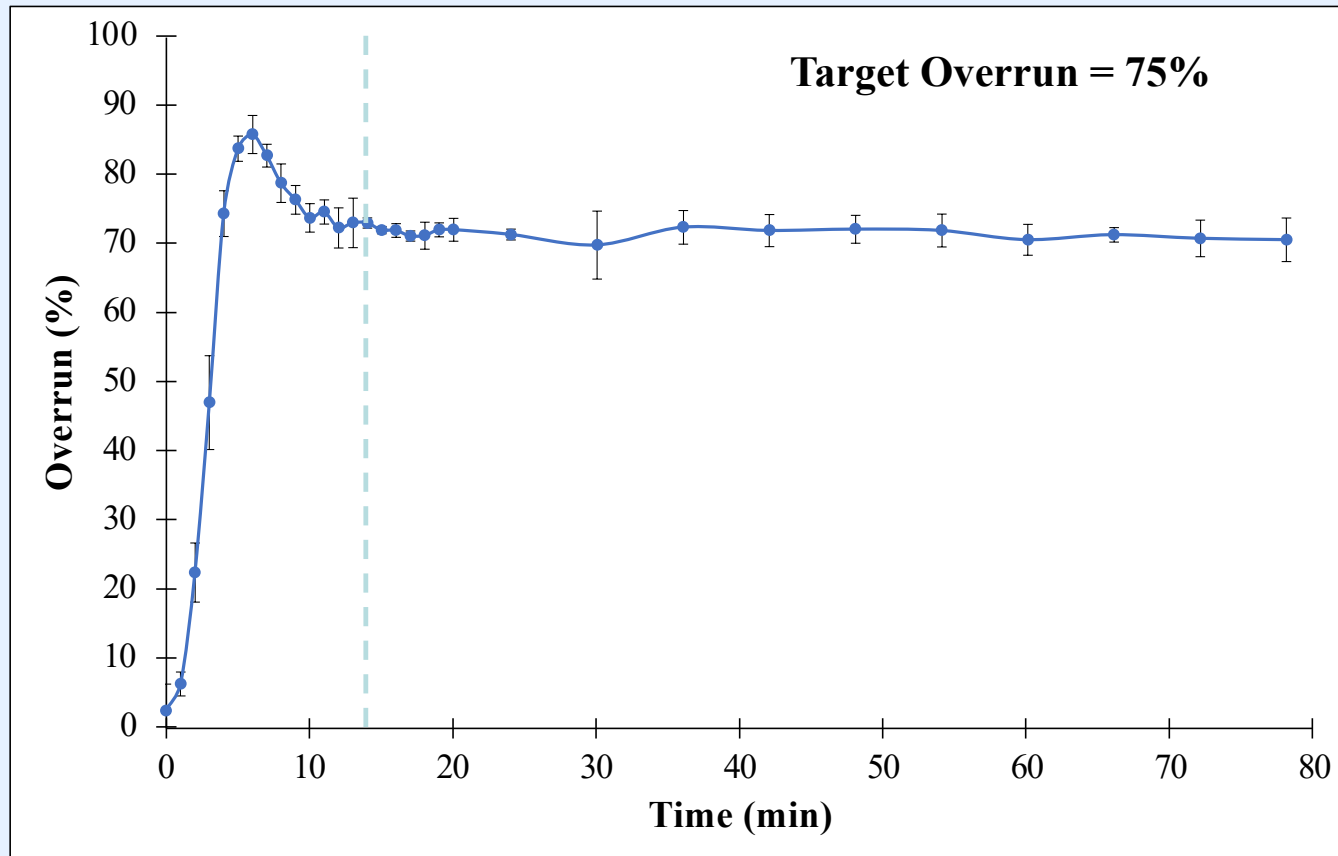
- Draw Temperature
- “Viscosity”
- Overrun
- Microstructure
  - Ice
  - Air
  - Fat

# Processing Parameters after Start-up

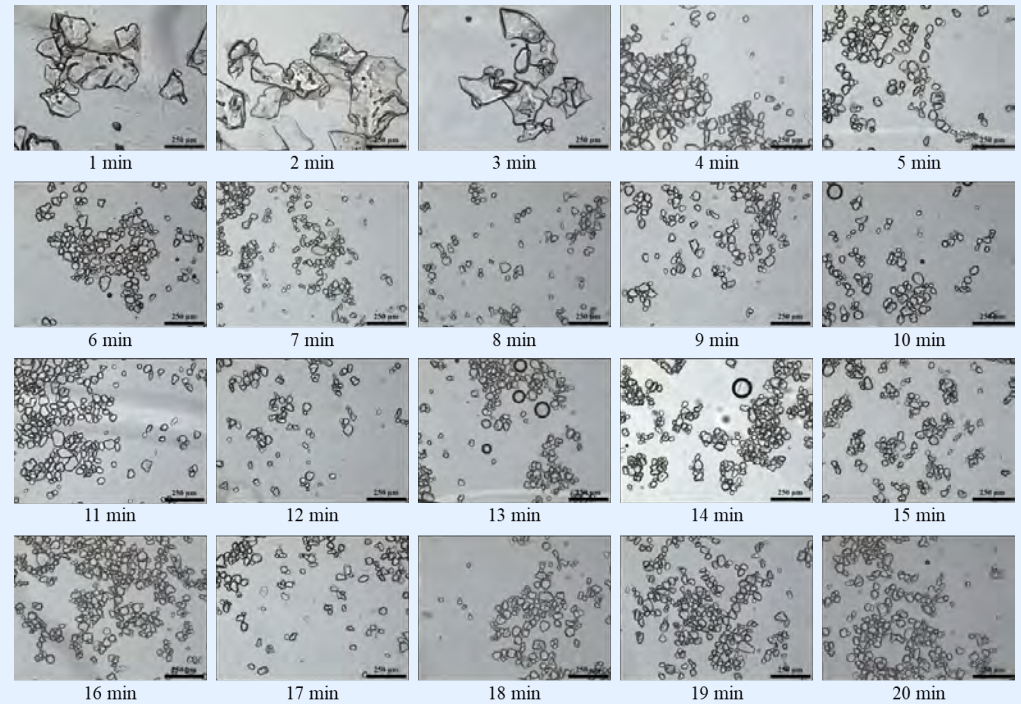
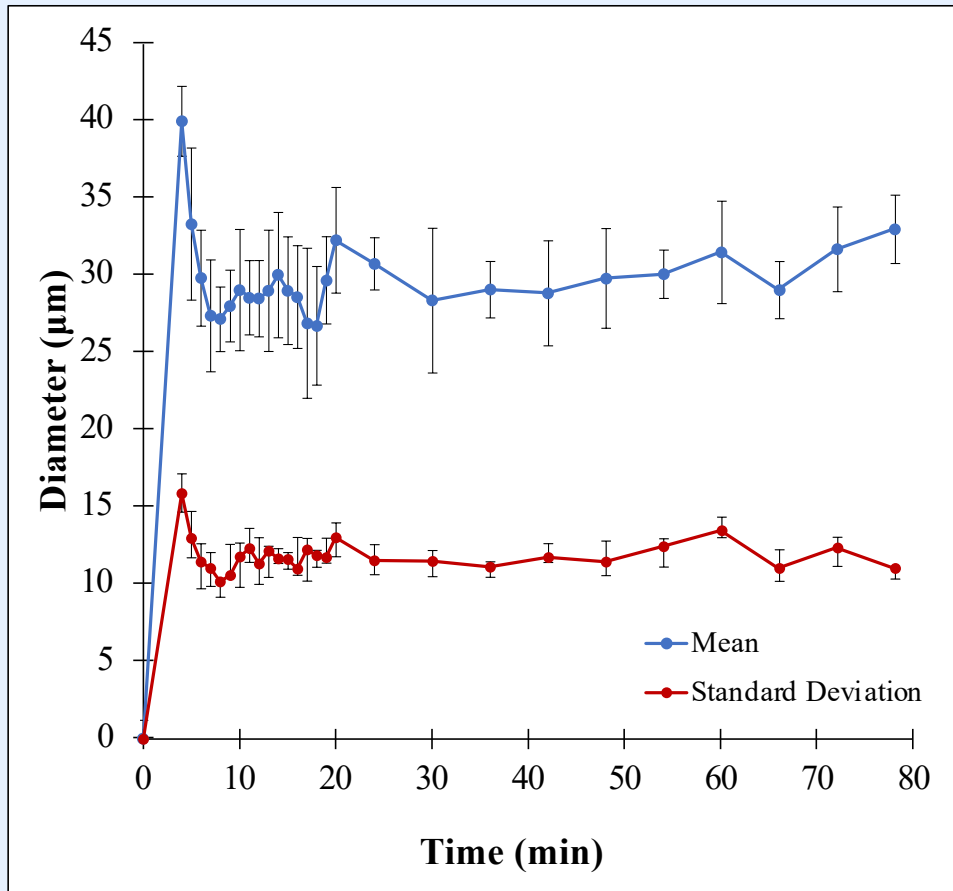


“Viscosity” = torque on dasher motor as the percentage of its total capacity

# Processing Parameters after Start-up

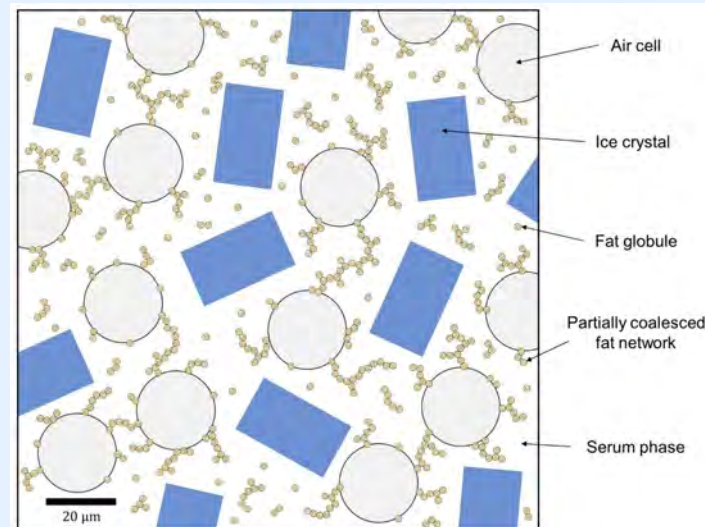


# Microstructural Attributes after Start-up



# 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



# “No-Melt” Ice Cream

- Periodical uproar about ice cream that doesn't "melt"
- Of course it melts, it just doesn't collapse because of the structures



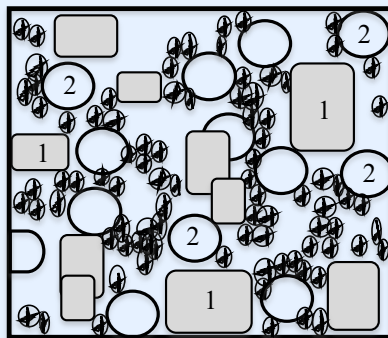
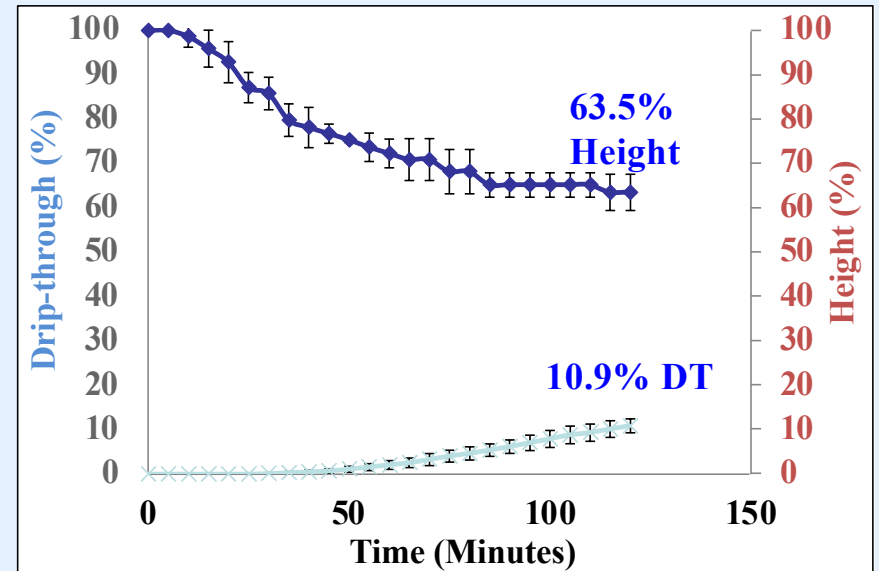
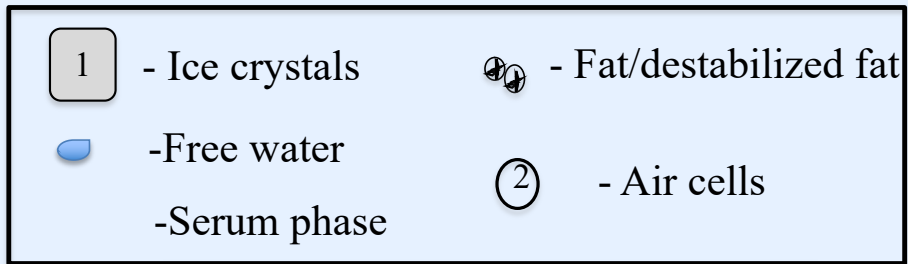
## Ice Cream Melting

- 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

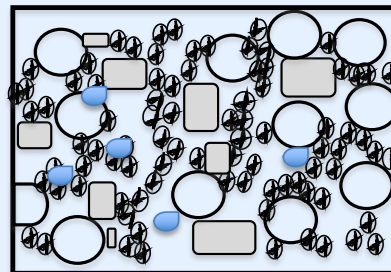


*Which is better? That's up to you  
and what the manufacturer wants*

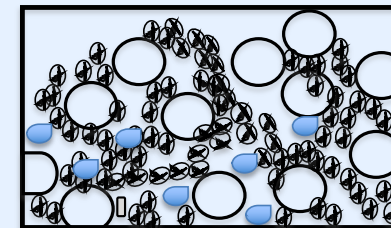
# High Fat Destabilization Minimal Collapse



t = 0 minutes



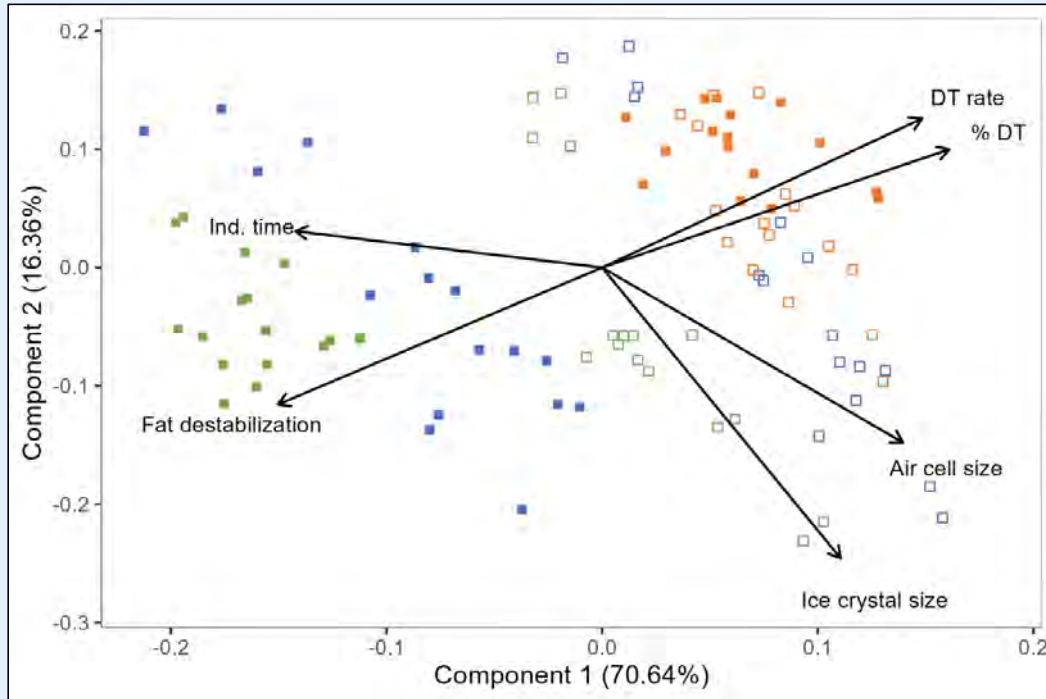
t = 60 minutes



t = 120 minutes



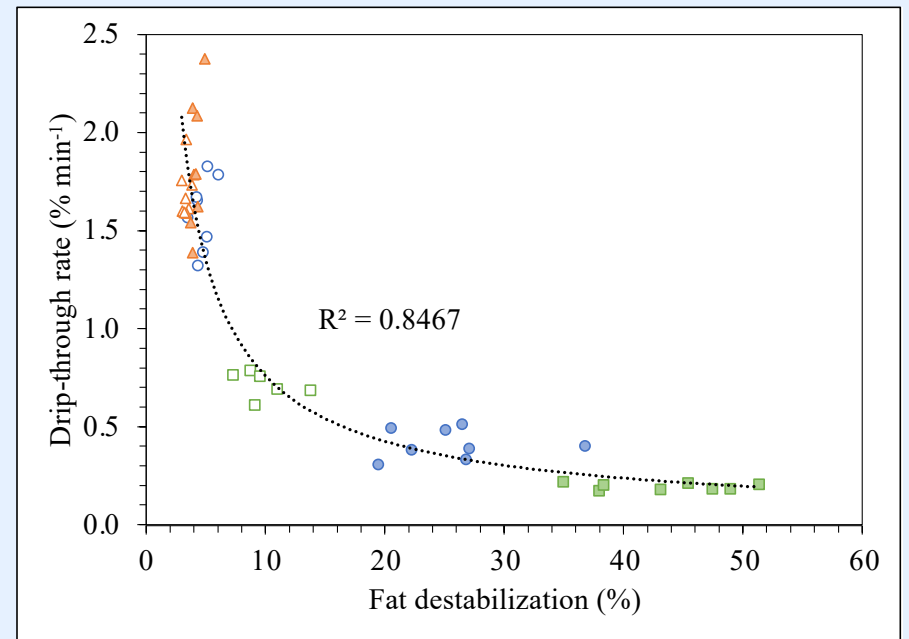
# Structures and Melt-Down



MPC (■) NaCN (■) WPI (■)

VanWees, 2024

*Fat destabilization plays a critical role in how ice cream melts down*



$$DT \text{ rate} = 5.12(FD)^{-0.828}$$

MPC (○●) NaCN (△▲) WPI (■)

# No-Melt Ice Cream?

- Japanese "no-melt" ice cream
  - Strawberry extract added
  - (juice concentrate, citric acid & pectin?)
- 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

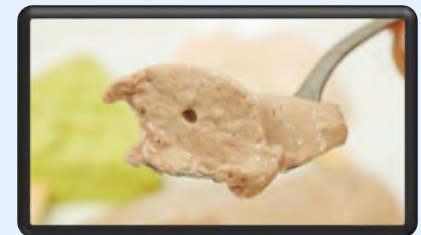
*"Polyphenol liquid has properties to **make it difficult for water and oil to separate** so that a popsicle containing it will be able to retain the original shape of the cream for a longer time than usual and be hard to melt"*

**Tomihisa Ota**

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



After 30 mins



# Bringing delight by investigating a no-melt ice cream

June 10, 2024 | By [Elise Mahon](#)



<https://www.youtube.com/watch?v=4fRVqG96vFM&t=2s>

Evaluate tannic acid in frozen dessert systems with different fat/protein content.

### Ice cream formulations

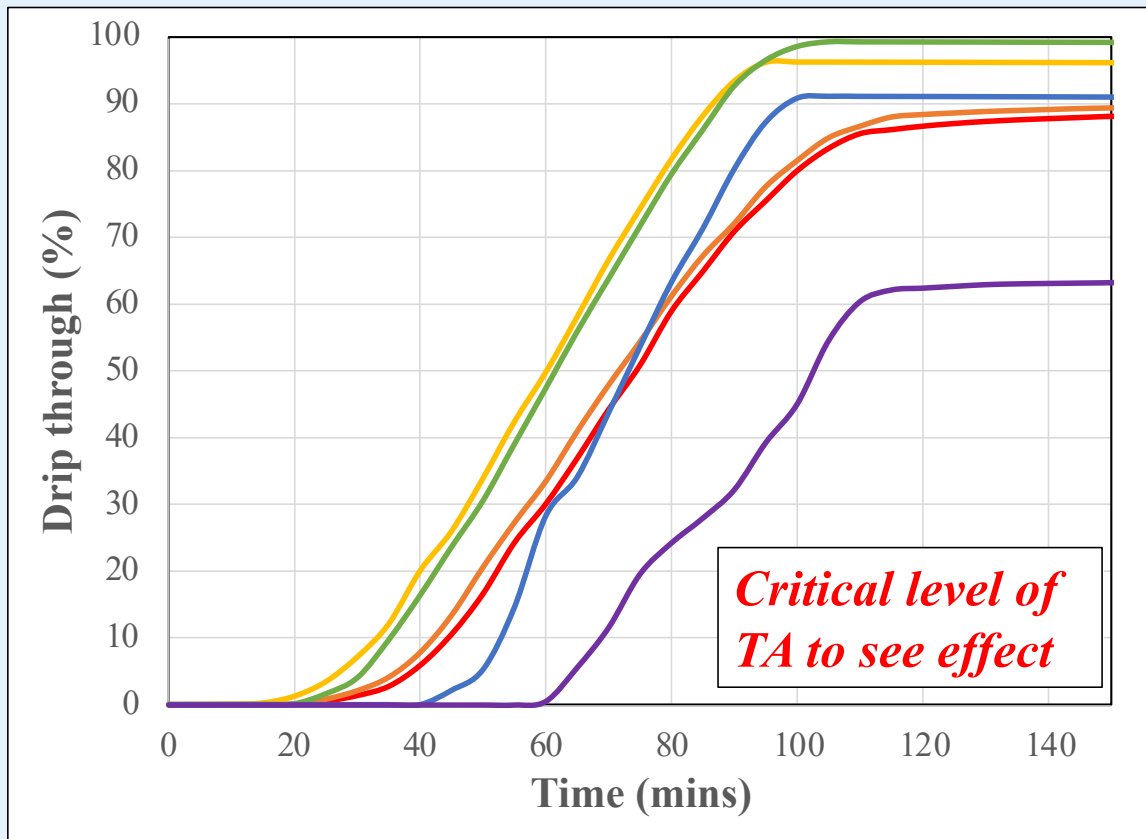
	fat (%)	protein (%)	TA (%)
Base	12	3	0
	12	3	0.5
	12	3	1
	12	3	1.5
	12	3	2
	12	3	2.5
Higher Protein	12	5	0
	12	5	2.5
Higher Fat	15	3	0
	15	3	2.5

### Methods:

- Mix Preparation with polyphenol
- Batch freezing
- Fat globule Size Distribution
- Microscope Images
- pH of mix
- Overrun
- Rheology
- Melting Rate
- Ice Recrystallization

# Melting Profiles

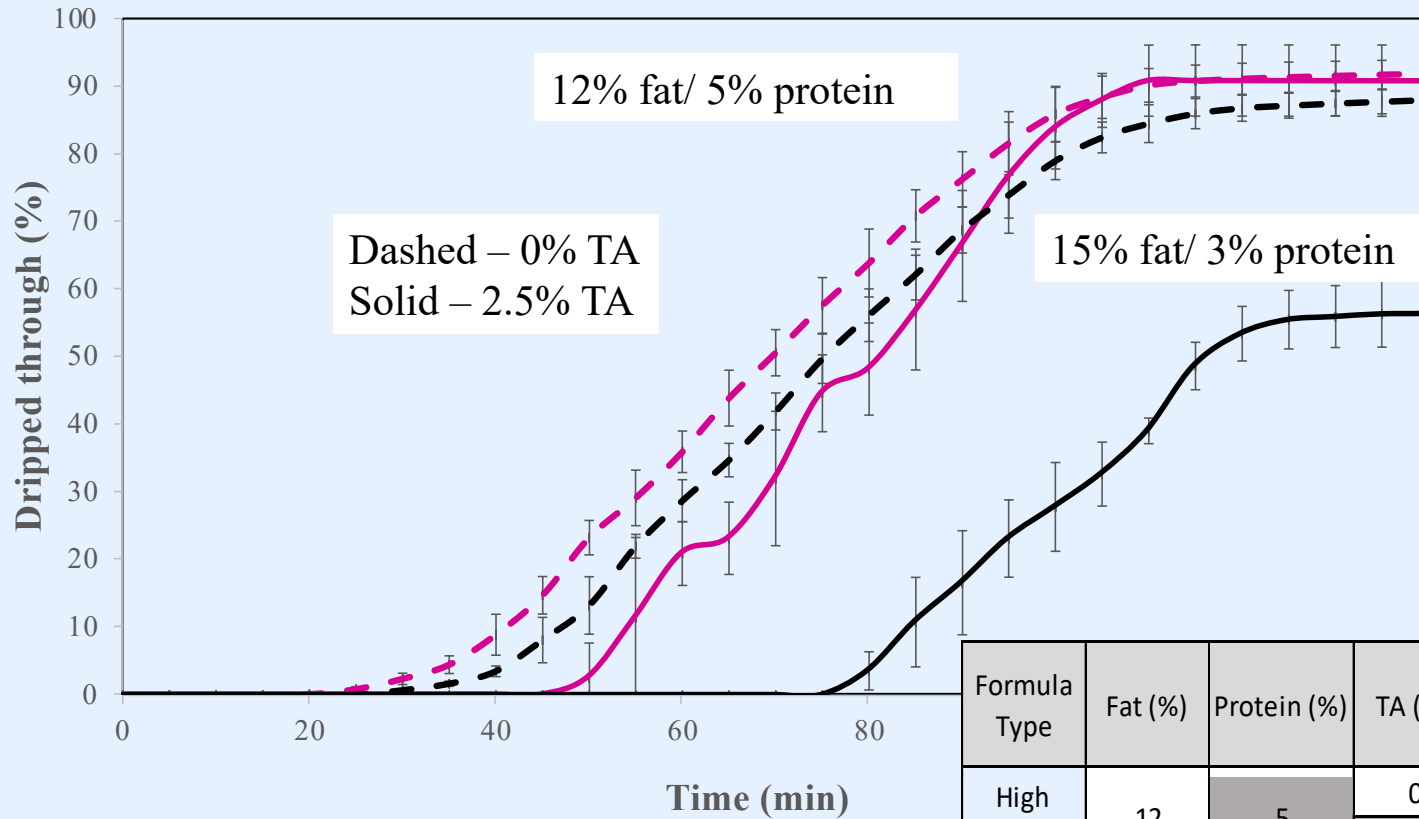
for Base (12% fat / 3% protein) ice creams with increasing TA%



TA (%)	Melting Rate (%/min)	Residual (%)
0	1.29 <sup>bc</sup>	10.9 <sup>b</sup>
0.5	1.38 <sup>ab</sup>	9.8 <sup>b</sup>
1	1.55 <sup>ab</sup>	3.7 <sup>b</sup>
1.5	1.55 <sup>ab</sup>	0.7 <sup>b</sup>
2	1.75 <sup>a</sup>	8.8 <sup>b</sup>
2.5	0.72 <sup>d</sup>	36.2 <sup>a</sup>



# Melting Profiles



*Fat content seems to be more important factor to melt-down than protein level*

Formula Type	Fat (%)	Protein (%)	TA (%)	Melting Rate (%/min)	Induction Time (min)	Residual Weight on Screen (%)
High Protein	12	5	0	1.43 ± 0.01 <sup>ab</sup>	18 ± 0.0 <sup>c</sup>	7.4 ± 1.4 <sup>b</sup>
			2.5	1.74 ± 0.07 <sup>a</sup>	51.7 ± 2.5 <sup>b</sup>	9.2 ± 2.9 <sup>b</sup>
High Fat	15	3	0	1.39 ± 0.05 <sup>ab</sup>	25.8 ± 0.0 <sup>c</sup>	10.5 ± 0.6 <sup>b</sup>
			2.5	0.91 ± 0.15 <sup>cd</sup>	79.2 ± 1.7 <sup>a</sup>	42.9 ± 4.0 <sup>a</sup>

# Tannic Acid in IC Mix: Microscope Images

**BASE:**

12% fat & 3% protein

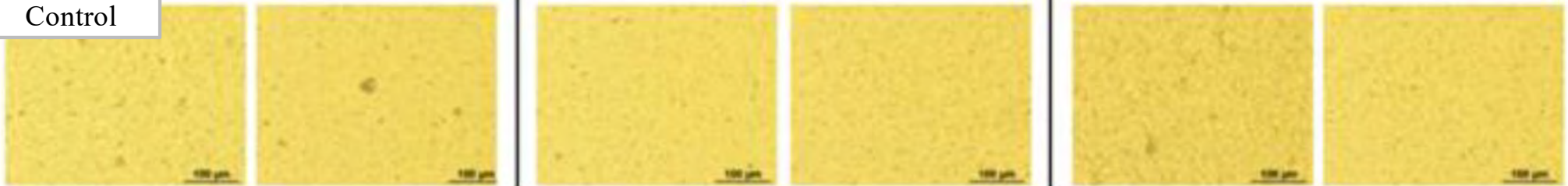
**HIGH PROTEIN:**

12% fat & 5% protein

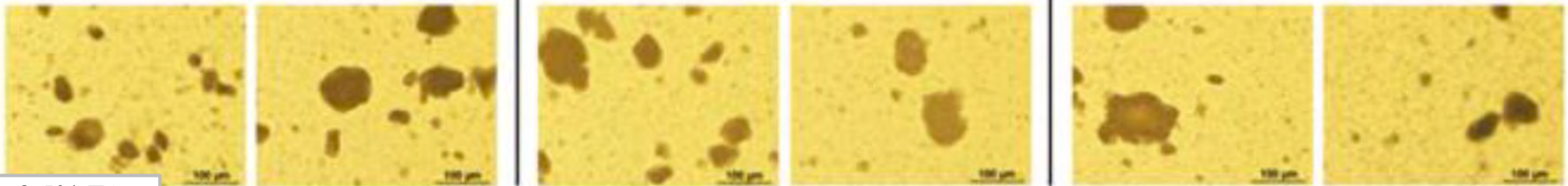
**HIGH FAT**

15% fat & 3% protein

Control

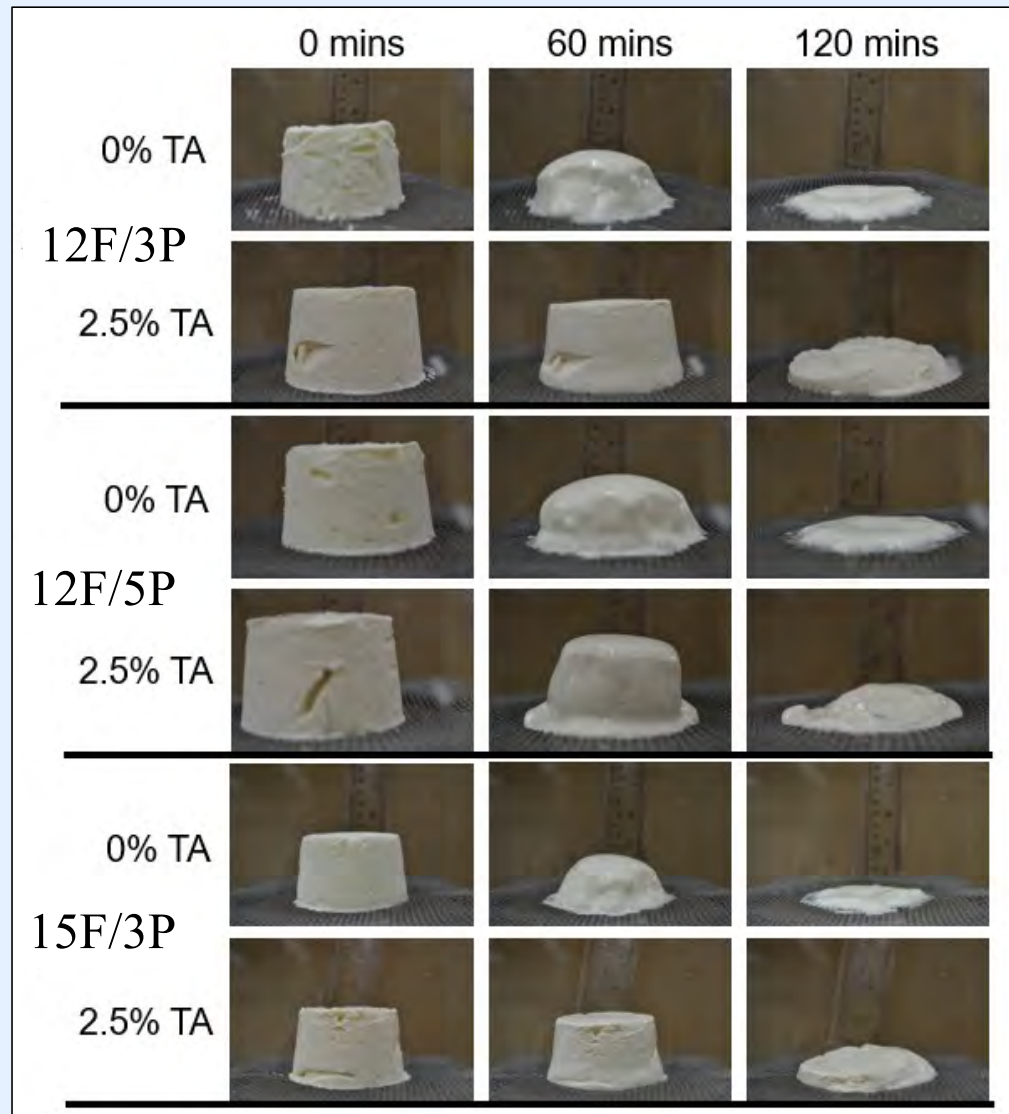


2.5% TA



# Melting Ice Cream

- None are really what could be called “no melt or collapse” ice creams
- Some effect of tannic acid at 2.5%, but not complete stopping of melt-down



## Ice Recrystallization

- TA inhibits ice recrystallization in storage
  - Not clear how the aggregated structures influence ice crystal growth?

Formula Type	TA (%)	Week 0	Week 2	Week 4
Standard	0	36.4±3.7 <sup>DEFGH</sup>	53.2±5.7 <sup>ABCDE</sup>	67.9±9.4 <sup>A</sup>
	0.5	31.6±0.0 <sup>FGH</sup>	51.7±0.0 <sup>ABCDEF</sup>	58.3±0.0 <sup>AB</sup>
	1.5	33.8±4.9 <sup>EFGH</sup>	42.9±1.4 <sup>BCDEFGH</sup>	52.0±2.1 <sup>ABCDEF</sup>
	2.5	29.8±2.2 <sup>H</sup>	41.1±2.5 <sup>BCDEFGH</sup>	42.7±0.7 <sup>BCDEFGH</sup>
Higher Protein	0	33.9±0.9 <sup>EFGH</sup>	56.4±4.5 <sup>ABCD</sup>	71.1±7.0 <sup>A</sup>
	2.5	31.3±3.1 <sup>FGH</sup>	32.7±0.6 <sup>EFGH</sup>	36.8±3.3 <sup>CDEFGH</sup>
Higher Fat	0	30.6±3.0 <sup>GH</sup>	57.9±4.5 <sup>ABC</sup>	67.6±3.3 <sup>A</sup>
	2.5	32.5±4.0 <sup>EFGH</sup>	34.8±0.2 <sup>EFGH</sup>	38.7±0.7 <sup>BCDEFGH</sup>

# Phenolic Extracts

- Polyphenol extracts (high phenolic %) shown to decrease melting rate in previous studies
- Could these extracts replace stabilizers in ice cream?

Each extract has at least 85% Polyphenols

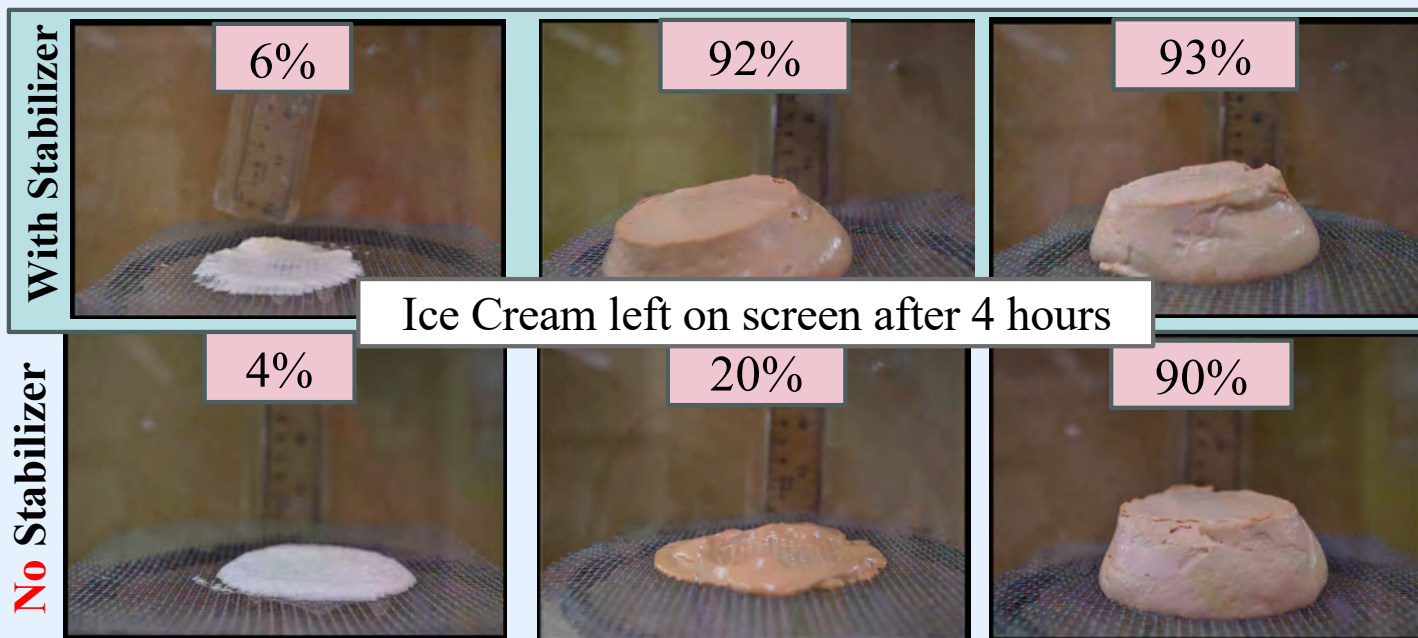
	<b>With Stabilizer</b>	<b>Without Stabilizer</b>
<b>Control</b>	Control + Stabilizer	Control + No Stabilizer
<b>Grapeseed</b>	Grapeseed + Stabilizer	Grapeseed + No Stabilizer
<b>Green Tea</b>	Green Tea + Stabilizer	Green Tea + No Stabilizer

Stabilizer blend  
locust bean gum,  
guar gum, and  
carrageenan

## Control

## Grapeseed

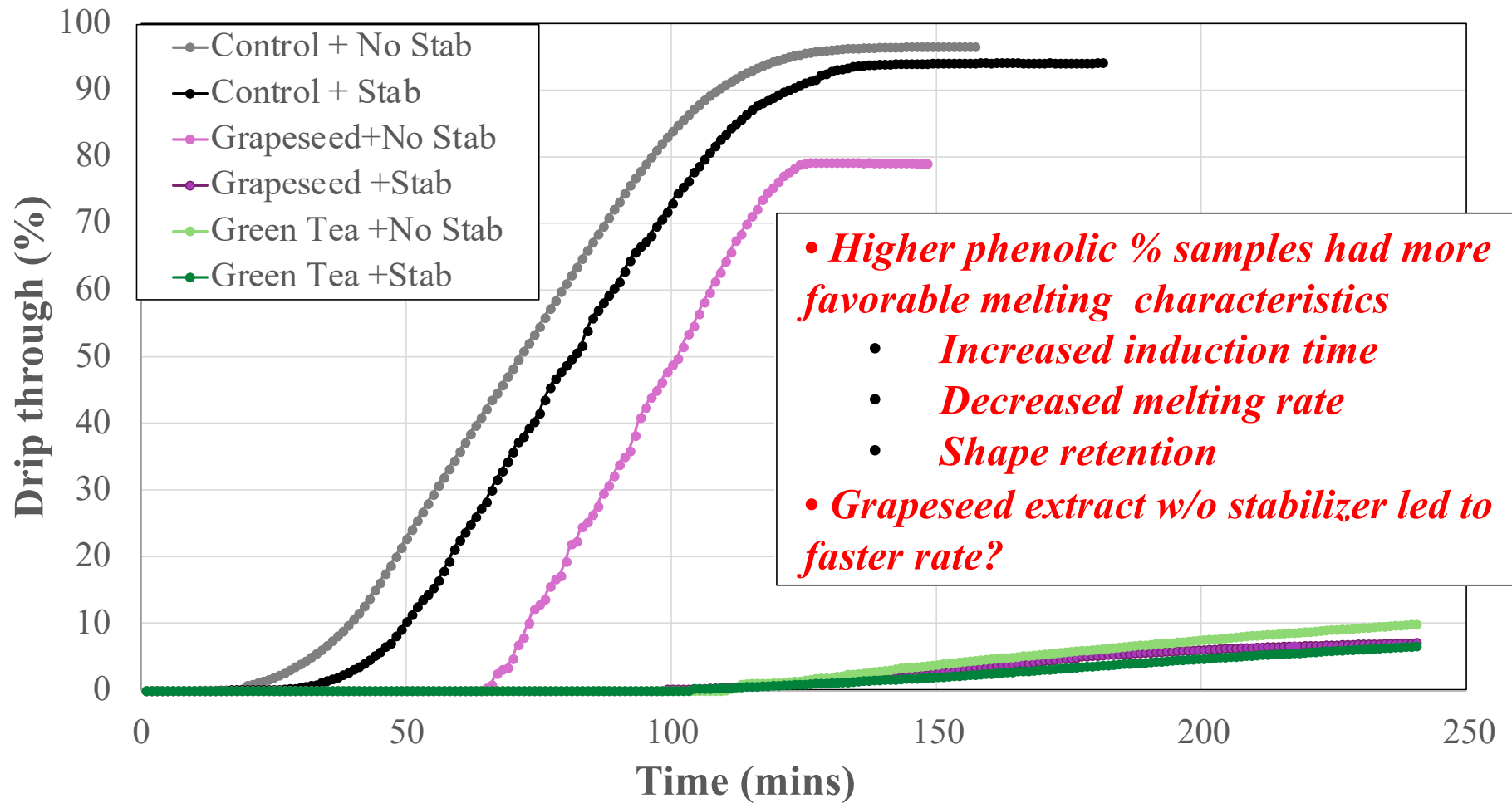
## Green Tea



Extract	Total polyphenols in extract (%)	Total PAC in extract (%)	Total polyphenols in ice cream (%)	PAC content in ice cream (%)
green tea extract	97.6 ± 8.0	38.7 ± 7.4	2.9	1.2
grape seed extract	80.7 ± 7.0	13.8 ± 0.3	2.4	0.4

*Type of PP different between samples*

# Melting Profiles



## Ice Recrystallization

- Both extracts inhibit ice recrystallization in storage

Extract	Stabilizer (%)	Week 0	Week 4
Control	0	36.2±0.2 <sup>B</sup>	86.4±14 <sup>A</sup>
	0.2	39.5±4.3 <sup>B</sup>	82.5±13 <sup>A</sup>
Grapeseed	0	34.2±1.7 <sup>B</sup>	33.2±3.8 <sup>B</sup>
	0.2	31.1±3.2 <sup>B</sup>	35.7±0.1 <sup>B</sup>
Green Tea	0	32.7±0.2 <sup>B</sup>	34.9±0.9 <sup>B</sup>
	0.2	32.9±1.3 <sup>B</sup>	37.6±0.7 <sup>B</sup>

*Again, mechanism for inhibition effect is unknown.*



# Fruit Extract/Sources in Ice Cream

- Some previous studies have shown that fruit extracts can inhibit melting, as in the Japanese “no melt” popsicles

Ice Cream  
Formula  
15% fat  
3% protein

Experimental Design:

% Addition to Ice Cream

Fruits	Standardized extract	Freeze-dried powder	Juice concentrate
<b>Strawberry</b>	<b>3.5%</b>	3.5%	20%
<b>Blueberry</b>	3.5%	3.5%	
<b>Cranberry</b>	3.5%		

Extract Phenolic Content

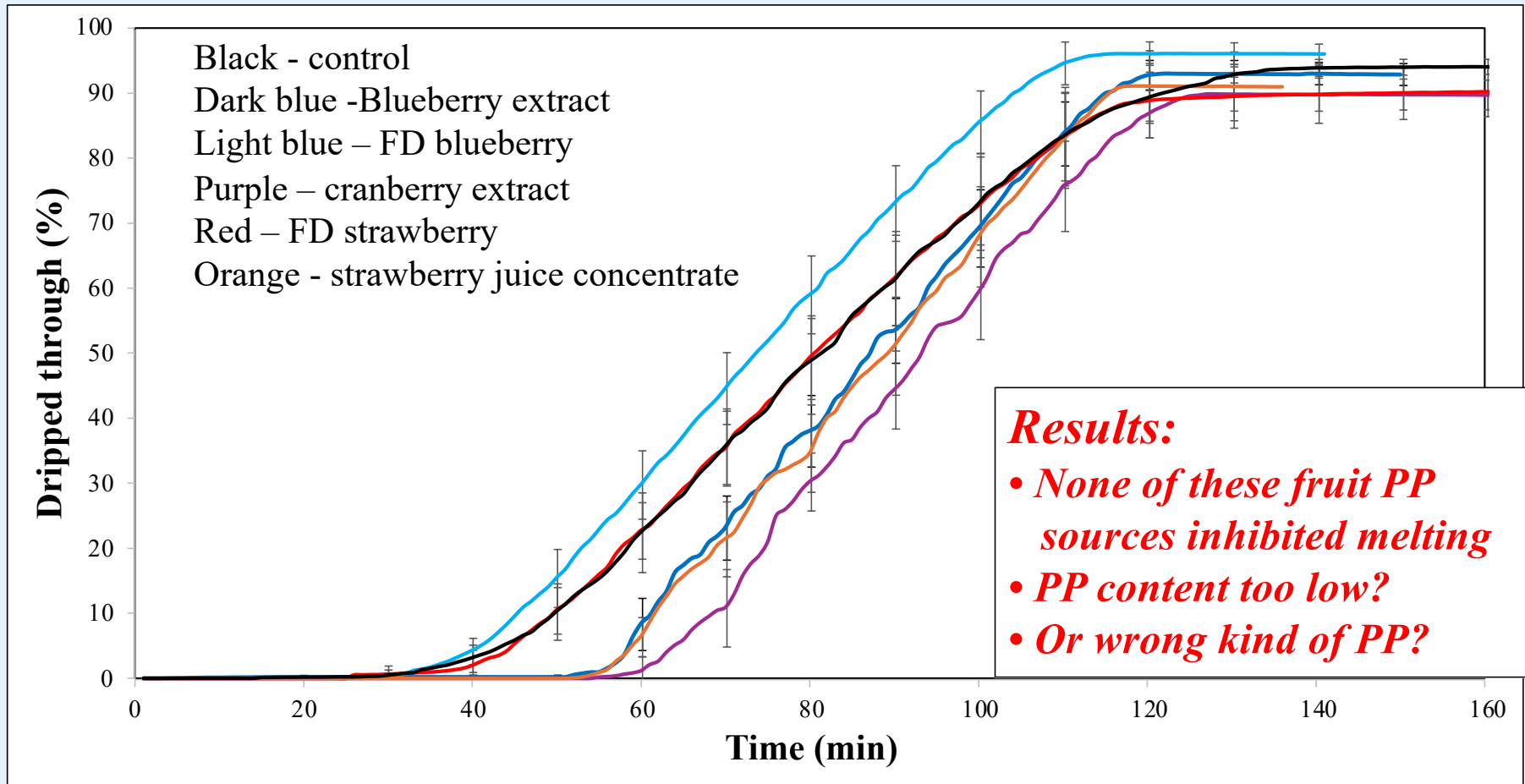
- Strawberry = ~1%
- Blueberry = 30%
- Cranberry = 15%

## Fruit Extract/Sources in Ice Cream

- Lower polyphenol (and proanthocyanadin, PCA) content, below the threshold value found in our previous studies
  - May contain fibers and other compounds

Crude Extract	Total polyphenols in source (%)	Total PAC in source (%)	Total polyphenols in ice cream (%)	PAC content in ice cream (%)
Blueberry extract	39±1.3	5.3±0.4	1.4	0.2
Cranberry extract	36±0.2	3.4±0.3	1.2	0.1
Strawberry freeze-dried powder	0.3±0.01	0.02±0.0	0.01	0.001
Blueberry freeze-dried powder	0.8±0.06	0.06±0.001	0.03	0.002
Strawberry juice concentrate	1.1±0.1	0.042±0.001	0.04	0.001

# Melting Profiles



# Summary of Polyphenols in Ice Cream

- Although it seems the effects of polyphenols relate to the protein-mediated fat globule aggregates, the mechanisms are not so clear
  - Concentration effect
  - pH effect
  - Interactions with other components (e.g., stabilizers)
  - Specific type of polyphenol is probably important
    - Another focus of this study showed that effect of viscosity and protein-aggregated fat globules was mostly dependent on degree of polymerization of the PP – longer chains resulted in stronger bonding with proteins
- How do PP affect ice crystal growth?

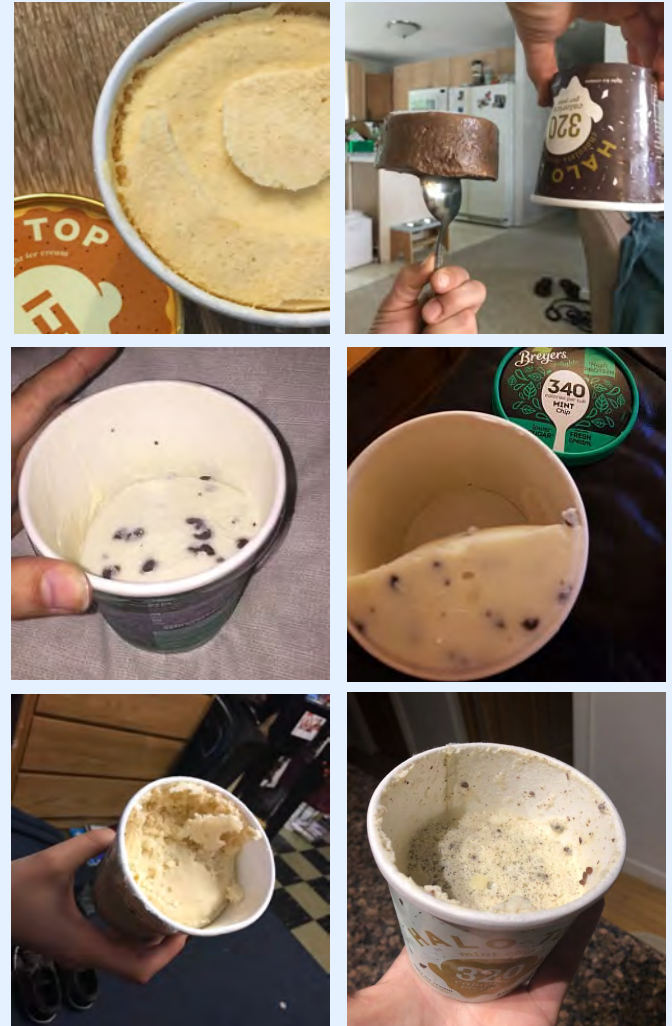


# Shrinkage in Ice Cream

- Texture defect in the air phase of frozen desserts
- Product no longer fills the volume of the container
- Destabilization and collapse of the frozen foam

*Dr. Sam VanWees*

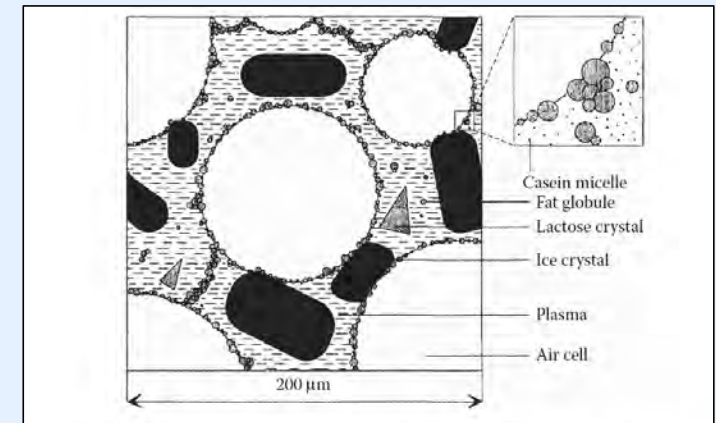
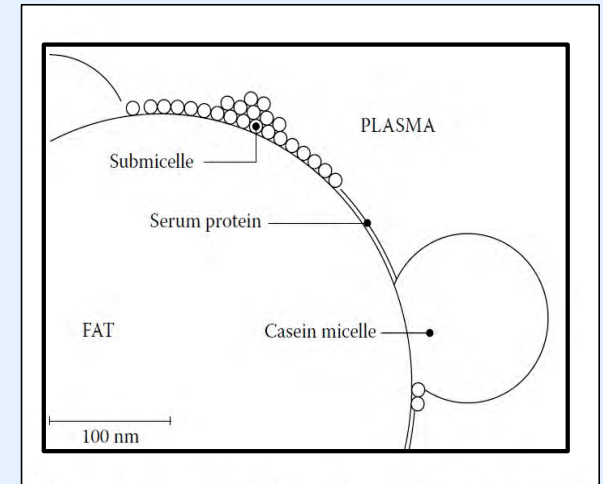
Funding: Dairy Management Inc.



# Proteins in Frozen Desserts

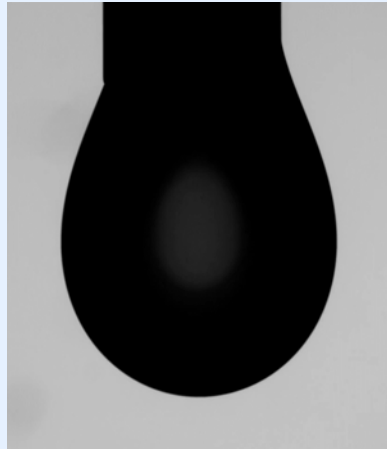
- Functionality
  - Emulsification
  - Foaming
  - Water-holding capacity
- Structure-function relationships within highly complex emulsions and foams
- Storage stability, shrinkage, and air interface viscoelasticity

*How do interfacial proteins respond to expansion and contraction?  
Could this correspond to shrinkage?*

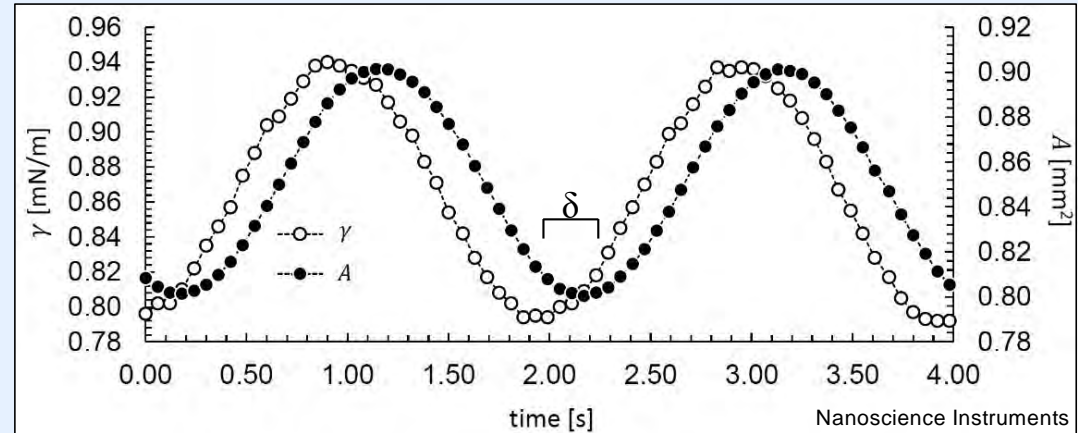


Walstra et al. (2006)

# Oscillatory Dilatational Rheology



Pulsating drop



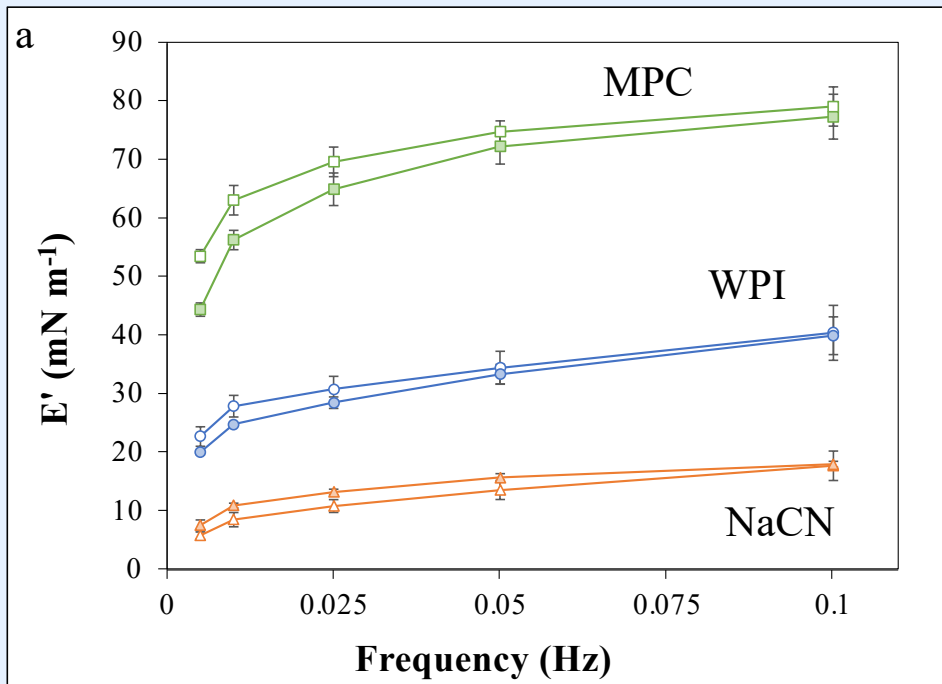
Dilatational modulus

$$E = \frac{\Delta \gamma}{\Delta \ln A}$$

$$E' = E \cos \delta \quad E'' = E \sin \delta$$

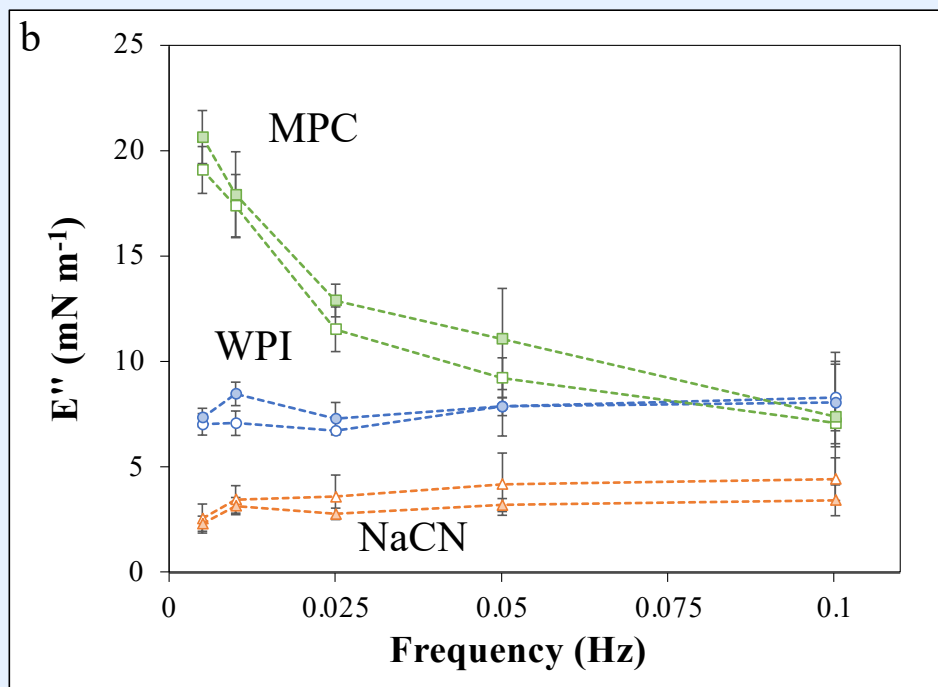
$$E(\omega) = E'(\omega) + iE''(\omega)$$

- *What air interfacial properties are stabilized by dairy proteins?*
- *Does protein concentration affect rheological properties?*
- *How might different structure-function relationships impact air cell stability?*



Open – 4% protein  
 Closed – 8% protein

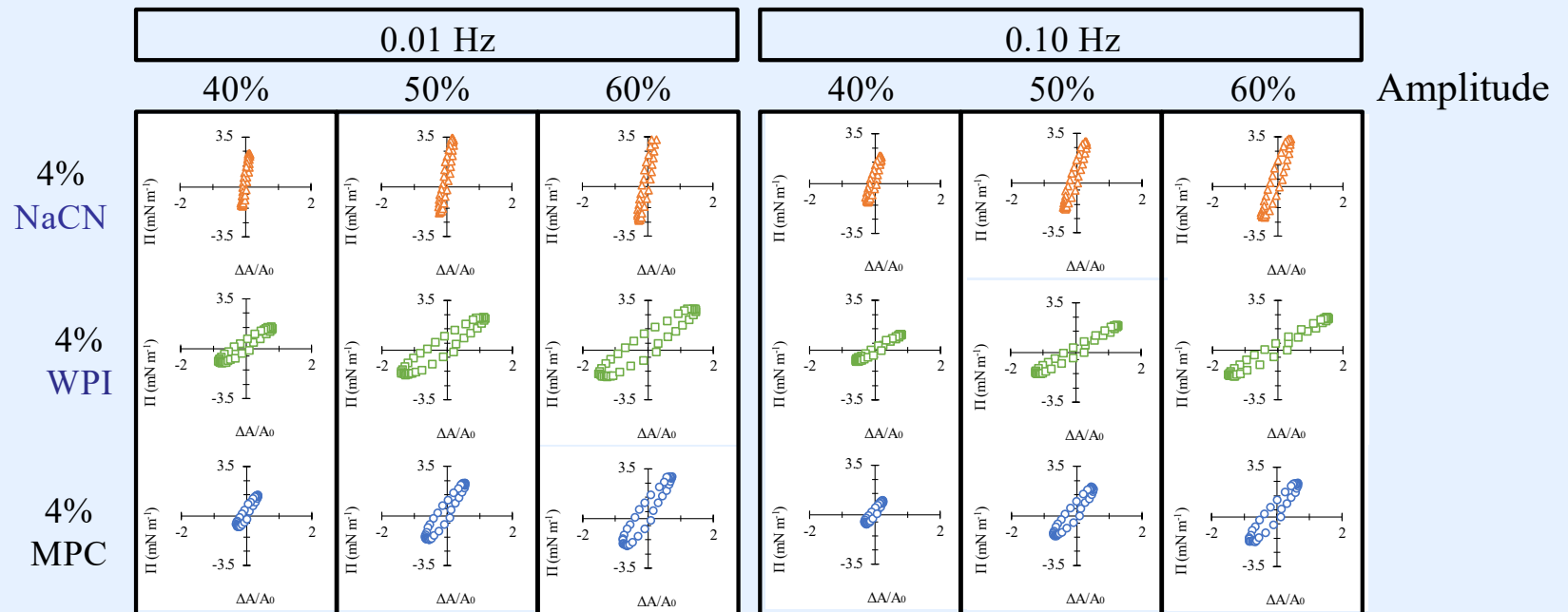
MPC creates a much firmer interface than NaCN, with WPI between





# Large Angle Oscillatory Dilation

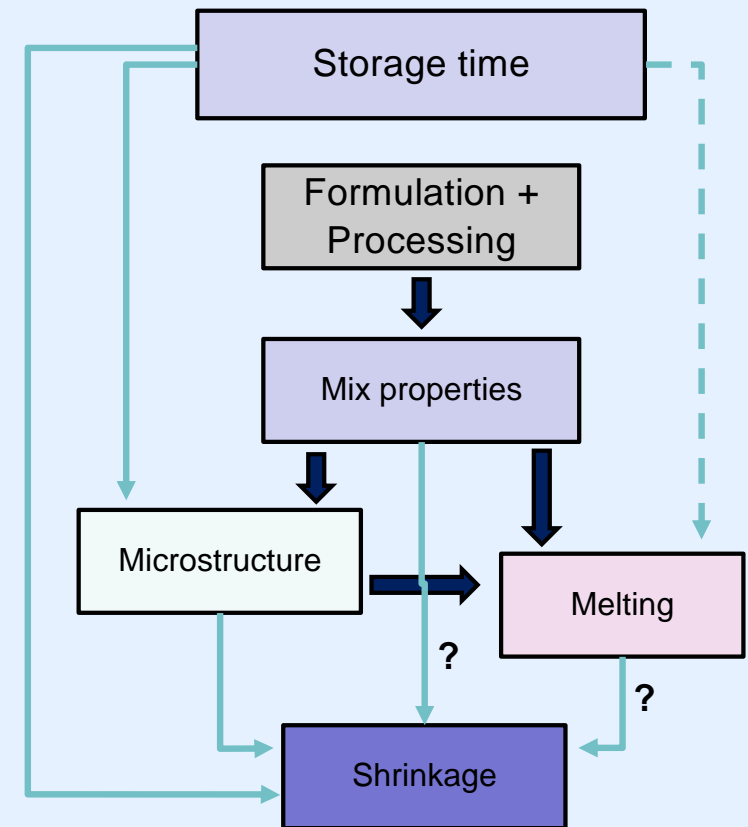
- No obvious asymmetry, which would be reflective of stress/strain hardening, but these are high concentrations of protein and effects might not show up



# Evaluate Ice Creams

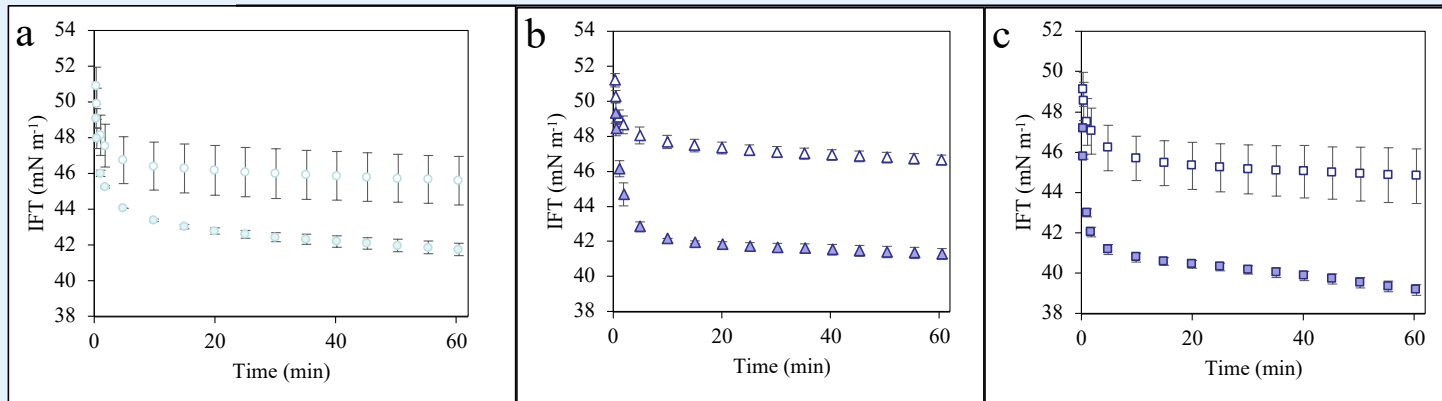
- Protein source
  - Milk protein concentrate (MPC)
  - Sodium caseinate (NaCN)
  - Whey protein isolate (WPI)
- Emulsifier addition
  - 0.0%; 0.15% MDG
- Overrun
  - 100%; 150%
- Storage time
  - 0, 2, 4, 6 weeks

Fat	12.0%
MSNF	13.3%
- Protein	6.0%
- Lactose	6.3%
Milk minerals	1.0%
Sucrose	14.5%
Stabilizer	0.2%
MDG	0.0 or 0.15%
Total solids	40%



# Mix Properties

Protein source	MDG (%)	Density (g mL <sup>-1</sup> )	IFT (mN m <sup>-1</sup> )	Viscosity (50 s <sup>-1</sup> ) (mPa·s)
MPC	0.0	1.11 ± 0.00 <sup>a, A</sup>	45.6 ± 1.36 <sup>a, A</sup>	299 ± 7.30 <sup>a, A</sup>
	0.15	1.12 ± 0.00 <sup>a, A</sup>	41.8 ± 0.35 <sup>a, B</sup>	359 ± 19.4 <sup>a, B</sup>
NaCN	0.0	1.12 ± 0.01 <sup>a, A</sup>	46.7 ± 0.28 <sup>a, A</sup>	466 ± 22.2 <sup>b, A</sup>
	0.15	1.12 ± 0.01 <sup>a, A</sup>	41.3 ± 0.31 <sup>ab, B</sup>	507 ± 30.5 <sup>b, A</sup>
WPI	0.0	1.11 ± 0.01 <sup>a, A</sup>	44.8 ± 1.36 <sup>a, A</sup>	123 ± 0.57 <sup>c, A</sup>
	0.15	1.13 ± 0.01 <sup>a, A</sup>	39.2 ± 0.26 <sup>b, B</sup>	134 ± 3.90 <sup>c, A</sup>



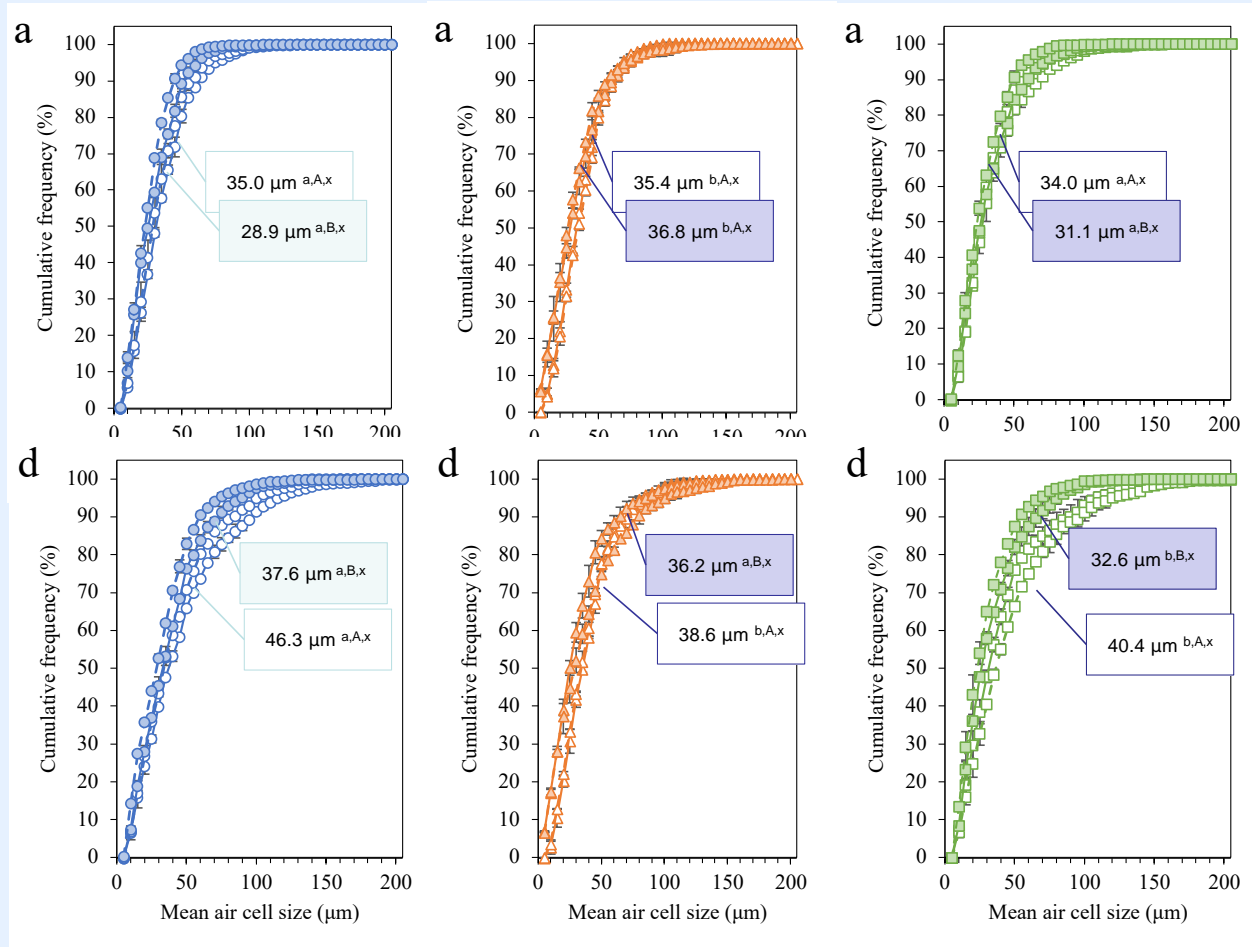
MPC (○●) NaCN (△▲) WPI (■)

a, b, c = by protein source; A, B = by MDG addition

	0.0%	0.15%
MPC	○	●
NaCN	△	▲
WPI	■	■

# Air Cells

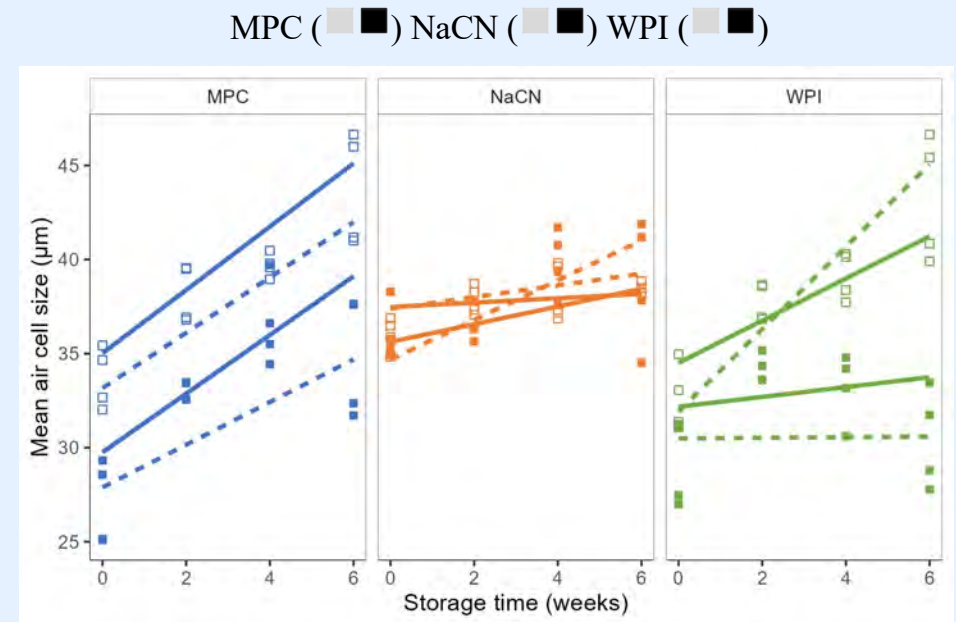
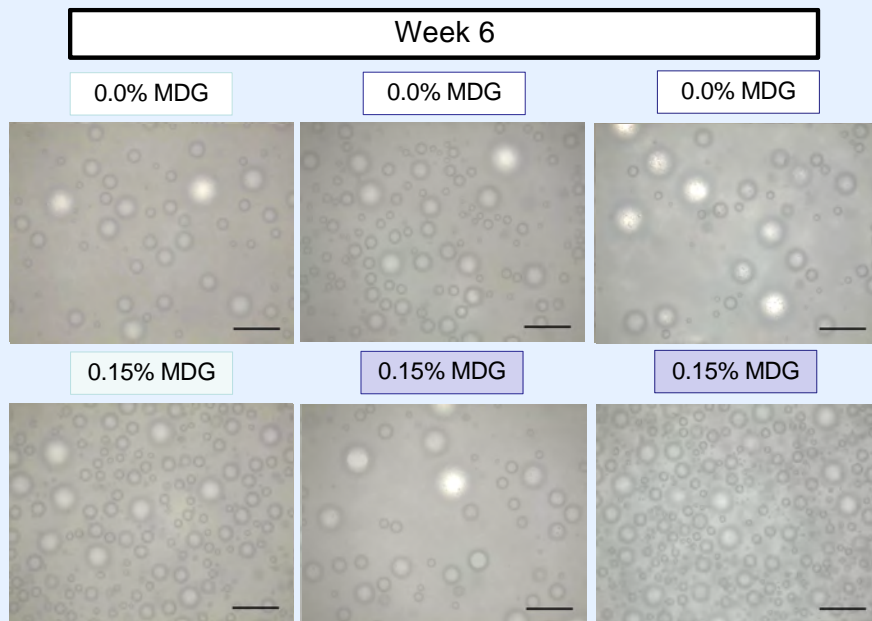
Week 0



Week 6

MPC (○●) NaCN (△▲) WPI (■) a, b, c = by protein source; A, B = by MDG addition; x, y = by overrun

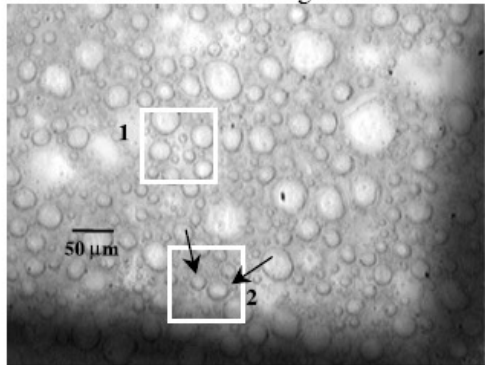
# Air Cell Coarsening



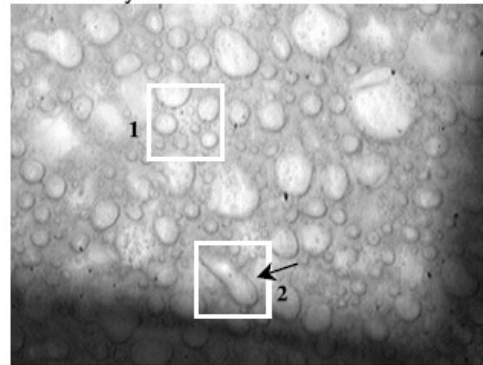
- *Coalescence, disproportionation, drainage*
- *Matrix phase properties; interfacial properties*

# Air Cell Accretion

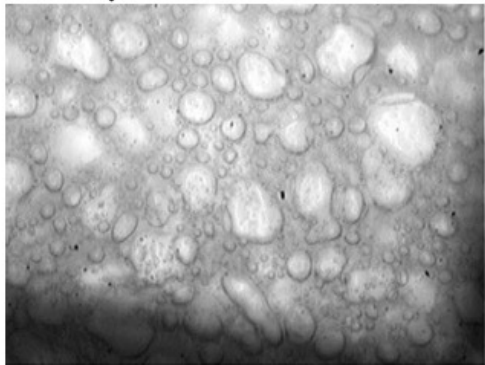
24 hours after hardening at  $-28^{\circ}\text{C}$



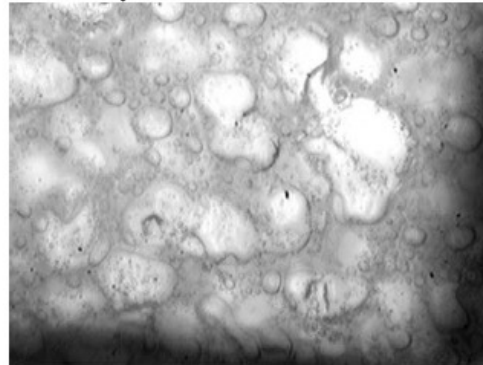
2<sup>nd</sup> day at  $-15^{\circ}\text{C}$



3<sup>rd</sup> day at  $-15^{\circ}\text{C}$



9<sup>th</sup> day at  $-15^{\circ}\text{C}$



Chang and Hartel (2002)

NaCN, 0.15% MDG,  
150% OR, 2 weeks



# Shrinkage



Protein source	MDG (%)	OR (%)	Storage time (weeks)			
			0	2	4	6
MPC	0.0	100	NS	2.82 ± 2.10 <sup>a, A, x</sup>	1.83 ± 3.50 <sup>a, A, x</sup>	0.84 ± 3.50 <sup>a, A, x</sup>
		150	NS	3.56 ± 1.05 <sup>a, A, x</sup>	1.58 ± 2.45 <sup>a, A, x</sup>	0.59 ± 1.05 <sup>a, A, x</sup>
	0.15	100	NS	3.06 ± 3.85 <sup>a, A, x</sup>	2.82 ± 2.10 <sup>a, A, x</sup>	3.61 ± 0.979 <sup>a, A, x</sup>
		150	NS	2.57 ± 0.350 <sup>a, A, x</sup>	2.07 ± 1.05 <sup>a, A, x</sup>	5.54 ± 0.350 <sup>a, B, x</sup>
NaCN	0.0	100	NS	2.92 ± 0.559 <sup>a, A, x</sup>	0.84 ± 3.50 <sup>a, A, x</sup>	2.82 ± 2.10 <sup>a, A, x</sup>
		150	NS	5.09 ± 0.280 <sup>a, A, x</sup>	1.09 ± 2.45 <sup>a, A, x</sup>	3.66 ± 1.19 <sup>a, A, x</sup>
	0.15	100	NS	1.21 ± 0.168 <sup>a, A, x</sup>	0.34 ± 1.40 <sup>a, A, x</sup>	1.18 ± 1.05 <sup>a, A, x</sup>
		150	NS	<b>14.6 ± 3.64<sup>b, B, y</sup></b>	<b>14.7 ± 2.10<sup>b, B, y</sup></b>	<b>32.0 ± 1.40<sup>b, B, y</sup></b>
WPI	0.0	100	NS	0.59 ± 3.15 <sup>a, A, x</sup>	2.32 ± 1.40 <sup>a, A, x</sup>	1.53 ± 3.08 <sup>a, A, x</sup>
		150	NS	1.18 ± 0.210 <sup>a, A, x</sup>	6.28 ± 1.40 <sup>a, A, x</sup>	4.30 ± 1.40 <sup>a, A, x</sup>
	0.15	100	NS	0.59 ± 1.05 <sup>a, A, x</sup>	2.32 ± 1.40 <sup>a, A, x</sup>	1.53 ± 0.280 <sup>a, A, x</sup>
		150	NS	3.56 ± 1.05 <sup>a, A, x</sup>	5.09 ± 0.280 <sup>a, A, x</sup>	3.31 ± 2.80 <sup>a, A, x</sup>

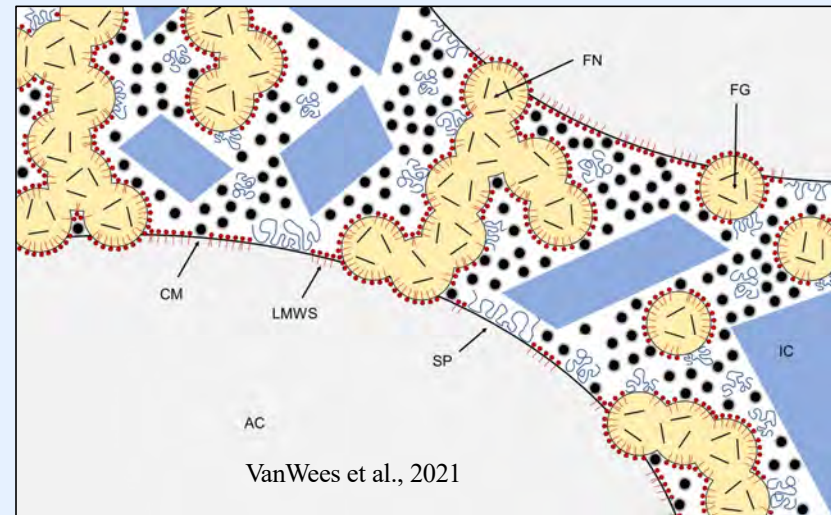


a, b, c = by protein source; A, B = by MDG addition; x, y = by overrun

NS = no shrinkage

# Understanding Shrinkage

- Air phase destabilization is thermodynamically favorable, the best we can do is kinetically inhibit it
- Dependent upon:
  1. Composition and rheological properties of the air interface
  2. Composition and rheological properties of the matrix
  3. Ability of matrix to withstand temperatures and/or pressure changes.



*The problem of shrinkage remains an issue*



*Ice cream is complex!*

**Questions?**



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