

Effects of Freezing in Sweetened Dried Cranberry Production

Sponsored by Ocean Spray®

A Major Qualifying Project Report
Submitted to the Faculty of the

WORCESTER POLYTECHNIC INSTITUTE

in partial fulfillment of the requirements for the degrees of

Bachelor of Science in Chemical Engineering
& Bachelor of Science in Mechanical Engineering

By:

Errando B. Jayapurna (ChE)

Logan Mies (ME)

Stephen Stephansky (ChE)

Valentina Zapata Macias (ChE)

Approved by:

Professor Stephen J. Kmiotek
Chemical Engineering, WPI

Professor Sarah Jane Wodin-Schwartz
Mechanical Engineering, WPI

April 22nd, 2018

Abstract

The purpose of this project was to study and identify the effects of different freezing conditions on cranberries in Ocean Spray's Sweetened Dried Cranberry (SDC) process. By understanding how different freezing conditions affect the cellular structure of cranberries, these effects can be better accounted for during the SDC manufacturing process. The project involved a preliminary SDC lab-scale process design, and the development of a freezing method for yielding full batches of cranberries that underwent uniform freezing conditions. The freezing method concluded by this year's project should be used by future teams in carrying and testing batches through the lab-scale process.

Acknowledgements

The team would like to acknowledge the following people for their contribution, expertise, and assistance throughout this project. Without their support this project would not have been possible.

Ocean Spray®

Ryan Moriarty

Soumya Roy

Steve Nojeim

Alexander Gorman

Stephen Peabody

Andrea Kliora

Bill Russell

Nicole Garvin

Ellen Guilfoyle

Suneet Khanna

WPI Chemical Engineering Department

Prof. Stephen Kmiotek

Prof. Andrew Teixeira

Douglas White

Tom Partington

WPI Mechanical Engineering Department

Prof. Sarah Wodin-Schwartz

Peter Hefty

Table of Contents

Abstract	1
Acknowledgements	2
List of Tables & Figures	4
Chapter 1: Background	5
1.1. Overview of OceanSpray’s Full-Scale Craisin Process.....	5
1.2. Literature Review of the Effects of Freezing in Food Processing	6
1.3. Possible Effects of Freezing Rates in Ocean Spray’s Craisin Process.....	9
Chapter 2: Lab-Scale Process (Methodology)	10
2.1. Overview of the Lab-Scale SDC Process	10
2.2 Freezing	10
2.3 Slicing.....	11
2.4 Extraction	12
2.5 Infusion	13
2.6 Drying	14
2.7 Comparison of Full Scale and Lab Scale Processes	15
Chapter 3: Data Acquisition, Evaluations, & Testing	16
3.1. Freezing Rate Data Acquisition	16
3.2. Number & Size of Ice Crystals	17
Chapter 4: Process Development	18
4.1 First Run Challenges	18
4.2 Freezer Profiling	19
4.3 Introduction of a Mini Fan to the Freezer	21
4.4 Freezer Rack System	22
4.5 Uniform Freezing Conditions and Methodology	23
Chapter 5: Uniform Freezing Results	24
Chapter 6: Recommendations	27
6.1 Storing Refrigerated Berries	27
6.2 Freezing	27
6.3 Slicing.....	27
6.4 Extraction/Infusion.....	27
6.5 Drying	28
6.6 Testing/Experimentation	29
6.6.1 Lyophilizing and SEM	29
6.6.2 Thawing Leakage Test	30
6.6.3 Infusion Rate	30
6.6.4 Syrup Expression	31
6.7 Points of Contact.....	32
References	33
Appendices	34
Appendix A: Fast Freezing Treatment (-25C) Data	34
Appendix B: Fast Freezing Treatment (-40C) Data	35
Appendix C: Slow Freezing Treatment (-40C) Data	36
Appendix D: Slow Freezing Treatment (-25C) Data	37

List of Tables & Figures

Figure 1: Full Scale Craisin® Process Schematic	5
Figure 2: Freezing Curve of Plant Tissues (Brown et al, 1974)	6
Figure 3: Slow vs Fast Freezing in Plant Tissue (Roy, 2017)	7
Figure 4: Slow vs Fast Freezing Rates Cranberry SEM Images (Mahadevan, 2014)	8
Figure 5: Photos of the Freezer	10
Figure 6: Sterilite® Container	11
Figure 7: Alligator® Slicer In Use.....	11
Figure 8: Custom Extraction Mixer	12
Table 1: Extraction Specification Summary	12
Figure 9: Syrup Infusion of Cranberries	13
Table 2: Infusion Specification Summary	13
Figure 10: Dehydrator and Drying Rack	14
Table 3: Drying Specification Summary	14
Figure 11: Schematic of Thermocouple Placement Inside the Sterilite® Containers	16
Figure 12: Bench-top Freeze Dryer and Photos of Lyophilized Cranberries	17
Figure 13: Example SEM Images of Lyophilized Cranberries at 104x Magnification	17
Figure 14: Freezing curve of cranberries frozen at a rate of -3C per hour from -10C to -40C	18
Figure 15: Schematics of thermocouple location for temperature profiling of the freezer	19
Figure 16: Freezer Profiling (Corners, Faces)	20
Figure 17: Freezer Profiling with Mini-Fan Comparison (Corners, Faces).....	21
Figure 18: Rack System Schematic with Thermocouple Placement	22
Figure 19: Freezing Curves for Cranberries Frozen to -40°C and -25°C at Fast and Slow Rates	24
Figure 20: Box Plots for Cranberries Frozen to -40°C and -25°C at Fast and Slow Rates	25
Figure 21: Freezing Curve for a Single Cranberry Frozen to -40°C at a Fast Rate.....	26
Figure 22: Magnification of the Initial Phase Change (Left) and Intracellular Freezing (Right).	26
Figure 23: Orientation of the Thawing/Leakage Test.....	30
Figure 24: Components and Set-Up of the Syrup Expression Test	31

Chapter 1: Background

Ocean Spray is an agricultural cooperative owned by 700+ cranberry growers across the US, Canada, and Chile. The cooperative's products include juices, sauces, dried cranberries, and fresh fruit. The purpose of this background section is to provide adequate knowledge for understanding the purpose and scope of this project.

1.1. Overview of OceanSpray's Full-Scale Craisin Process

Full scale production of Ocean Spray Sweetened Dried Cranberries (SDC), sold under the trade name Craisins[®], requires a manufacturing process consisting of 5 main steps: freezing, slicing, extracting, infusing, and drying.

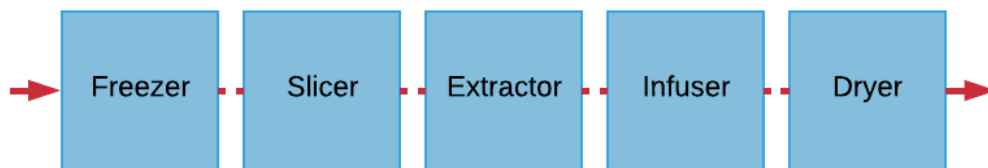


Figure 1: Full Scale Craisin[®] Process Schematic

The process formally begins with freezing harvested and cleaned cranberries at 4°F in industrial sized freezers. In the freezers, cranberries are stored in, 42''W x 48''L x 46''H, 1300 pound, wooden crates. Next, the frozen cranberries are sent through slicers to be cut at 7 mm width.

After the berries are sliced, they are sent through an extractor to remove their natural juices. Once extracted, the berries are sent through an infusion process. Extraction and infusion use nearly identical screw-conveyor, counter-current systems. Each screw conveyor transfers the fruit through a cylindrical shell filled with extraction water or infusion syrup. The screw conveyor is equipped for forward and reverse motions. For example, the screw will rotate to push fruit forward, then reverse rotation to push fruit backward, so there is net forward motion.

Finally, the cranberries are dried through a three stage (A, B, & C), continuous dryer with transfer sections between stages. Fruit is loaded onto a belt conveyor through section A, then dropped from one belt to another through section B, and so on.

1.2. Literature Review of the Effects of Freezing in Food Processing

Cranberry harvesting occurs only once a year due to their natural growing season. Because of this, and because one of the main causes in food spoilage is microorganism growth, Ocean Spray's cranberries can spend over a year in freezing before being processed. Freezing helps to inhibit microbial growth on food surfaces (ASHRAE, 1997).

When food is frozen, a significant amount of free water is immobilized in solid state. As a result, there is less water available to mobilize reactants, reducing rates of reaction and increasing concentration of extracellular solutions. Temperature reduction immobilizes the concentrated liquid phase and dehydrates microbial cells, which in turn inhibits microorganism growth (Petzold, 2009).

As observed in Figure 2 below, freezing of fruit involves a series of steps that affect the resulting cellular damage. The first step of the process, supercooling, results when the fruit is first introduced into low temperature conditions. The fruit's internal temperature drops below freezing without incurring any phase change. At the liquid/solid equilibrium point, between supercooling and extracellular freezing, nucleation occurs. Nucleation is the process during which molecules of a reactant phase, rearrange into a cluster of the product phase large enough to have the ability to grow (Anderson, 2010). Before the phase change, the cooling rate slows and then spikes up due to latent heat being released as a result of crystallization, where liquid water crystallizes to form solid ice.

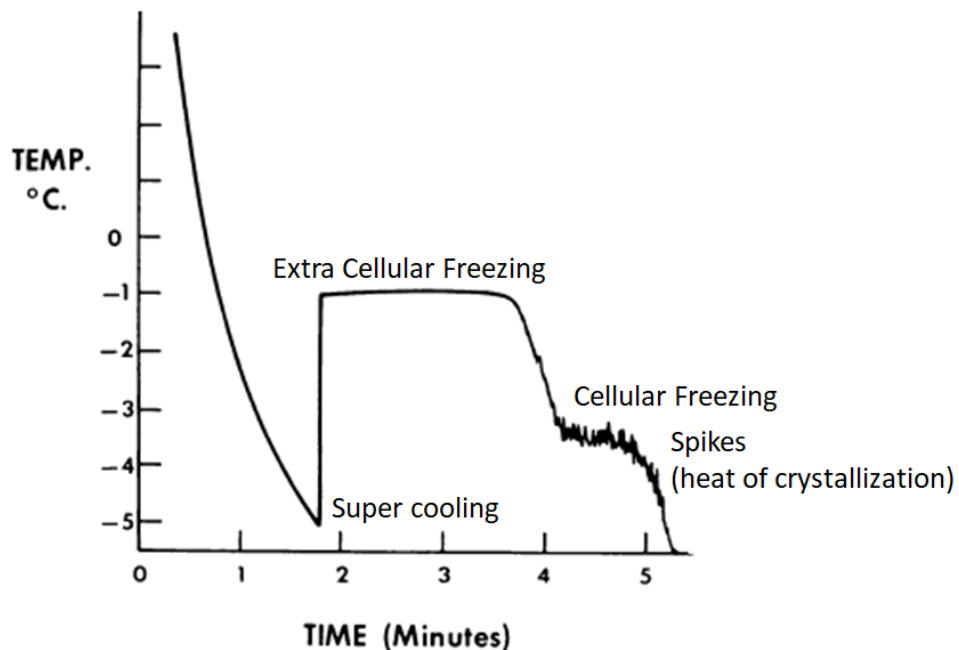


Figure 2: Freezing Curve of Plant Tissues (Brown et al, 1974)

The next step, extracellular freezing, results when the water surrounding the plant cells experiences a phase change and becomes ice. The plateau at 0°C is the release of latent heat as the water transitions into ice. The final step in the curve is intracellular freezing. During intracellular freezing, the temperature drops again and the water inside the plant cells begins to undergo a phase change to become ice. As the phase changes begin, we observe small increases in temperature, or spikes, in the freezing curves. These spikes are the result of the heat of crystallization as ice forms inside the plant cells. The larger the spikes, the larger the ice crystals.

The quality of thawed food largely depends on the ice morphology during freezing. Ice morphology refers to the size and shape of ice crystals, and it is directly related to freezing rates (Petzold, 2009). The rate at which a fruit freezes affects both the chemical and physiological aspects of the food, including quality, texture, and nutritional and sensory properties (ASHRAE, 1997). In industry, there are multiple methods for freezing food, but most freezing methods may be organized in two categories: slow freezing and fast/rapid freezing. The figure below shows the cellular effects of fast versus slow freezing in plant tissues.

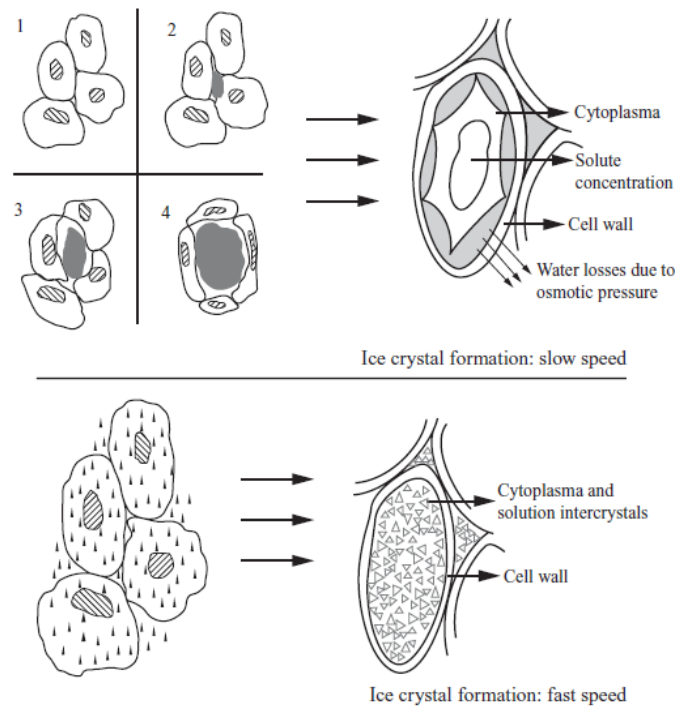


Figure 3: Slow vs Fast Freezing in Plant Tissue (Roy, 2017)

In slow freezing, the temperature is generally maintained between -8 and -20°C, and freezing penetration speed is relatively low. This causes ice crystals to grow in extracellular locations, which results in large crystal size, maximum dislocation of water due to osmotic pressure, and shrunken appearance of cells in the frozen state (Petzold, 2009). Additionally, due to poor nucleation and crystallization, less ice crystals are formed. Together, large crystal size and low number of crystals causes a degradation of texture and a loss of natural juices during thawing (ASHRAE, 1997).

In fast Freezing, the temperature is generally maintained between -30 to -50°C, and the freezing penetration speed is relatively high. Rapid cooling produces small ice crystals that form in both extracellular and intracellular locations, and thus there is no osmotic pressure to displace water outside the cells (Petzold, 2009). Furthermore, due to good nucleation and uniform crystallization, a larger number of ice crystals form. Fast freezing is usually preferable to slow freezing because it results in less damage to the fruit's cell walls, giving better shelf life and preservation of quality, as seen in Figure 4 below. (ASHRAE, 1997).

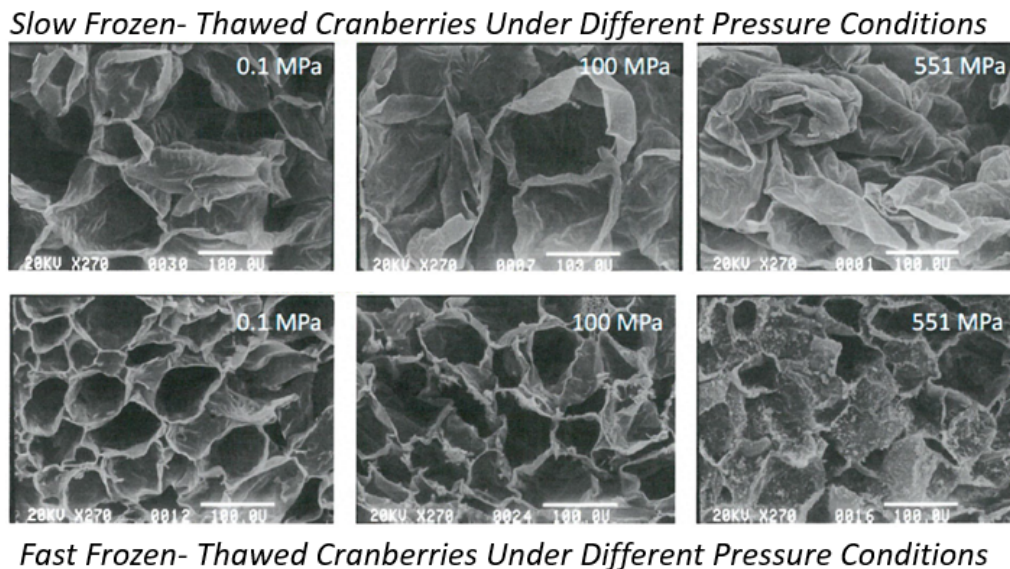


Figure 4: Slow vs Fast Freezing Rates Cranberry SEM Images (Mahadevan, 2014)

The SEM images in Figure 4 show greater cellular damage during slow freezing conditions as the cell walls seem to lose rigidity compared to the fast frozen cranberries. This effect is observed at all pressure conditions, however less cellular damage is seen at lower pressures.

1.3. Possible Effects of Freezing Rates in Ocean Spray's Craisin Process

For Ocean Spray's SDC products, some degree of cellular damage during freezing is favorable for manufacturing. The extraction step involves draining the sliced cranberries of their natural juices. Therefore, slow freezing conditions are favorable for extraction because they produce more cellular damage and yield greater juice loss during thawing.

The next step in the SDC manufacturing process is infusion which involves the absorption of syrup in the extracted cranberries. If there is too much cellular damage in the cranberries, the infusion process would be hindered because the fruit would not retain the syrup. As a result, the final product would experience syrup expression, or leakage. Therefore, it would be beneficial to determine the freezing conditions that yield enough cellular damage in the cranberries to enhance extraction and infusion, but to minimize syrup expression.

Chapter 2: Lab-Scale Process (Methodology)

This section describes the lab scale process for SDC manufacturing, equipment specifications, and unit operation protocols.

2.1. Overview of the Lab-Scale SDC Process

Our team attempted to implement a 300 gram-per-batch lab-scale SDC process in order to analyze the effects that different freezing conditions have on the properties of cranberries and the resultant SDCs. The lab scale process was a scaled down version of Ocean Spray's full scale manufacturing process, as referred to in Section 1.1.

2.2 Freezing

A Thermo Scientific™ Revco™ Ultra-Low Temperature Chest Freezer was used to freeze the cranberries. The Freezer has a storage volume of 12.7 ft³ and a temperature range of -10°C to -40°C. Photos of the freezer are shown below.



Figure 5: Photos of the Freezer

Initially, two Sterilite® plastic containers were half filled with cranberries (2,720 g) and placed in the freezer. One container was taken out when the freezer temperature reached -25°C, while the other remained until -40°C was reached. Holes were drilled into the plastic containers to allow for more air flow through the containers. The Sterilite® container is shown in Figure 6 below.



Figure 6: Sterilite® Container

In our experimental procedure, four different freezing conditions were specified. Two different temperatures were used, the lowest being -40°C and the highest being -25°C . Two different freezing rates were implemented. The fast rate involved setting the freezer set point directly to the final temperature. The slow rate involved decreasing the temperature 3°C per hour until the final temperature was reached.

2.3 Slicing

An Alligator® Slicer was used to slice the frozen berries to a 5 mm width. Frozen berries were placed in the cutting bed, and the slicer blades were pushed through the berries. Photos illustrating the cutting bed and blades are shown below.



Figure 7: Alligator® Slicer In Use

2.4 Extraction

Once the berries were sliced, they were placed in a two-gallon bucket. Water was added to the bucket at a 3:1 water-to-cranberry ratio by weight. A custom-built mixer was used to agitate the cranberries for 12 hours. The mixer used a 12 Volt stepper motor, a mixing shaft made from PVC, and Arduino boards for control. The mixer was coded to make one full rotation clockwise at 5 rpm, then a half rotation counterclockwise at 5 rpm, continuously. Photos of the mixer are shown below.

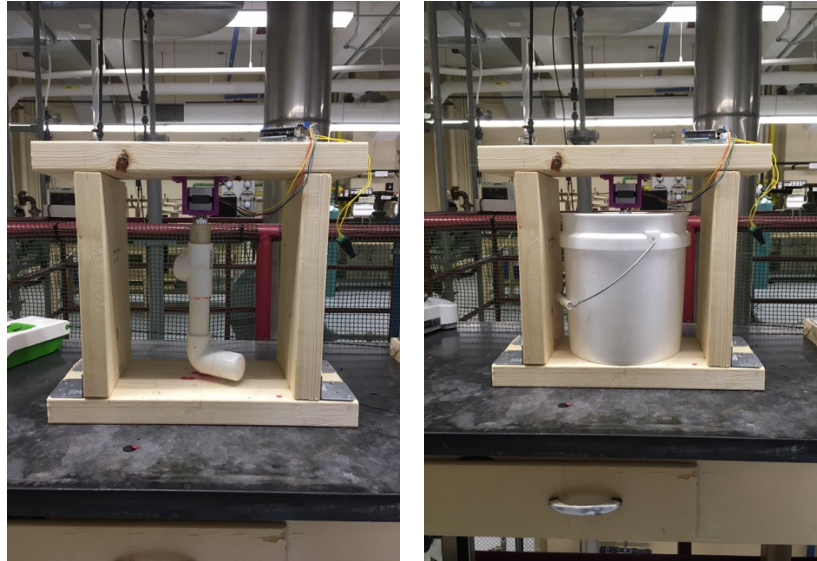


Figure 8: Custom Extraction Mixer

After 12 hours, soluble solids content of the slurry was measured using an Atago RX-5000 Digital Refractometer and was expressed as degree a Brix. Degree Brix is an indirect the measurement of soluble solids content in an aqueous solution. Although degree Brix accurately reflects the percent of sucrose content in pure sucrose solutions, the test provides a good estimation of total soluble solids in mixed solutions. The test was performed to ensure that the slurry was less than 1.5 Brix.(1.5% solids). To measure percent Brix, a cranberry water slurry with a 3:1 water-to-cranberry ratio by weight was blended for three minutes. Due to the ratio of the slurry, observed Brix (percent solids) Brix was multiplied by four to obtain the actual percent solids of the cranberries. A table outlining the extraction specifications is presented below.

Table 1: Extraction Specification Summary

Weight Ratio (Cranberries: Water)	Time (hours)	Percent Brix Requirement
3:1	12	<1.5% Br

2.5 Infusion

To begin infusion, the extracted cranberries were moved to a stainless-steel pot. Infusion syrup was then added at a 9:1 syrup-to-cranberry ratio by weight. Infusion syrup is a 68% Brix mixture mainly composed of juice concentrate, water, and sugar. The pot was then placed on a hot plate and heated to 120-130°F. A duplication of the custom mixer used for extraction was then used to continuously stir the infusing berries for 12 hours. Photos of the infusion set up are illustrated in the photos above.

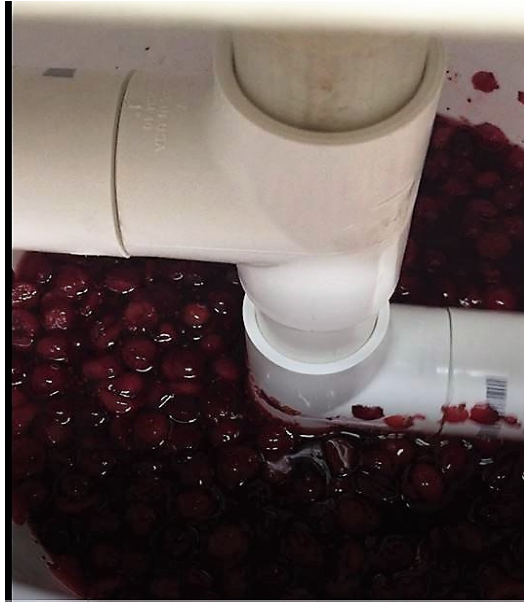


Figure 9: Syrup Infusion of Cranberries

Infusion was completed when a Brix test showed that the cranberry slurry reached 54-55% Brix. A table outlining the infusion specifications is presented below.

Table 2: Infusion Specification Summary

Weight Ratio (Cranberries: Water)	Time (hours)	Percent Brix Required (%)	Temperature (°F)
9:1	12	54-55%	120-130

2.6 Drying

A Magic Mill ® MFD-1010 Food Dehydrator was used to dry the infused cranberries. The dryer has a single fan located on its back face, is equipped with timed-drying capabilities, and can reach a maximum temperature of 167°F.



Figure 10: Dehydrator and Drying Rack

After infusion was complete, the infusion syrup was drained from the cranberries using a strainer over a sink. The infused cranberries were then spread out on a drying rack and placed in the Magic Mill ® Food Dehydrator for 20-24 hours at 167°F. Drying was complete when a Brix test showed that the cranberry slurry reached 78- 82% Brix. A table outlining the drying specifications is presented below.

Table 3: Drying Specification Summary

Time (hours)	Percent Brix Required (%)	Temperature (°F)
20-24	78-82%	167

2.7 Comparison of Full Scale and Lab Scale Processes

In the lab scale process, several factors differ from the full-scale SDC manufacturing process. Ocean Spray's full scale slicer cuts berries at 7 mm widths, while the blades of the slicer used in the lab scale process are 5 mm in width. In the extraction and infusion processes, Ocean Spray's counter current systems allow for more effective mass transfer, and thus require less time than the lab scale mixing system. Additionally, the full scale infusion system operates at a temperature approximately 30°F less than the lab scale process.

The drying configurations also differ between the full scale and lab scale processes. Where OceanSpray has 3 sections to the dryer, all operating at different temperatures, the lab scale process has a limitation to only operate at a single temperature that is approximately 80°F less than the full scale dryer. Consequently, the drying time required in the lab scale process is considerably higher than that of the full scale process. Since the variables of interest in this project were freezing temperatures and rates, the lab-scale process differences, including slice width, extraction temperature, and drying procedure, were considered to be negligible given that the value of these variables were held constant throughout the lab-scale experimental procedure.

Chapter 3: Data Acquisition, Evaluations, & Testing

It was necessary to acquire data throughout the lab scale process. The purpose of this section is to describe tests and test procedures.

3.1. Freezing Rate Data Acquisition

The data acquisition hardware used throughout the course of the project included two TMP1101_0 Thermocouple Phidgets, along with one HUB0000_0 VINT Hub Phidget, which was then connected to the lab computer. A LabVIEW program was used to collect and store the thermocouple readings.

To obtain diverse freezing data inside the container, thermocouples were strategically placed inside the two Sterilite® plastic containers. Each container had four thermocouples. Three thermocouples were placed in the bottom half of the container, inside cranberries. One in a lower corner, one in the middle, and one in an upper corner. The fourth thermocouple was suspended in the middle of the empty top half of the container to log the ambient temperature inside the container. A schematic illustrating the placement of the thermocouples placed inside berries is presented below in Figure 11. The ambient thermocouples are not represented. Two containers were stacked on top of each other inside the freezer.

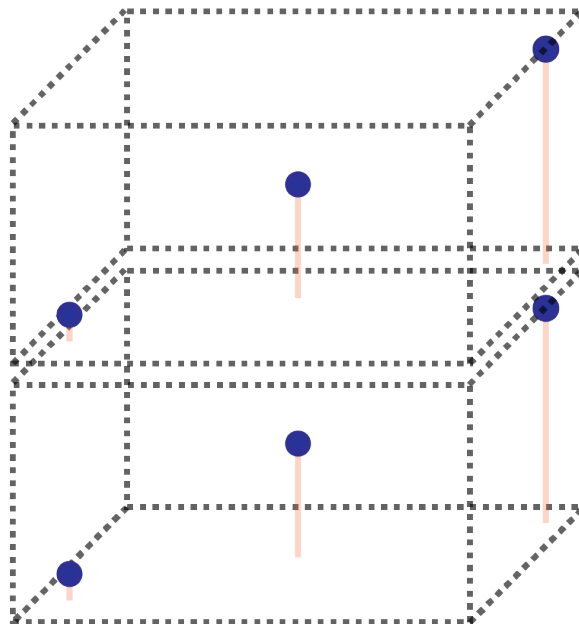


Figure 11: Schematic of Thermocouple Placement Inside the Sterilite® Containers

3.2. Number & Size of Ice Crystals

To prepare SEM samples for visualizing cellular damage, cranberry samples must first be lyophilized. A benchtop freeze dryer, BenchTop Pro with Omnitronics, was used to dry the samples for 24 hours at a pressure of around 50 mT and temperature of around -100 °C. Photos of the lyophilizing equipment and lyophilized cranberries are presented in the photos.



Figure 12: Bench-top Freeze Dryer and Photos of Lyophilized Cranberries

After the berries were lyophilized, they were sliced using a handheld razor blade and sputtered with a Denton Desk II using a gold target. The samples were then imaged at 104 times magnification. Examples of images produced from the SEM are presented in the photos below.

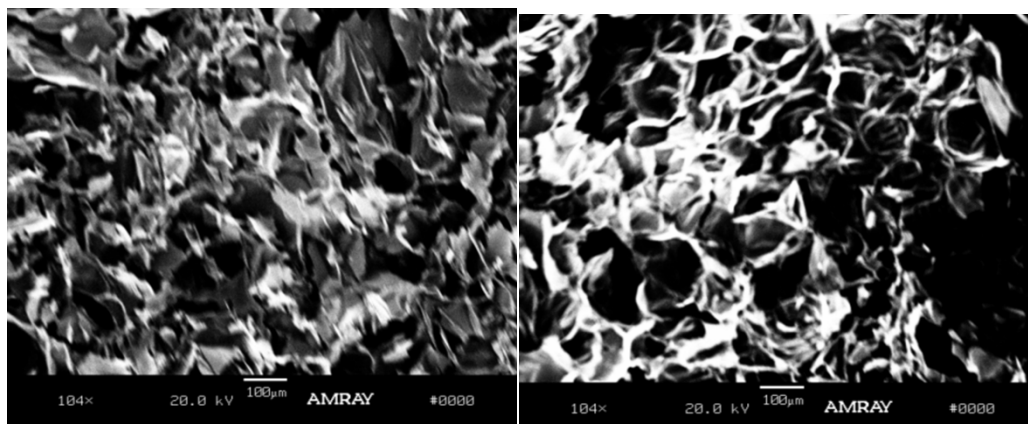


Figure 13: Example SEM Images of Lyophilized Cranberries at 104x Magnification

Chapter 4: Process Development

Due to challenges encountered in the lab scale freezing process, the goal of the project shifted to establish uniform freezing conditions for all berries inside the freezer. This section focuses on the group's efforts to achieving a uniform freezing environment through freezer profiling, the use of a monolayer rack system, and the introduction of a small fan.

4.1 First Run Challenges

The initial freezing method using Sterilite® containers produced non-uniform freezing curves, as seen in Figure 14, below. The temperature inside three berries was recorded: one located in a bottom corner of the container (green line), one in the middle (red line), and one in the top, opposite corner of the container (blue line).

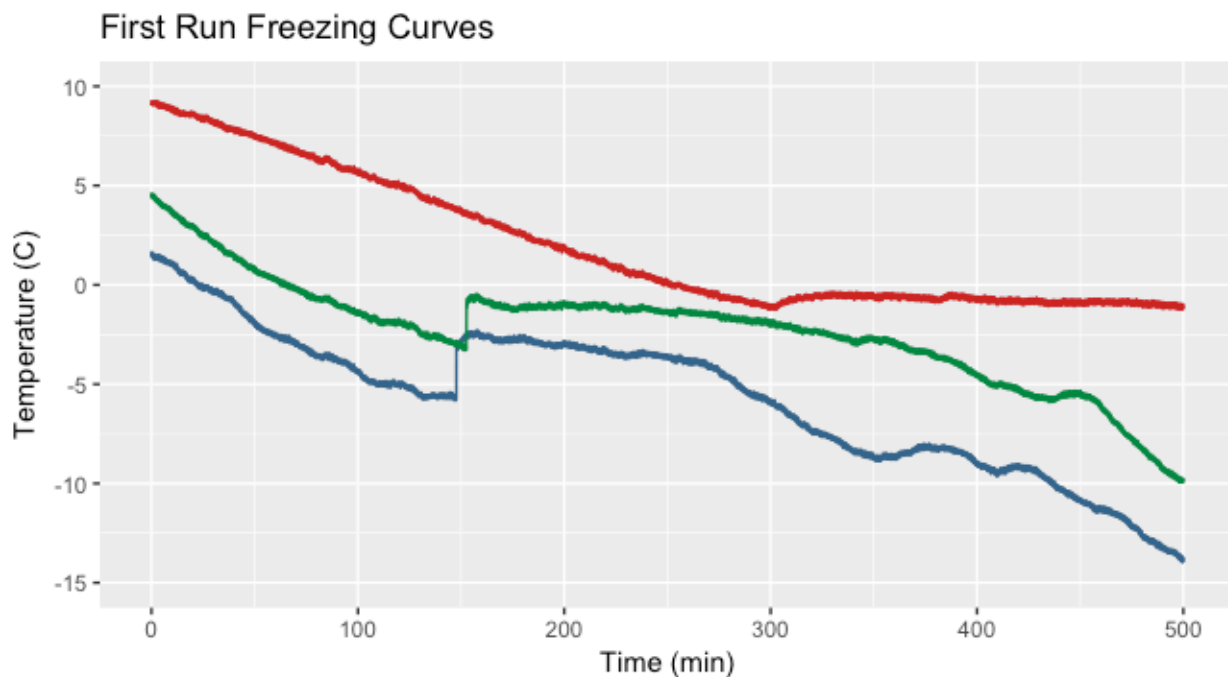


Figure 14: Freezing curve of cranberries frozen at a rate of -3C per hour from -10C to -40C

The main issue with this method is that it did not produce uniform freezing conditions for all berries in the batch, as indicated by the freezing curve of the berry located in the middle of the container. The surrounding cranberries insulated and limited heat transfer to the middle berry. Consequently, the berries in the middle of the container remained above freezing temperature until the fourth hour of freezing, and only achieved a minimum temperature of approximately -1°C.

Additionally, there are no visible spikes in the freezing curve for the middle cranberry, making it unclear whether a phase change occurred within the berry.

Due to the inconsistency of the freezing environment of each berry within a batch, it would not be possible to attribute any of the effects on the final product to the freezing treatments. If the cranberries do not undergo the same freezing treatment, the effects on the lab scale process and final product may be the result of some of the berries being underfrozen or overfrozen. Therefore, the correlation between the specific freezing condition and its effects could not be drawn, and no conclusion could be formulated. The team hence decided to switch the project's focus to developing uniform and replicable freezing conditions for cranberries exposed to different freezing treatments. Once this is achieved, future groups can carry out the full lab-scale SDC process to effectively study the effects of freezing on the final SDC products.

4.2 Freezer Profiling

The first step taken to produce a uniform freezing environment was to profile the freezer in order to identify cold and warm sections. To do this, thermocouples were used in two different profiling orientations. The first orientation had one thermocouple in each corner of the freezer, while the second orientation had one thermocouple at the center of each face, and one in the center of the freezer. A schematic illustrating these orientations is presented in Figure 15 below.

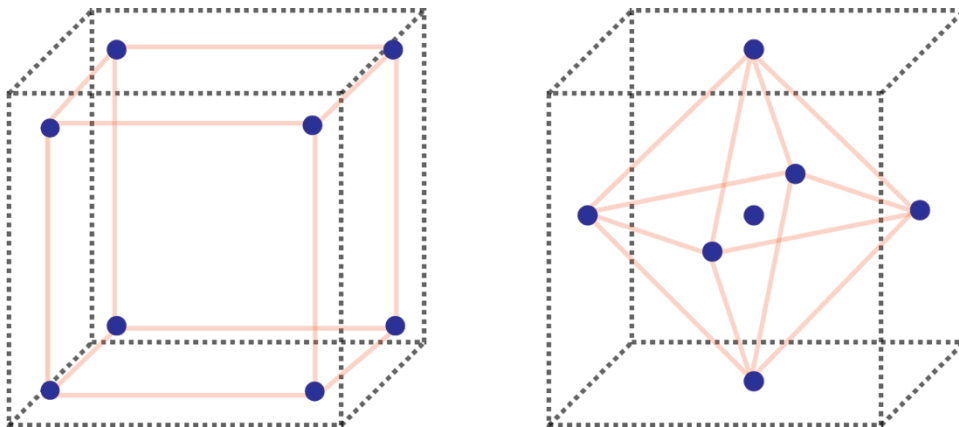


Figure 15: Schematics of thermocouple location for temperature profiling of the freezer

For each profiling run, the freezer was first brought to -10°C and allowed to equilibrate. The LabVIEW program was then started, and data acquisition began. After 2-5 minutes of initial data collection, the temperature set-point on the freezer was changed to -40°C . Data was then collected for the next 60 minutes, and the LabVIEW program was stopped. The resulting temperature profile graphs are presented in Figure 16 below.

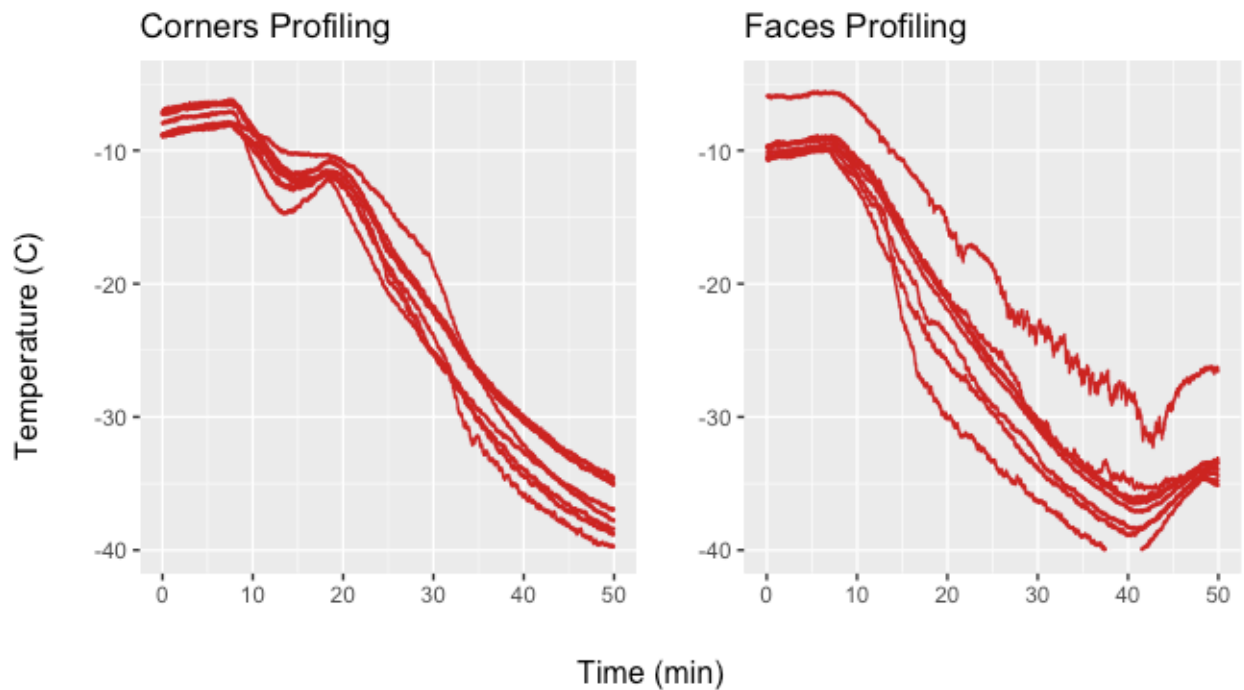


Figure 16: Freezer Profiling (Corners, Faces)

The two freezer temperature profilings show that the top of the freezer is warmer than the bottom of the freezer by approximately $2\text{-}3^{\circ}\text{C}$. It can also be seen, from the shape and distribution of the curves, that temperature is more uniform at the corners of the freezer than at the faces and center. Therefore, placing the berries at the corners of the freezer would expose them to a more uniform freezing environment. However, this would be impractical and space inefficient. In order to create a uniform freezing environment throughout the entire freezer, a mini fan was introduced.

4.3 Introduction of a Mini Fan to the Freezer

Because of the non-uniform conditions inside the freezer, a Holmes Mini High Velocity Personal 4 inch diameter fan was placed at the bottom of the freezer, facing directly upwards. This fan, theoretically, would improve the air circulation inside the freezer and create a more uniform freezing environment.

The fan was placed in the bottom of the freezer because cold air is denser than hot air. Therefore, the fan forces the cold air upwards to mix with the warmer air. The continuous flow of cold air to the top of the freezer allows for a uniform temperature distribution. Figure 17 below illustrates the temperature profilings of the freezer in the corners, faces, and center of the freezer with the addition of the mini fan. This figure, compared to the freezer profilings without the fan, shows that a more uniform distribution was achieved.

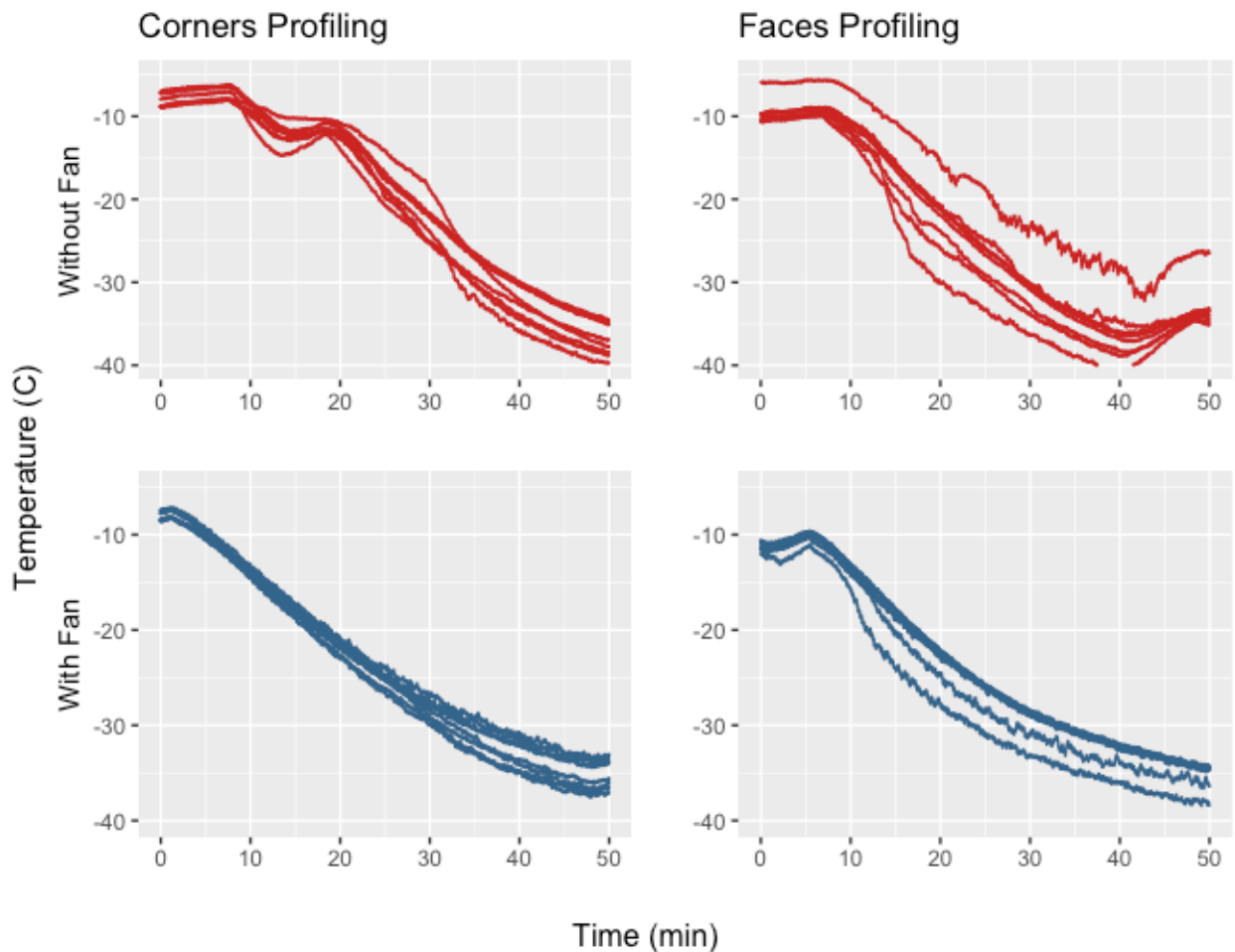


Figure 17: Freezer Profiling with Mini-Fan Comparison (Corners, Faces)

4.4 Freezer Rack System

In order to further promote a more uniform freezing environment for the cranberries, a monolayer rack system was built. With the addition of the rack system, the surface area of the cranberries exposed to the air in the freezer was increased, and insulation due to berry-to-berry interaction was minimized.

The rack was 15" x 15" in length and width. The rack had three subsidiary layers, with heights of 6 in, 10.75 in, and 15.5 in. Six thermocouples were used to monitor the temperature of the cranberries throughout the freezing process. On the bottom rack, thermocouples were placed in berries in the back, left and front, right corners. In the middle rack, they were placed in the front, left and back, right corners. On the top rack, they were placed in the back, left and front, right corners.

Two thermocouples were used to monitor the ambient temperature inside the freezer. These were placed in the front, left and back, right corners of the bottom and top racks, respectively. A schematic of the rack thermocouple placement is presented in Figure 18, below. Thermocouples measuring the ambient temperature are not represented.

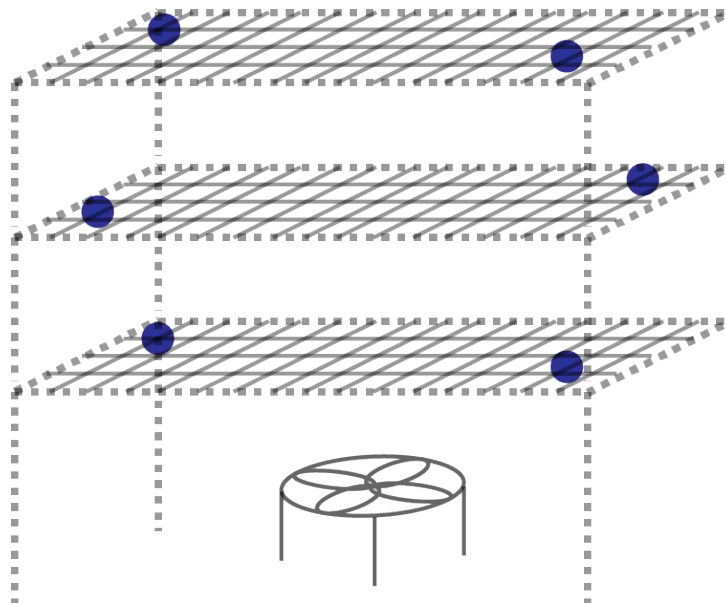


Figure 18: Rack System Schematic with Thermocouple Placement

4.5 Uniform Freezing Conditions and Methodology

There were four conditions tested in the uniform freezing experimental procedure, which mimic the previous conditions in the lab scale process. Out of the various parameters, freezing temperature and freezing rate were chosen to be tested. Two different temperatures were used, the lowest being -40°C and the highest being -25°C . Two different freezing rates were implemented. The fast rate involved setting the freezer set point directly to the final temperature. The slow rate involved decreasing the temperature 3°C per hour until the final temperature was reached.

Each uniform freezing experimental run was performed in the following manner. First, the freezer temperature set-point was set to -10°C , and the freezer temperature was allowed to equilibrate. The rack was then deconstructed into its three layers, and one bag of cranberries were randomly spread across each rack. A circular space was cleared in the middle of each layer, approximately 5 inches in diameter to allow for proper air flow from the mini fan. The mini fan was then placed in the center of the bottom of the freezer, facing directly upwards. Each rack was then placed in the freezer one at a time, and thermocouples were placed accordingly. Once all racks were placed in the freezer, the freezer door was closed and the LabVIEW program was started. Depending on the rate of the run, the freezer temperature set-point was adjusted.

Chapter 5: Uniform Freezing Results

Four different freezing treatments, with three replicates each, were studied in this project. There were four variables tested, including two temperature set points, -40°C and -25°C , and two freezing rates, fast and slow. Fast freezing involved setting the freezer directly to the final temperature set point, while slow freezing involved decreasing the temperature at a rate of -3°C/hr . Once temperature uniformity was established across all freezer locations, variability between each freezing treatment was analyzed. The results of one of the runs for each treatment are shown in Figure 19 below. Refer to appendices for the results of all the runs.

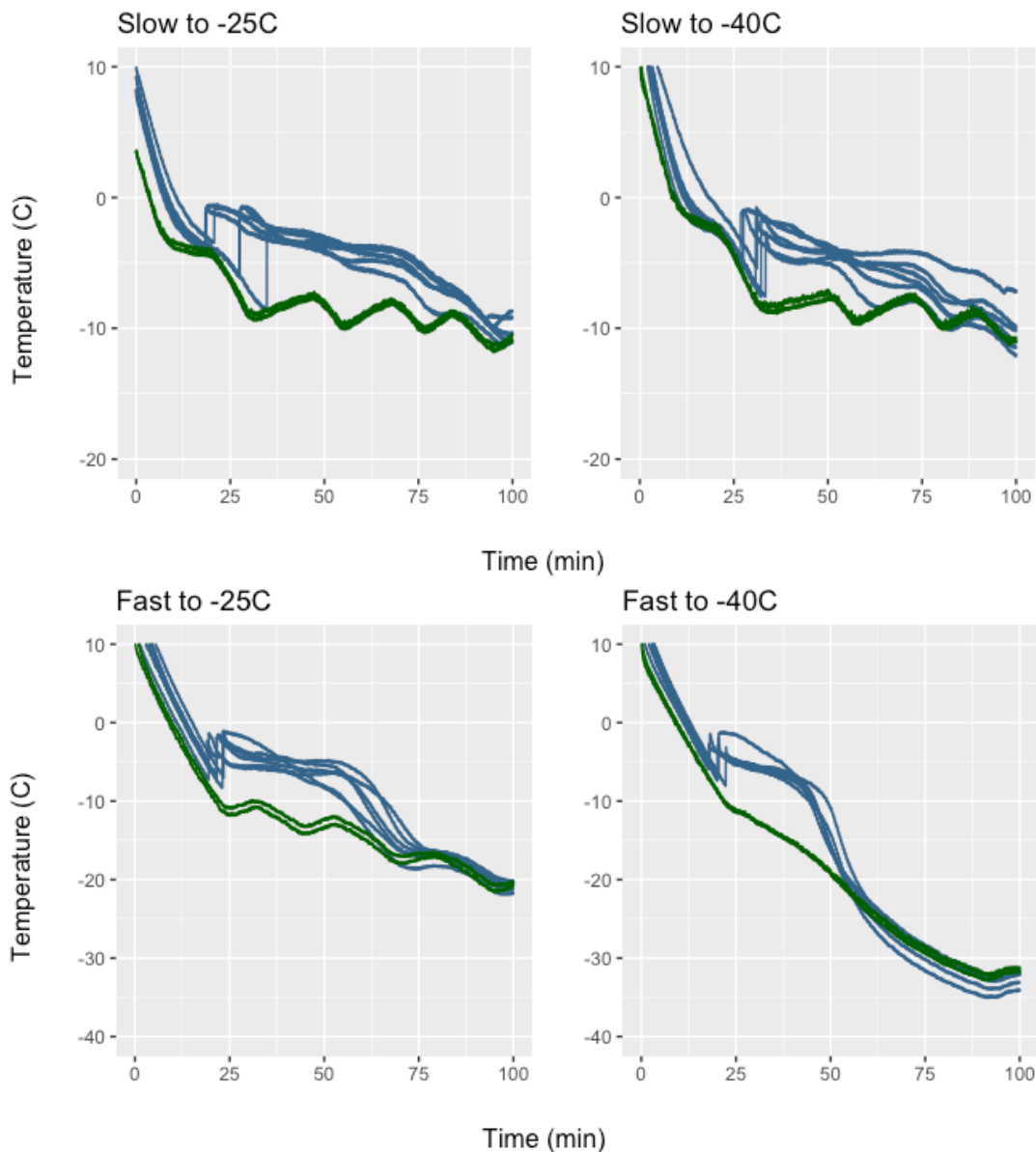


Figure 19: Freezing Curves for Cranberries Frozen to -40°C and -25°C at Fast and Slow Rates

Figure 19 shows the freezing curves for cranberries exposed to each freezing treatment using the uniform freezing method described in Section 4. The plots show that for both the slow runs, the phase change started between the first 20-30 minutes, while for both the fast runs, the phase change started before the first 20 minutes. Additionally, there is greater discrepancy in the initiation to phase change between the two slow runs than between the fast runs. The box plots in Figure 20 below better portrays the differences between the fast and slow treatments.

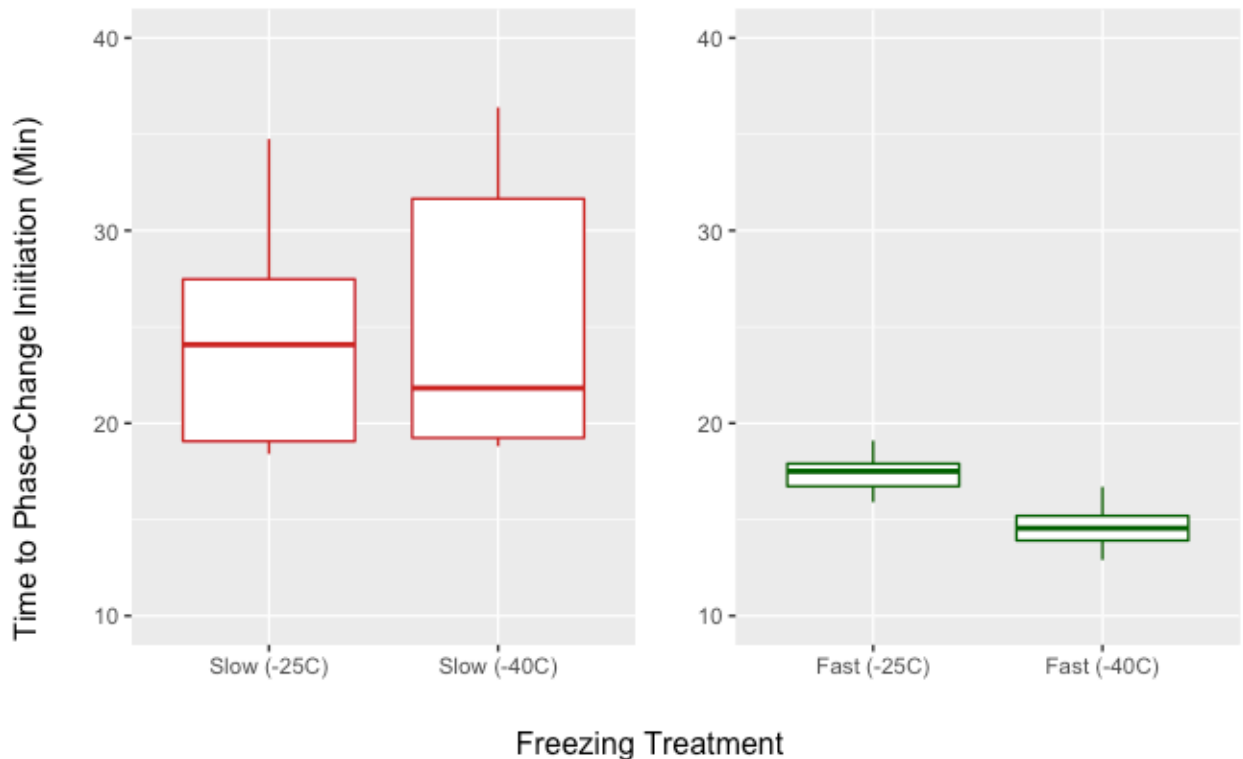


Figure 20: Box Plots for Cranberries Frozen to -40°C and -25°C at Fast and Slow Rates

As indicated by the box plots, slow freezing yields a much greater range in time to phase-change initiation compared to fast freezing. A possible explanation to this phenomenon could be attributed to a larger initial temperature difference between the cranberries and their freezing environment for the fast freezing rate. In fast freezing, temperature drops quickly, increasing the heat transfer driving force, decreasing the cranberries' temperature faster, and ultimately leading to faster nucleation and phase change. This is consistent with literature reviews that predict better nucleation and uniform crystallization for faster freezing rates. As a result, many but smaller ice crystals probably formed, both intracellularly and extracellularly, in the berries that were frozen at the fast rate. This prediction is supported by the complete freezing curve for cranberries frozen to -40°C at a fast rate, as seen in Figures 21 & 22 below.

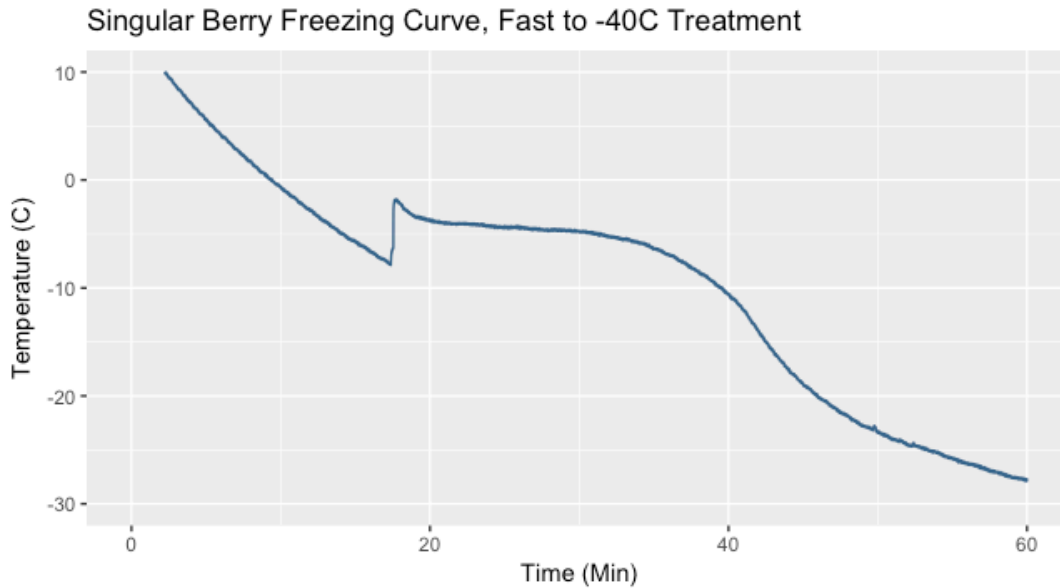


Figure 21: Freezing Curve for a Single Cranberry Frozen to -40°C at a Fast Rate

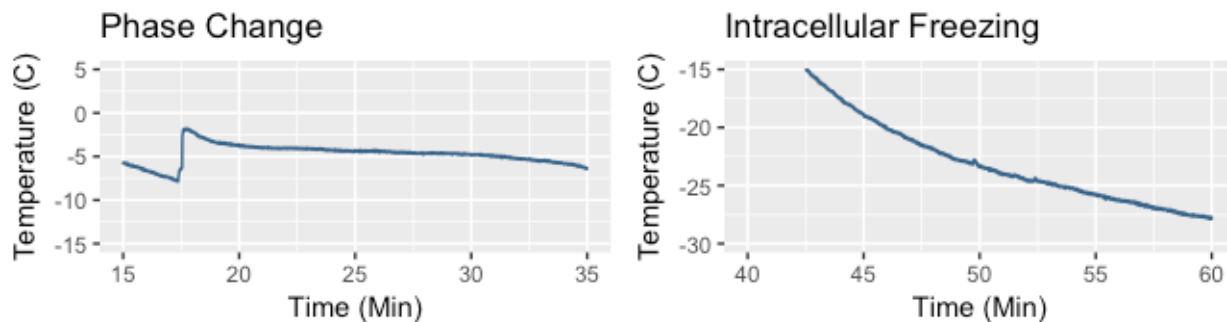


Figure 22: Magnification of the Initial Phase Change (Left) and Intracellular Freezing (Right)

As seen in Figures 21 & 22, the cranberry exposed to this treatment first experienced supercooling, then underwent extracellular freezing (as indicated by the jump and plateau in temperature), and finally experienced intracellular freezing (as indicated by the small temperature spikes at the end of the curve). This is consistent with the freezing curve presented in Section 1.2. No small temperature spikes were observed in any of the other treatments.

This is expected for the slow rate freezing treatments given that the freezing penetration speed is relatively low, and, thus, ice crystals tend to grow in extracellular locations. For the fast freezing treatment to -25°C, the temperature must have still been too high to allow for intracellular freezing. As a result, it can be predicted that the slower freezing treatments yield greater cellular damage to the cranberries, followed by the fast treatment to -25°C, and the fast treatment to -40°C yields the least amount of cellular damage. However, further testing must be done, including SEM imaging and thawing leakage tests, to prove the degree of damage to the cranberries' cell walls.

Chapter 6: Recommendations

The ultimate goal of this project is to determine how different freezing conditions affect both the cellular damage in frozen cranberries and the quality of the SDC final product. In order to do this, the design of a lab scale SDC process is necessary. The following section details our team's recommendations for future teams regarding all steps of the lab scale process design as well as testing and data acquisition.

6.1 Storing Refrigerated Berries

It is recommended to begin the project as close to the September - October harvest season as possible to ensure getting "fresh" cranberries in good condition. A project starting in the spring semester means the berries have already been stored refrigerated for at least three months. The managers at Morgan Hall's Pulse on Dining proved to be extremely helpful in offering refrigerated storage space for both cranberries and infusion syrup. Our team found it convenient to use a mini fridge to keep a small supply of cranberries and syrup in the lab. We would recommend obtaining a mid-sized mini fridge for this purpose.

6.2 Freezing

It is advised that future teams use the uniform freezing method, as described in Section 4.5. The LabVIEW code created should be used to collect and store temperature measurements throughout the freezing process. The code has been uploaded to the project's google drive.

Further research may also be conducted to analyze the effect of other variables, or factors, in the freezing of cranberries. These experiments can be planned out by increasing the amount of factors used for the Design of Experiments (DOE) in the Minitab software. Such factors may include cranberry size, color and firmness, and different freezing rates and temperatures.

6.3 Slicing

The Alligator slicer used was proficient in slicing the berries. A second slicer may be helpful in decreasing the required time for slicing and increasing the lab scale batch size.

6.4 Extraction/Infusion

Mixing is essential for the extraction and infusion processes. Without mixing, it would take over 24 hours to reach the target Brix percent for infusion and extraction, pushing back the progress of the project. However, mixing is challenging because it is imperative for mixers not to slice or damage the berries. Most commercial mixers have speed settings that go too fast, and/or blades

that are too sharp, which would end up damaging the berries. This is why our team built two custom mixers from PVC pipes using stepper motors powered by an Arduino board. However, the fastest these mixers could go without blowing out the motor was 5 rpm.

Our team recommends that future teams explore other mixer designs that are able to reach faster speeds. The PVC pipes proved to do a good job at preserving the quality of the berries. However, we recommend to explore the option of using a DC motor instead of a stepper motor to power the mixer. The DC motor can control the speed through applied voltage, and might be able to provide faster mixing speeds.

Our team found that leaving a batch of berries extracting overnight (for 12 hours), is enough to reach the target extraction percent Brix. The team also found that applying heat helps accelerate the infusion process. The team recommends, however, to find a hot plate that allows to set specific target temperatures. This way teams can better control the applied heat to the batch, and test out which temperatures achieves better infusion rates. However, the team recommends not to exceed 130°F during infusion to avoid boiling and burning of the batch.

6.5 Drying

The dryer used in the preliminary lab scale process design proved to be inefficient for drying for two main reasons. The first reason was that the maximum temperature of the dryer was not capable of reaching the temperature of Ocean Spray's full-scale dryer (over 200°F). Secondly, the dryer's air circulation was inefficient. Finding a dryer that can reach a higher temperature and has better air circulation, preferably that pushes hot air from below the berries, may prove to be more efficient in drying infused berries.

6.6 Testing/Experimentation

There are a variety of testing and experimentation procedures that may be used to both qualitatively and quantitatively assess the cellular damage experienced from freezing. These tests include lyophilizing/scanning electron microscopy, thawing leakage infusion rate, and syrup expression.

6.6.1 Lyophilizing and SEM

Lyophilizing is necessary in order to prep the cranberries for the SEM. Lyophilizers are located on the 4th floor of WPI's Gateway labs. Special access and safety training is required to enter the lab. In order to preserve the conditions of the cranberries before lyophilization, and ensure they don't thaw while being transported to Gateway, the berries were placed in a cooler filled with dry ice. Dry ice is available at the Goddard Stock Room.

Training and authorization is needed to use SEMs on campus. Doug White is in charge of the SEM located in Goddard Hall. Doug helped our team with SEM imaging, however the lyophilization was carried out by team before handing him the samples. His contact information is in the Points of Contact section below.

In order to speed up the SEM process, it might be convenient for future teams to get trained and authorized to use an SEM on campus. This way they do not have to bother, nor rely on, third parties who are already busy with their own work. There are SEMs located in Goddard Hall and Higgins Lab.

6.6.2 Thawing Leakage Test

To determine the effect of ice crystal formation on the cellular structure of the frozen cranberries, thawing leakage tests may be carried out. The initial procedure our team used is the following. 10 grams of whole frozen cranberries were removed from each batch and placed in funnels over graduated cylinders. The orientation of the cranberries, funnel, and graduated cylinder is displayed in Figure 23 below.



Figure 23: Orientation of the Thawing/Leakage Test

The cranberries were left in ambient conditions for one week. The cranberries were then removed from the funnel and the mass was recorded. The weight difference between the initial 10 grams and the mass after thawing was calculated and recorded. When this test was performed, no liquid was collected. Rather, liquid evaporated from the berries, leaving dried cranberries. In order to observe leakage from the cranberries, our team recommends adding pressure to berries in the form of weight.

6.6.3 Infusion Rate

Another way to test the effect of cellular damage from freezing on cranberries is to test how the different freezing treatment affect the cranberries' infusion rate. In order to do this, future teams should conduct test runs to determine what temperature and mixing parameters yield the best infusion conditions. Once these parameters are established, they should be held constant for each experimental run. The team should then closely monitor the infusion rate of each batch after each

freezing treatment to determine how they differ. In theory, the longer it takes for a batch to reach the target infusion percent Brix, under the same infusion conditions, the higher the degree of cellular damage. This is because more cellular damage makes it harder for berries to retain fluids.

Our team found that a Brix test should be carried out hourly after the first 10 hours of infusion, but more frequently as the batch approached the target percent Brix to avoid exceeding it. In order to read accurate percent Brix values, our team suggests to use the digital refractometer provided by OceanSpray.

6.6.4 Syrup Expression

To determine the effectiveness of the SDCs' retention of infusion syrup, a syrup expression test may be performed on a random sample of the final lab-scale SDCs. The following section describes the procedure used for this test.

Absorbent pads were first cut out using a circular cutting template and massed. The mass was recorded. The circular absorbent pad was then placed in a cylindrical plastic container. 15 grams of SDCs were then weighed and evenly distributed over the area of the circular absorbent pad inside the cylindrical container. Next, a 360g circular weight was placed on top of the SDCs in the cylindrical container. The container was then shut and allowed to sit for one week. After one week, the circular weight, cranberries, and absorbent pad was removed from the cylindrical container. All SDCs were removed from the absorbent pad, and the absorbent pad and SDCs were weighed and recorded.



Figure 24: Components and Set-Up of the Syrup Expression Test

6.7 Points of Contact

The following section outlines relevant people from Ocean Spray and WPI who may be of assistance in future project work. Their contact information is listed below.

Ocean Spray

Ryan Moriarty (Director of Engineering) - RMoriarty@oceanspray.com

Bill Russell (Lab Scale Process) - brussell@oceanspray.com

Guy Duarte (Cranberry/Syrup Pickup) - GDuarte@oceanspray.com

Steve Nojeim - SNojeim@oceanspray.com

Soumya Roy - SRoy@oceanspray.com

Alex Gorman - agorman@oceanspray.com

Stephen Peabody - speabody@oceanspray.com

Suneet Khanna - skhanna@oceanspray.com

Ellen Guilfoyle - equilfoyle@oceanspray.com

Andrea Kliora - akliora@oceanspray.com

WPI

Professor Stephen Kmiotek (ChE Advisor) - sjkmiotek@wpi.edu

Professor Sarah Jane Wodin-Schwartz (ME Advisor) - swodinschwartz@wpi.edu

Professor Andrew Teixeira (LabVIEW Expert) - arteixeira@wpi.edu

Tom Partington (Unit Operation Lab Manager) - tpartington@wpi.edu

Doug White (Unit Operation Lab Manager / SEM Technician) - douglas@wpi.edu

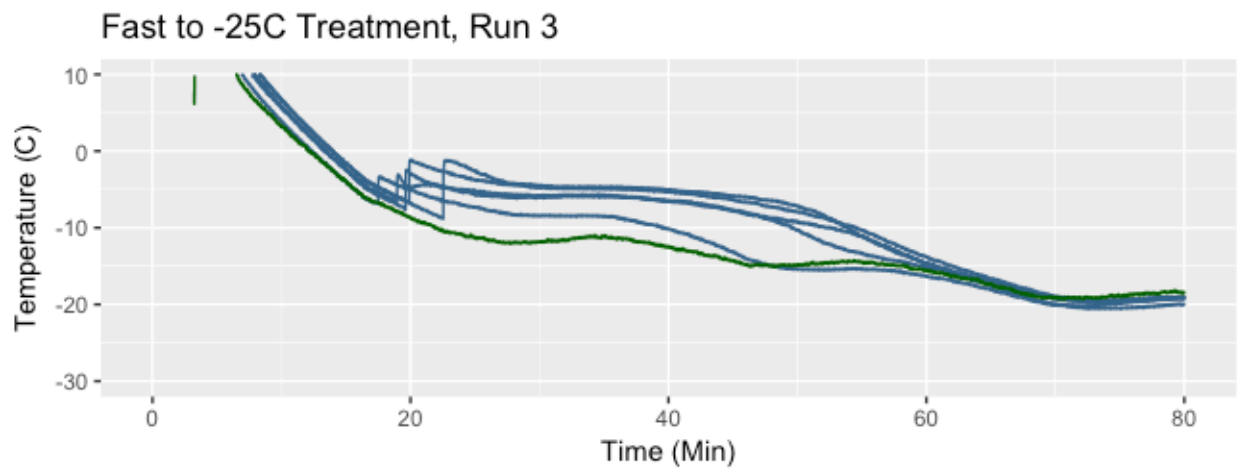
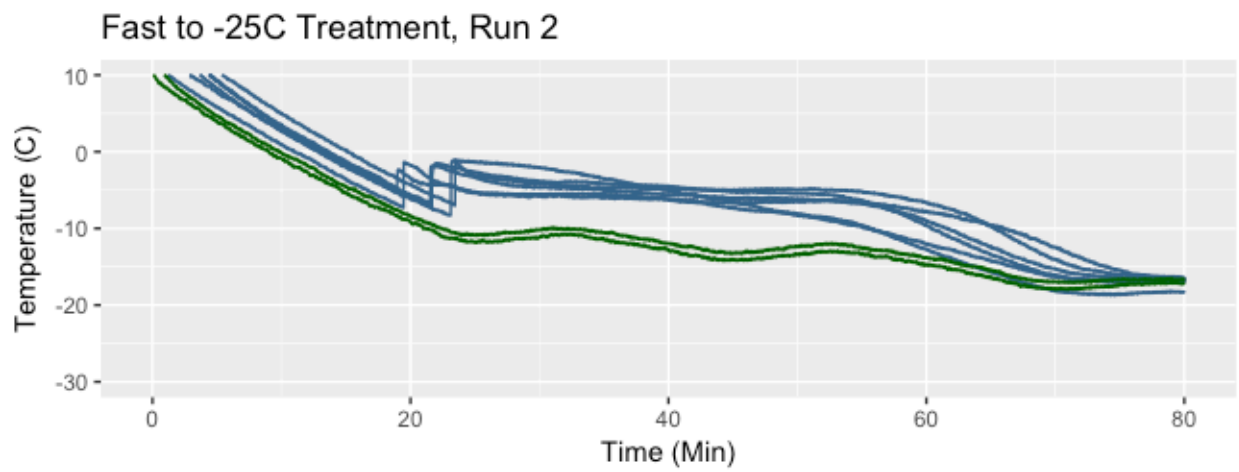
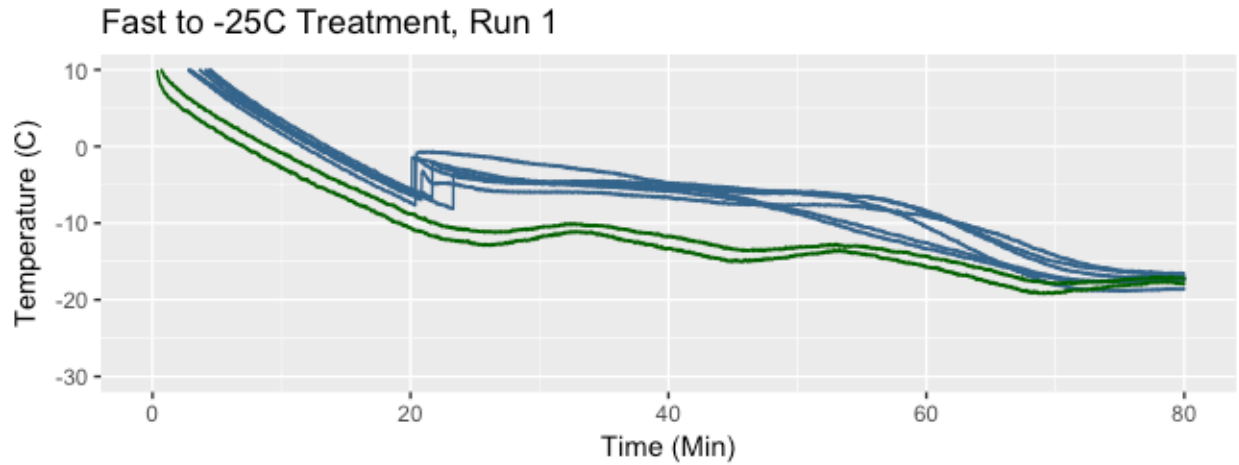
Daryl Johnson (Gateway Core Lab Technician/ Lyophilizer Contact) - drjohnson@wpi.edu

References

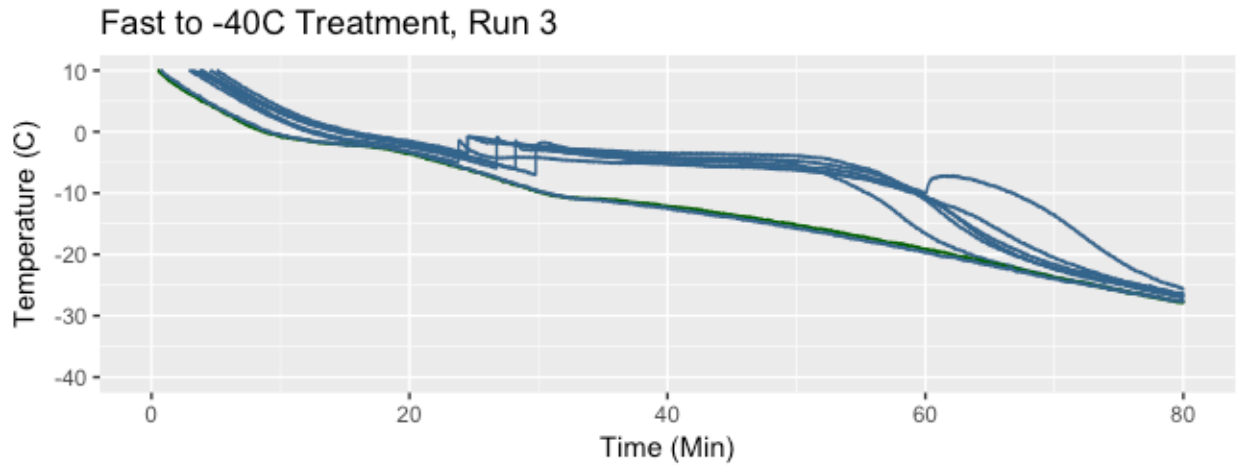
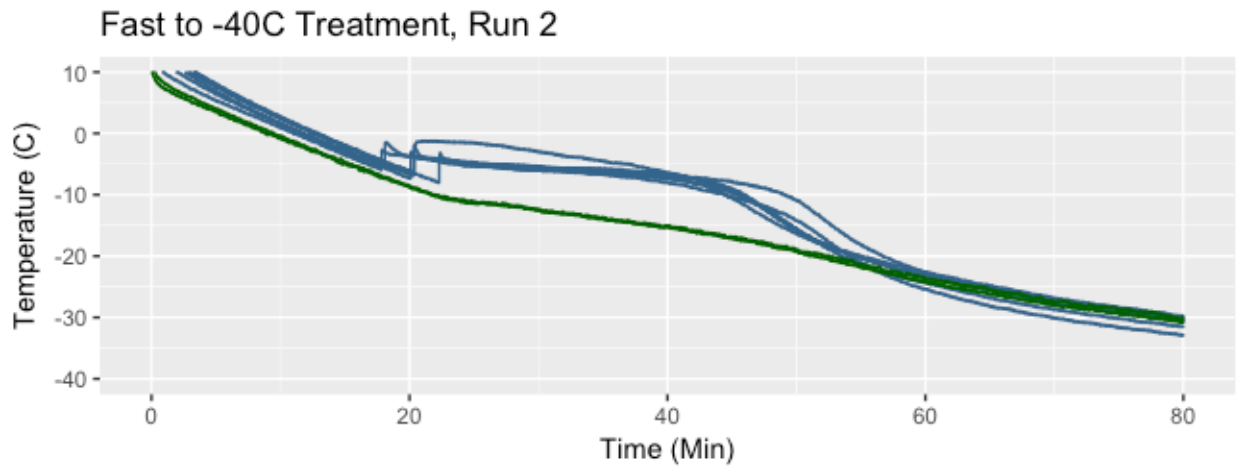
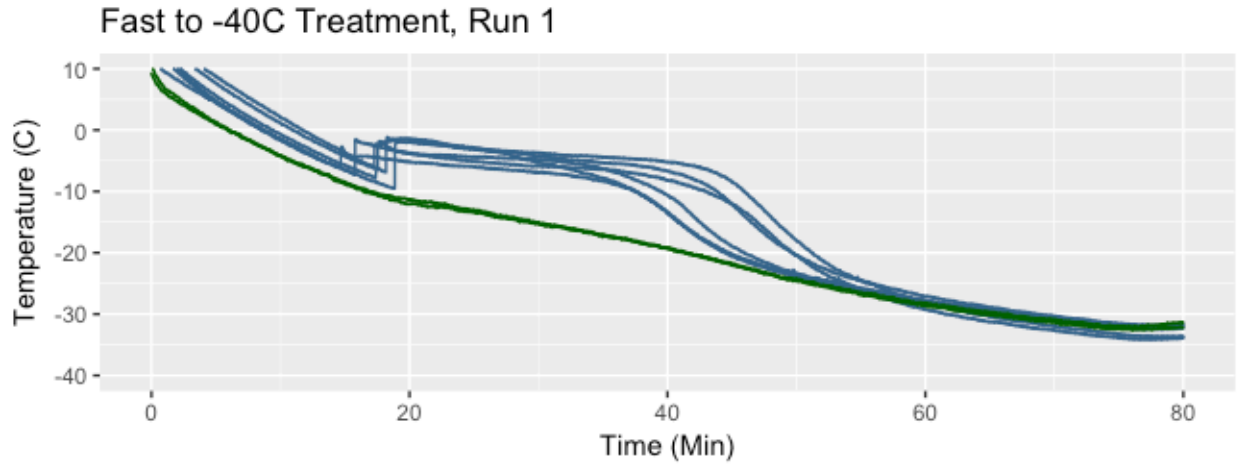
- Anderson, M. W., & Cubillas, P.(2010). *Synthesis Mechanism: Crystal Growth and Nucleation*. Weinheim: Wiley-VCH.
- ASHRAE, Handbook of Refrigeration. SI version. Atlanta, GA: American Society of Heating, Refrigerating, and Air-Conditioning Engineers, Inc., 1994.
- Mahadevan, S. , Salvi, D. and Karwe, M. V. (2016), High Pressure-Enhanced Infusion in Fresh and Frozen-Thawed Cranberries: A Comparative Study. *J Food Process Eng*, 39: 53-60.
- Petzold, G., & Aguilera, J. M. (2009). Ice Morphology: Fundamentals and Technological Applications in Foods. *Food Biophysics*, 4(4), 378-396. doi:10.1007/s11483-009-9136-5

Appendices

Appendix A: Fast Freezing Treatment (-25C) Data

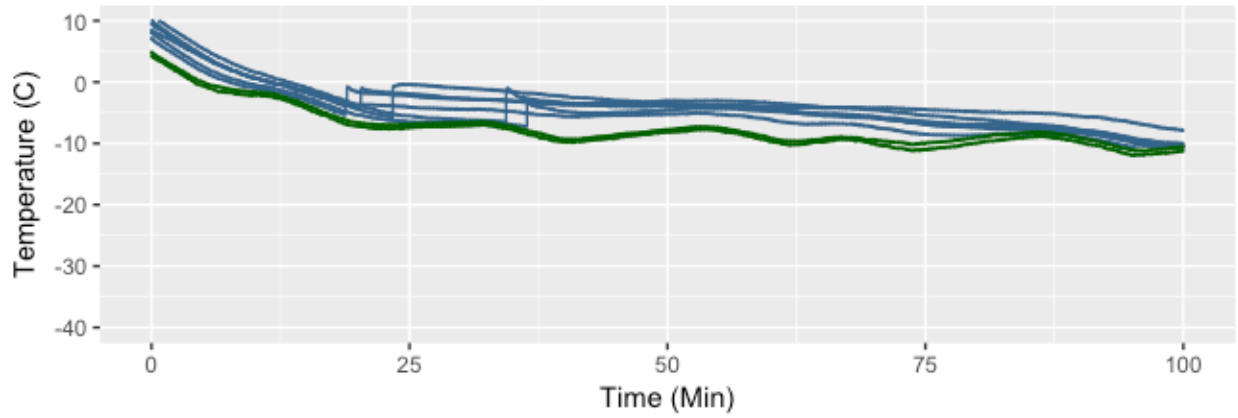


Appendix B: Fast Freezing Treatment (-40C) Data

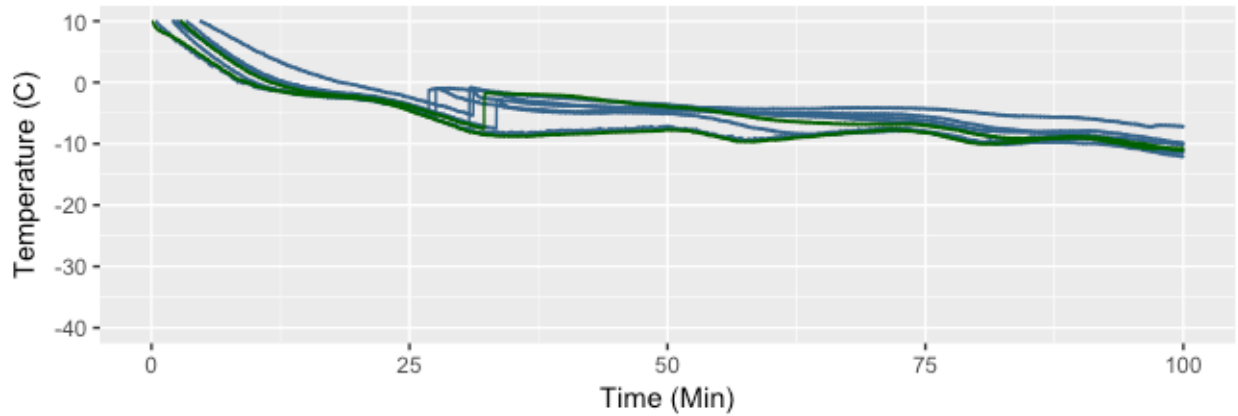


Appendix C: Slow Freezing Treatment (-40C) Data

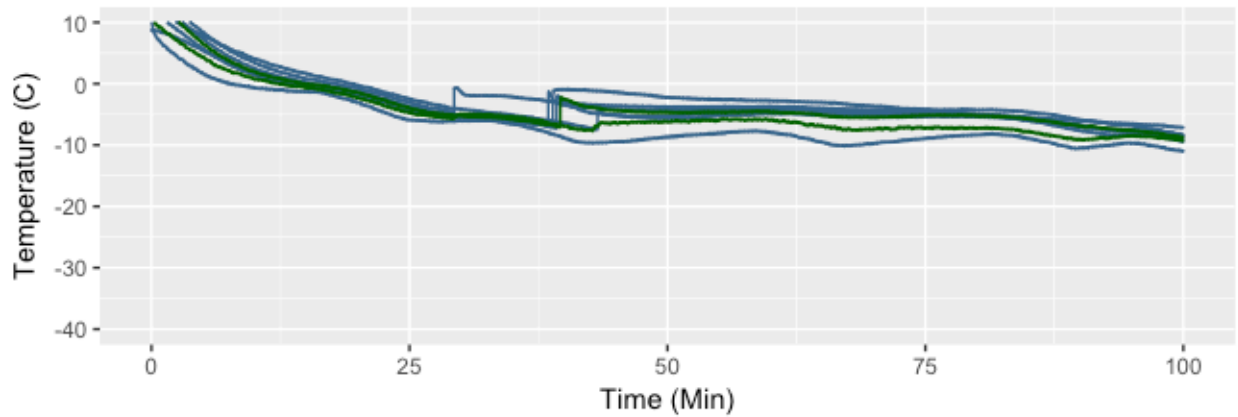
Slow to -40C Treatment, Run 1



Slow to -40C Treatment, Run 2

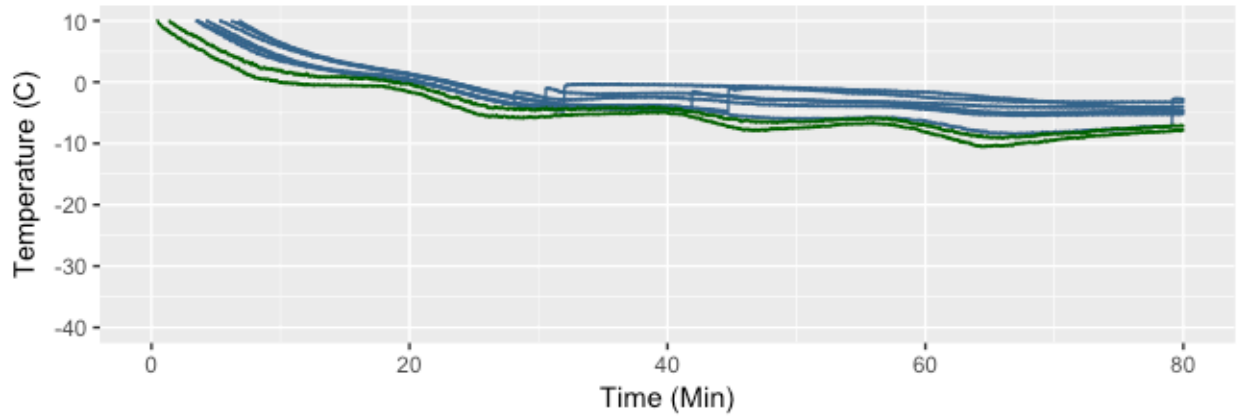


Slow to -40C Treatment, Run 3

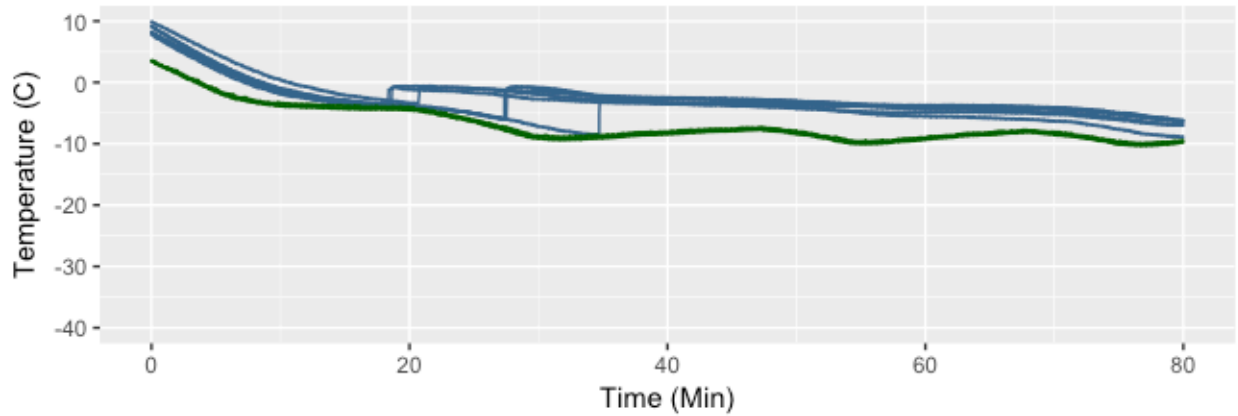


Appendix D: Slow Freezing Treatment (-25C) Data

Slow to -25C Treatment, Run 1



Slow to -25C Treatment, Run 2



Slow to -25C Treatment, Run 3

