Analysis of Frozen Desserts

The Frozen Dessert Center offers a wide range of analytical services. We provide the following comprehensive tests:

Ice crystal size distribution

Ice crystal size and changes in ice crystal size are one important component of ice cream and frozen dessert quality. In general, as ice crystals grow, the icier the finished product will be perceived. Measuring ice crystal size is a way to gain insight into how formulation, processing parameters, and freezing systems are impacting ice crystal size and growth over time.

Ice crystals are measured by using a light microscope housed in an insulated glove box system (Donhowe and others, 1991) at a temperature of −15 °C. For analysis, product samples are taken from the middle of the container in the glove box, disposing the top layer of the product, and are transferred to a chilled microscope slide. A drop of chilled 50% pentanol and 50% kerosene dispersing solution is added to aid in dispersing ice crystals and to assist in image quality. A chilled cover slip is placed on top of the sample and by applying a twisting motion and minimal pressure with chilled tweezers, the sample is gently spread thin for additional image clarity. Ten-20 images at 40x magnification are taken to insure that at least 300 crystals can be counted and sized for each replicate. Ice crystal images are edited and then analyzed using Image Pro Plus software.

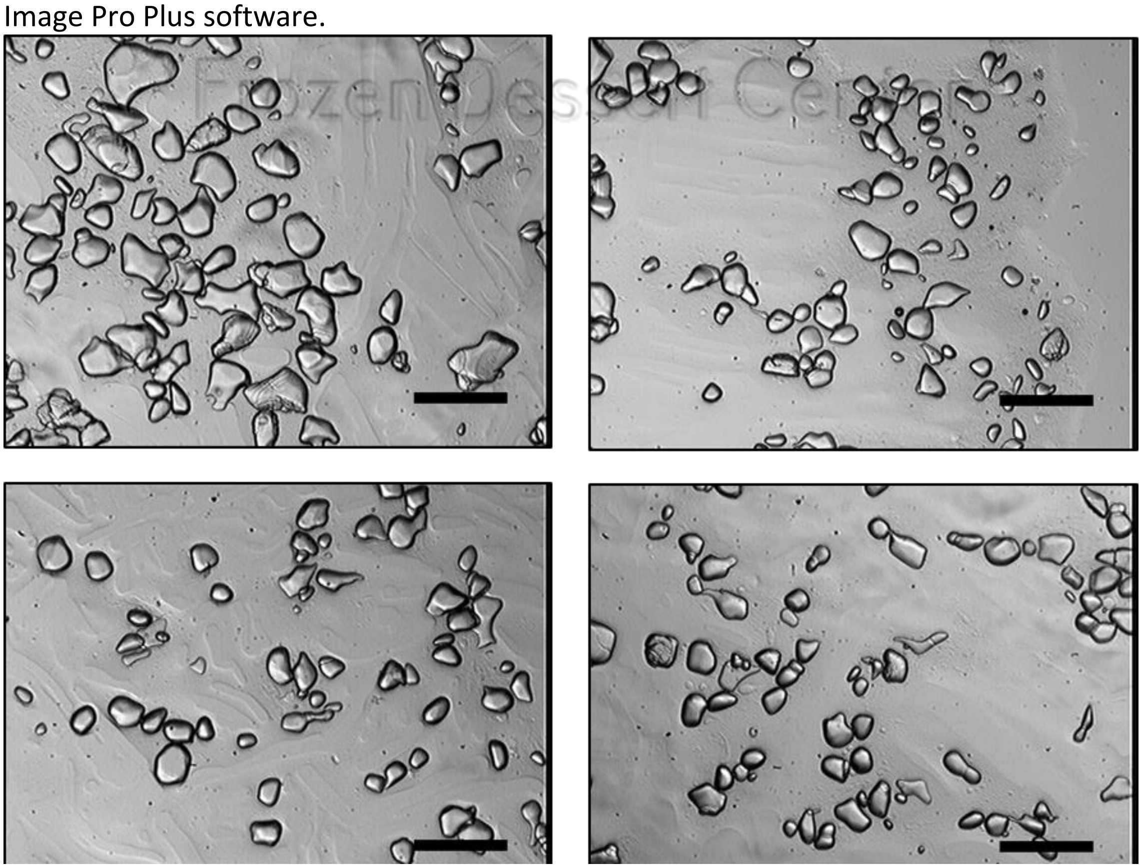


Photo Credit Sam VanWees. Ice crystal images taken at 40X magnification.

References:

Donhowe, D. P., Hartel, R. W., & Bradley, R. L. (1991). Determination of ice crystal size distributions in frozen desserts. Journal of Dairy Science, 74, 3334-3344.

Air cell size distribution

Air is an important ingredient in ice cream and some frozen desserts. If a product is high in overrun (amount of air whipped in) it will be fluffy and light, if a product is low in overrun, it will be denser. The same formula can have very different sensory properties at different levels of overrun. Mix formulation, freezer type, as well as processing conditions can have an impact on the size of the air cells in the final product. Air cell size, like ice crystal size, can change during storage and temperature abuse.

Similar to ice crystal size analysis, air cells are analyzed using the same refrigerated glove box (Chang and Hartel 2002). To properly image air cells, special slides are made by gluing 2 cover slips, approximately 1 to 1.5 cm apart, on a microscope slide, this creates a well, approximately 100 to 200 µm deep. In the glove box (−15 °C), a thin slice of hardened ice cream is taken from the middle of each container, discarding of the top layer of the product, and transferred to the well of the prepared microscope slide. A drop of chilled 50% pentanol and 50% kerosene dispersing solution is added to assist in image quality, where needed, and a cover slip is placed over the well. The glove box temperature is then raised to −6 °C allowing for air cells to float to the surface of the cover slip. Using a light microscope and camera, images of the air cells are taken at 40× magnification. Air cells are traced and analyzed for their size using Image Pro Plus software. For each replicate, at least 300 air cells are traced.

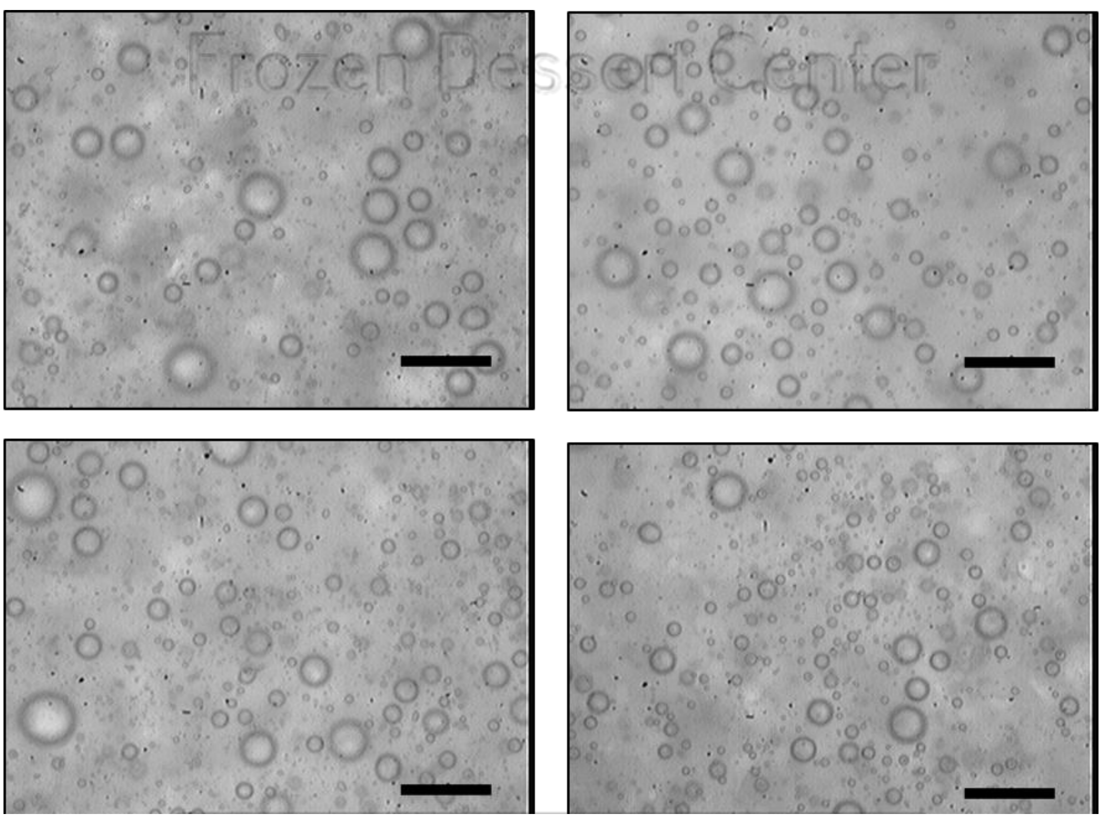


Photo Credit San VanWees. Air cell images taken at 40X magnification.

References:

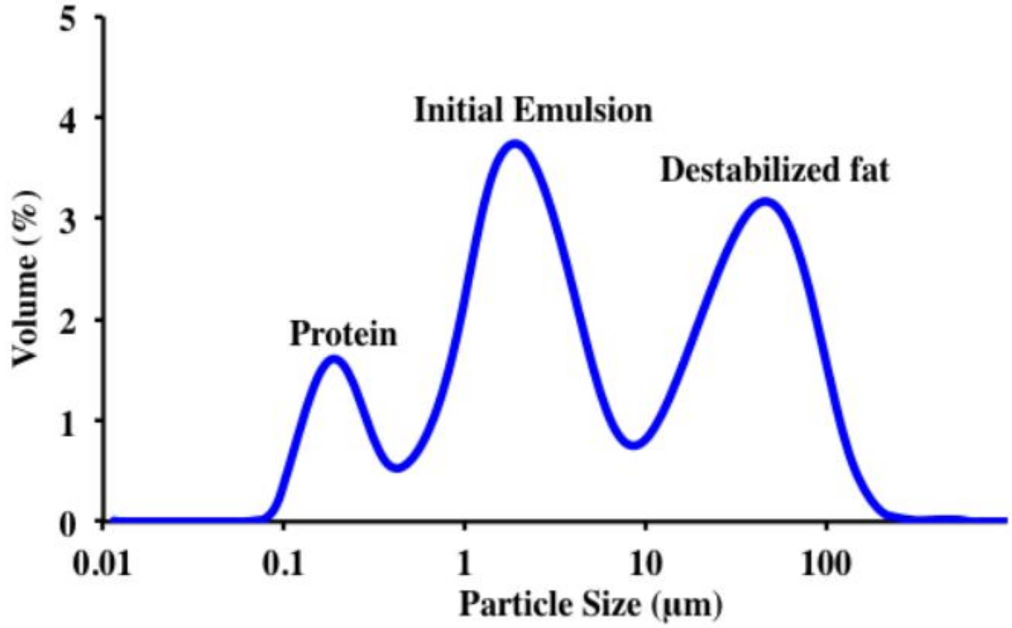
Chang, Y.-H., & Hartel, R. W. (2002). Development of air cells in a batch ice cream freezer. Journal of Food Engineering, 55, 71-78.

Fat Globule Size Distribution

The size distribution of fat globules in a sample can give us insight into an important structural element found in some frozen desserts. Partial coalescence, or fat destabilization, is a phenomenon that occurs in frozen desserts containing butterfat and possibly some types of vegetable fats. Because of the complex array of fatty acids and triglycerides, butterfat has a very wide melting range. Because of this property, even at low temperatures, within each fat droplet, there is a portion of solid and liquid fat. During the dynamic freezing process, when fat droplets collide, they stick together, but because of the solid fat portion, the majority of the original structure of each droplet is retained. This allows for clusters of fat droplets to form. These partially coalesced fat globules will help stabilize air cells and create a sort of internal scaffolding in the finished product. The degree of partial coalescence can impact melt rate as well as the perceived creaminess of the final frozen dessert. However, this phenomenon can be overexpressed, and lead to the formation of butter granules, which is considered a defect.

Fat globule size is measured using a Mastersizer 2000 (Malvern Instruments LTD, Malvern, Worcestershire, UK). Deionized water (refractive index (RI) 1.33) is used to disperse the melted frozen dessert. The refractive index of the dispersed phase is adjusted based on the fat source. A refractive index of 1.46 is used when the fat source of the dispersed phase is butterfat. Drops of diluted ice cream are added until obscuration values of 13-15% were obtained (Goff & Hartel, 2013).

This generates a particle size distribution. In general, the particle size distributions of ice cream mix and melted ice cream have characteristic peaks, see figure below (Bolliger and others, 2000). Ice cream mixes exhibit two peaks; the peak appearing at about 0.3-0.4μm represents the casein micelles in the sample, the second peak, usually centered around 1μm, is the emulsion peak, representing the individual fat globules. Melted ice cream generally has a third peak beginning somewhere between 3 and 10 μm; this peak represents the partially coalesced fat clusters that have formed during the freezing process (Goff & Hartel, 2013). The extent of partial coalescence can be determined by comparing the particle size distribution of aged ice cream mix with that of the melted ice cream.



To qualitatively verify the Mastersizer data, samples are diluted by mixing two drops of the melted sample with 2 mL of water. Drops of the dilute sample are placed on glass slides and viewed under the microscope at 400x magnification using a light microscope. Images are taken of unique features found in the sample.

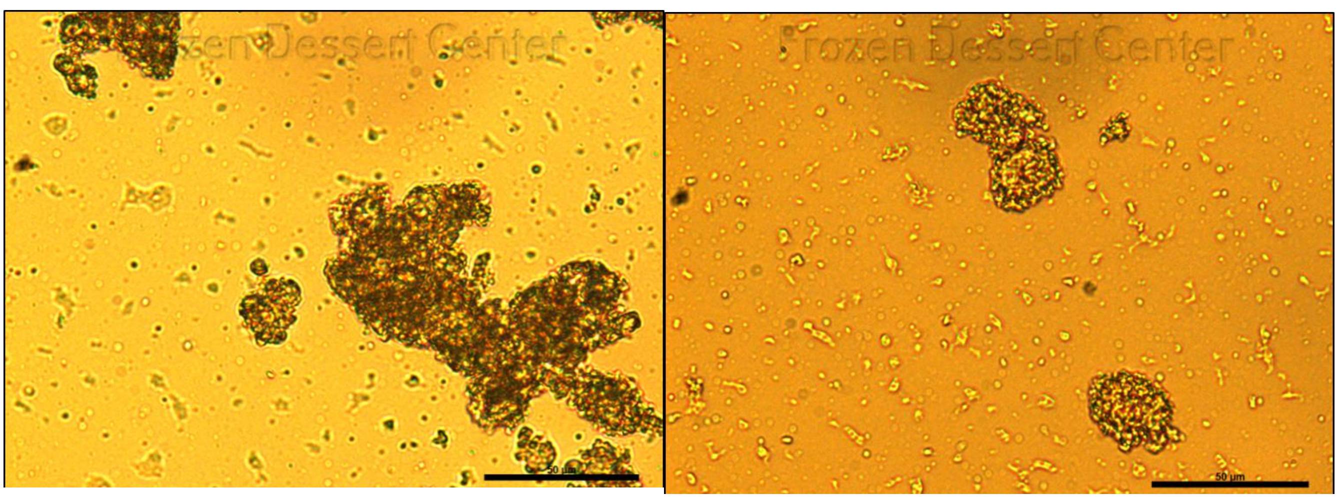


Photo Credit Sam VanWees. Images of destabilized fat taken at 400X magnification

References:
Bolliger S, Goff HD, Tharp BW. 2000. Correlation between colloidal properties of ice cream mix and ice cream. Int Dairy J 10:303–9.

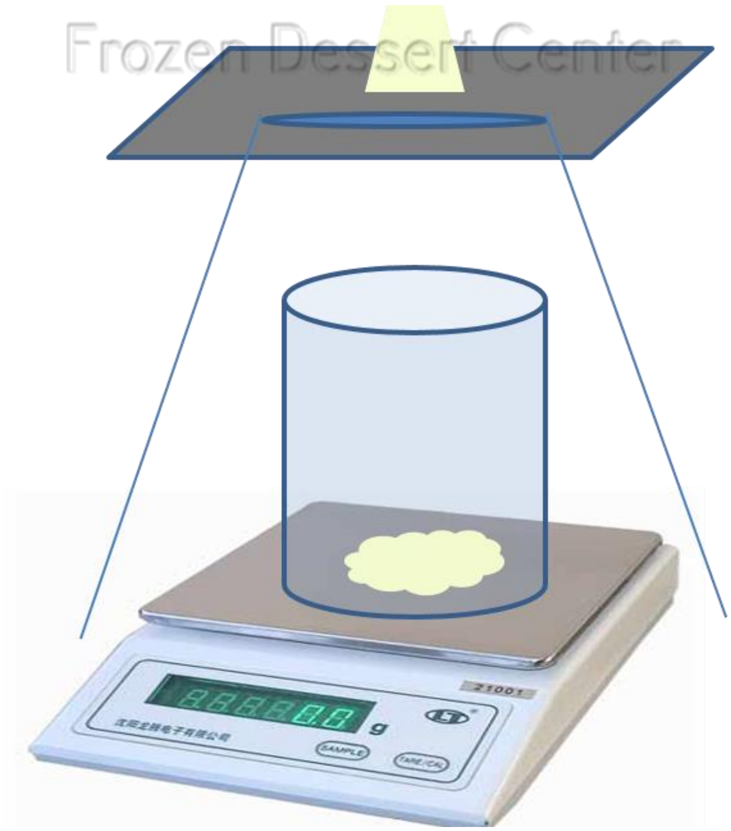
Goff, H. D., & Hartel, R. W. (2013). Ice cream (7th ed.). New York, NY, USA: Springer US.

Melt-down rate behavior

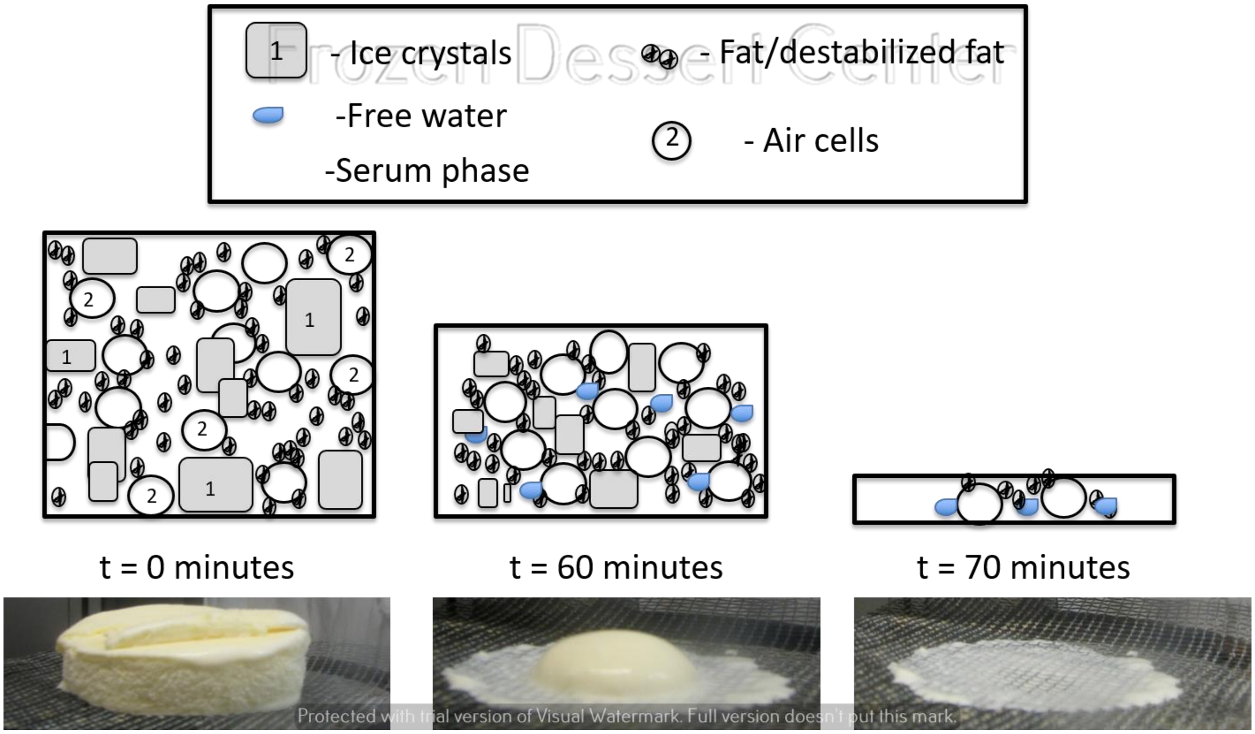
Melt-down behavior of a frozen dessert is influenced by a complex relationship between formulation, size and number of ice crystals and air cells, overrun, fat destabilization, mix viscosity, freezing point depression, and other properties yet to be discovered. Frozen desserts can melt quickly and completely into a homogenous puddle or can even appear to not melt and hardly lose their shape over time. This property is something that is very noticeable to the consumer, as seen in a viral video about ice cream sandwiches (https://youtu.be/MoqDoet_RtA). Depending on how a product is consumed, in a dish, on a cone, or between two chocolate wafers, melt rate is an important attribute to consider.

Because of the nature of the microstructural elements, there are competing forces that influence melt rate and behavior. For example, air is a good insulator, so ice creams with higher overruns, may melt slower than low or no overrun product. At the same time, ice is a good heat conductor, but because of the latent heat of fusion, this slows the melt rate. So higher ice phase volume may slow melt rate in comparison to products with lower ice phase volume. As ice melts, the liquid water dilutes the ice cream matrix, encouraging the serum to drain. This can be slowed by the network of partially coalesced fat. It may be possible to manipulate formula and processing conditions in order to influence the formation of the frozen dessert microstructure and potentially achieve a desired melt-down behavior and rate (Warren & Hartel, 2018).

The melt rate of a frozen dessert is determined by the screen drip-through test (Goff & Hartel, 2013). The sample is allowed to equilibrate to 15°C for 60 min. An 80 g sample of ice cream is then cut from the pint and placed on a metal screen (three holes/cm). The screen is placed on a ring stand suspended over a beaker on a scale. As the ice cream melts, the weight of the ice cream that drips through is measured every 5 min. The test is allowed to continue until all the ice cream has dripped completely through the screen or for a total of 120 min. The rate of melting is determined by plotting the dripped through weight (g) against time (minutes) and finding the slope of the linear portion of the curve (Muse & Hartel, 2004). The test is conducted in a room with an ambient temperature of 21.5 ± 0.5C.



A measured slice of ice cream is placed on a screen, suspended over a beaker on a scale. Weight measurements are taken every 5 mins to determine drip through rate. Credit: Maya Warren.



Credit Maya Warren

References:

Goff, H. D., & Hartel, R. W. (2013). Ice cream (7th ed.). New York, NY, USA: Springer US.

Muse, M. R., & Hartel, R.W. (2004). Ice cream structure elements that affect melting rate and hardness. Journal of Dairy Science, 87, 1-10.

Warren, Maya & Hartel, Richard. (2018). Effects of Emulsifier, Overrun and Dasher Speed on Ice Cream Microstructure and Melting Properties: Ice cream microstructure and melt rate…. Journal of Food Science. 83. 10.1111/1750-3841.13983.

Heat Shock and Temperature Abuse Studies

Exposing frozen desserts to intense temperature fluctuations can simulate an accelerated shelf-life. In general, shelf life is determined based on the stability of a product's physical, chemical, and microbiological safety, nutritional quality, and desired sensory retention when stored under recommended conditions. Frozen desserts are stored at very low temperatures, so microbiologic and nutritional degradation are not of significant concern, but loss of sensory acceptability and changes to physiochemical properties are.

Frozen desserts have numerous delicate structures, especially ice crystals and air cells, that can change dramatically during storage and especially when exposed to temperature fluctuations or heat shock (Goff & Hartel, 2013). An increase in course texture due to ice crystal growth, which is among the most common consumer complaint, product shrinkage, and development of gummy or sandy textures greatly diminished the quality perceptions of the product.

Frozen dessert product quality changes drastically at increasing storage temperatures. Putting a frozen dessert through cycles of temperature abuse, can give insights into how well product quality will hold up or deteriorate as it goes through the distribution chain, to store shelves, to a consumer's home freezer. If we focus on only ice crystals, studies have shown that when product is stored under supermarket conditions, with temperatures cycling between −9.4 and −15°C product can become noticeably icy in 1–4 weeks and unacceptably icy in 3–10 weeks. Another study showed that ice cream stored at −10°C had a shelf life of about 1 week, storage at −15.5°C (0°F) gave a shelf life of approximately 5 weeks, and as storage temperatures dropped to −30°C (-22°F), shelf life increased logarithmically. Estimating a shelf life for a frozen dessert is not a straight forward process, but testing a product's resilience under extreme conditions can guide formulation changes that will give the best insurance policy for product quality.

An accelerated shelf-life is simulated using a freezer cabinet that under goes regular temperature cycling. Product is put into a cabinet set to -13°C (8.6°F) that cycles between -10°C to -16°C (14°F to 3.2°F) every 30 minutes. A study typically lasts 4 weeks, but can extend as long as desired. Samples can be taken at various points during the study to measure changes in microstructural elements, most commonly to track changes in mean ice crystal size.

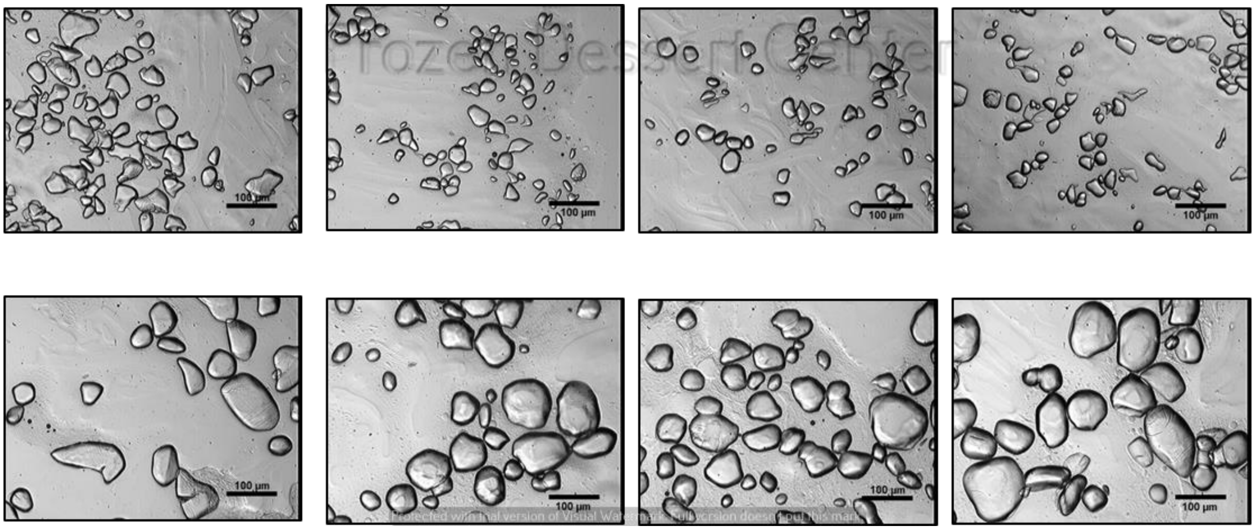


Photo credit Sam VanWees

Top Row: Ice crystals immediately after hardening. Bottom Row: Ice crystals after 3 weeks of heat shock.

References:
Giménez, Ana & Ares, Florencia & Ares, Gastón. (2012). Sensory shelf-life estimation: A review of current methodological approaches. Food Research International. 49. 311–325. 10.1016/j.foodres.2012.07.008.

Goff, H. D., & Hartel, R. W. (2013). Ice cream (7th ed.). New York, NY, USA: Springer US.

Labuza TP, Fu B. 1997. Shelf life testing: Procedures and prediction methods. In: Erickson MC, Hong YC (eds) Frozen Food Quality. pp 377–415. CRC Press, Denver CO

Park, J. & Koh, Jong-Ho & Kim, Jin-Man. (2018). Predicting Shelf-life of Ice Cream by Accelerated Conditions. Korean journal for food science of animal resources. 38. 10.5851/kosfa.2018.e55.

Wittinger SA, Smith DE (1986) Effect of sweeteners and stabilizers on selected sensory attributes and shelf life of ice cream. J Food Sci 51(6):1463–1466, 1470

Rheology
Rheology is the measure of how a substance deforms or reacts to a force. Given the complex structure of frozen desserts, there are many ways to measure these phenomena. We can help select or design a test that is right for your application.

Common tests include: mix viscosity, temperature ramps (structure deformation with temperature change), frequency or strain sweeps (structure deformation with strain/stress).

We have a DHR-2 rheometer (TA instruments) complete with multiple geometries for a variety of applications. We have cooling and heating capabilities that allow for testing from -15°C to ~40°C (5°F to 104°F) for frozen desserts.

Mix viscosity
Mix viscosity has been shown to have an inverse correlation with sensory perception of iciness. As mix viscosity increases, ice creams may be perceived to be less icy. Mix viscosity also influences melt rate, with higher viscosity mixes trending toward slower melt rates (Amador et al, 2017).

Mix viscosity is measured using a rheometer (Discovery HR-2 hybrid TA Instruments, New Castle, Del., U.S.A.). Mix samples are allowed to age for at least 4 hours and up to 24 hours. The samples are then equilibrated to 4 °C. Using bob and cup geometry, measurements are taken using a shear sweep from 100 to 1 s−1. The data are fit using the Herschel-Bulkley model:
$\sigma = \sigma_0 + k\dot{\gamma}\ n$
Where σ is the shear stress (Pa), σ_0 is the yield stress (Pa), $\dot{\gamma}$ is the shear rate (s^{-1}), k is the consistency coefficient (Pa s^n), and n is the flow behavior index. Apparent viscosity (Pa·s) is recorded at a shear rate of 50 s^{-1}.

Small amplitude oscillatory shear or Oscillatory Thermo-Rheometry (OTR)
The complex structure of frozen ice cream gives it very interesting rheological characteristics. Frozen aerated desserts are essentially a solid foam, with its microstructure influencing viscoelastic behavior of the frozen and melted product. When frozen, the ice phase is the biggest driver of viscoelastic character. When melted, the air and fat networks drive rheological behavior. This test method can show how the rheological behavior changes during temperature change. In order to get the greatest amount of context, this test should be done in combination with analysis of other physical attributes of a frozen dessert.

Oscillatory thermos-rheometry is measured by first tempering samples at -20°C. A chilled cylindrical cutting tool is used to cut a disk of frozen dessert with a diameter of 25 mm and height of 3 mm. Samples are conditioned at −20°C for 2 hr before measurements. A rotational rheometer (DHR-2, TA Instruments, New Castle, DE) with parallel plate geometry (25 mm diameter) is used for rheological analysis. The stepped bottom Peltier plate and upper plate are cross-hatched to prevent slip. In order to confirm that samples were within the linear viscoelastic regime (LVR) for oscillatory measurements, oscillatory strain amplitude sweeps (10

s^{-1} angular frequency) were conducted at −15, 0, and 20°C. Within the LVR, the test may be carried out without destroying the structure of the sample. A temperature ramp test is used to evaluate the rheological behavior of frozen desserts. The measurement temperature is continuously increased from −15 to 40°C and the storage (G') and loss (G'') moduli for the samples is recorded using TRIOS software (TA Instruments, New Castle, DE). The storage modulus, G', describes the elastic or solid-like properties of a material, and the loss modulus, G'', describes the viscous or liquid-like properties. The gap width is set to 1.8 mm for all measurements. Frozen desserts are analyzed at a frequency of 1.59 Hz (angular frequency 10 s^{-1}) and 0.01% strain from −15 to 0°C. To remain within the LVR for melted frozen desserts, strain are adjusted to 0.1% strain from 0 to 40°C. Measurement temperature are increased at a rate of 0.5°C min^{-1}. Measurements were recorded every 4 s for a total of 1,725 points (VanWees et al, 2020).

References:

Amador, Julia & Hartel, Rich & Rankin, Scott. (2017). The Effects of Fat Structures and Ice Cream Mix Viscosity on Physical and Sensory Properties of Ice Cream. Journal of food science. 82. 10.1111/1750-3841.13780.

VanWees, Samantha & Rankin, Scott & Hartel, Richard. (2019). The microstructural, melting, rheological, and sensorial properties of high-overrun frozen desserts. Journal of Texture Studies. 51. 10.1111/jtxs.12461.

Sensory Evaluation
Consumer evaluation and testing can be a great way to understand how your product will be received and perform in the market. We have capabilities to run a number of panel types and will design a test that is right for your application.

Example Tests:
Preference Testing
Typically, 50-100 panelists are needed. Panelists will be asked a series of questions about specific attributes (such as overall liking, appearance liking, texture liking, flavor/aroma liking, etc) of a product, with the panelists responding to how much they like or dislike that attribute. Panelists may also be asked ideality, Just about right (JAR), questions to determine how adequate an attribute is perceived to be.

For example:
9-point hedonic scale:
9 - like extremely
8 - like very much
7 - like moderately
6 - like slightly
5 - neither like nor dislike
4 - dislike slightly
3 - dislike moderately
2 - dislike very much
1 - dislike extremely

Just about right Scale:
5 - Too sweet
4 - Slightly too sweet
3 - Just about right
2 - Slightly not sweet enough
1 - Not sweet enough

Difference or discrimination tests:
These types of tests are designed to determine if there is a perceptible difference between samples.
Triangle test: panelists are presented with 3 unknown samples and must identify which is the odd sample.
Duo trio test: panelists are presented with a reference sample and 2 unknown samples. They must decide which unknown sample matches the reference sample.
Paired comparison: panelists are presented with 2 samples and asked to judge which one fulfills some specific criteria (such as sweetness, flavor intensity, etc).

In-home use tests: If a product relies on consumers to complete final preparation steps at home, this test can help determine if packaging configuration, written instructions, and timing recommendations are correct, easy to manage or where there may be room for improvement.
Focus groups: This method offers an opportunity to select a targeted consumer group and probe into specific attributes, needs, desires, and opportunities related to targeted product.

Trained descriptive panel analysis: This method is very time and labor intensive, but allows for human subjects to be calibrated on specific criteria and attributes related to a specific product. A well trained panel can function similarly to an analytical instrument. For example, a hedonic question can only ask a panelist their opinion about how much they like or dislike something, like the sweetness of a product, where as a trained descriptive panel can give a score to the degree of sweetness based on a specific reference scale. This can give more specific details about how something is different, rather than just identifying that something is generally different.

Basic product specs
If you need to know some general composition and quality attributes of internal or competitor samples, we can give you a basic profile using these methods:

Total milk fat determination- Babcock method
Moisture/Total Solids- CEM Smart System 5
pH- pH Meter
Microbial analysis- inquire for specific capabilities
Freezing point depression- differential scanning calorimetry

Schedule a meeting to discuss your unique opportunity. Quotes available on request.

Contact: (Emily Daw, FDC Director) edaw@wisc.edu