Influence of Freezing and Thawing Methods on Textural Quality of Thawed Frozen Potato Slices

THESIS

Presented in Partial Fulfillment of the Requirements for the Degree Master of Science in the Graduate School of The Ohio State University

By

Anita Wickramasinghe, B.A.

Graduate Program in Food Science and Technology

The Ohio State University

2014

Master's Examination Committee:

Dr. Dennis Heldman, Advisor

Dr. Sheryl Barringer

Dr. Luis Rodriguez-Saona
Abstract

The relationships between freezing rate and frozen food quality have been demonstrated in both research and commercial operations. In most cases, these relationships have suggested that maintaining small ice crystals within the product structure during the freezing process leads to retention of structure and original texture. The scientific literature contains limited information on the relationships between freezing rate and product quality attributes. The objective of this investigation was to develop relationships between freezing rate and food texture for a typical food structure.

For this investigation, potato slices were frozen to -20°C using a dry ice ethanol bath, a conventional freezer or a blast freezer. Frozen slices were then thawed to ambient temperature using a microwave oven or ambient air conditions. Textural Profile Analysis parameters (TPA hardness, TPA cohesiveness, TPA springiness, TPA gumminess, TPA chewiness and TPA resilience) were measured over a range of freezing rates.

The results of this study demonstrated that TPA hardness of samples decreased as a function of time to freeze until the point of maximum damage, where the TPA hardness remained statistically constant. It also indicated that there was no significant difference in the TPA hardness of frozen potato slices after thawing in the microwave oven versus the TPA hardness of frozen potato slices after thawing at ambient temperature. Lastly,
the distribution of product texture within stacks of frozen potato slices was a function of the rate of freezing (P>0.05). The thaw methods (ambient air or microwave) and position in stack had no effect on the final TPA hardness.

These results also confirmed that the potato texture was primarily dependent on time to freeze and was not influenced by the thawing method.

This study provides useful insight into the relationships between textural quality and time to freeze for the frozen food industry. Understanding the tradeoffs between freezing rate and textural quality allows companies to better select an optimal strategy for their needs.
Acknowledgements

I would like to sincerely thank my advisor Dr. Dennis Heldman for all his support and guidance through this process. It has been an honor working with such a distinguished food scientist and I am extremely grateful for all I have learned from him through this process. I would also like to thank Dr. Barringer, without whom I would not have come to The Ohio State University. While I did not manage to take any of her classes during my program, her kind words and support were what drove my decision to join The Ohio State University program, and I am honored to have her on my committee. I would like to thank Dr. Rodriguez for being an excellent mentor in classes, product development and for being on my committee. His dedication and passion for teaching students has taught me so much.

I have thoroughly enjoyed all of the opportunities placed before me at The Ohio State University and am grateful for the wonderful faculty staff and students who have helped make this journey. I especially like to thank my lab mate Meugyuan for keeping me company in the lab nights and weekends, and for always having a bright outlook. I would also like to thank David Phinney and Cheryl Wicks for their advice and help through this process. Thank you to all my family and friends who have supported me, especially my parents and my sister.
Vita

2004 ..............................................B.A. Economics, Middlebury College

2014 ..................................................M.S. Food Science and Technology, The Ohio State University

Publications


Fields of Study

Major Field: Food Science & Technology
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Chapter 1: Introduction

In 2012, frozen foods were a $224 billion category globally with 72% of Americans eating frozen prepared foods (Symphony IRI, 2012; MarketReserach.com, 2011). Projections for this category are expected to reach $294 billion by 2019 (Food Product Design, 2014). Despite being an extremely prominent part of the food supply today, frozen foods had a rough start. Early frozen foods, facilitated by the emergence of the home freezer in the late 20th century, were extremely poor in quality as compared to the products experienced today. Upon cooking, the products were often mushy, tasteless, lacking nutrients and lacked proper consistency. Although the food industry was not aware of it at the time, many of these issues were a consequence of slow freezing rates, which resulted in large ice crystals, promoting cell wall damage resulting in drip loss and other reactions (Joslyn 1961). In the early 1920s, Clarence Birdseye came up with the idea to quick-freeze foods. Quick-freezing involved exposing food products to intensely cold refrigerant, which shortened the freeze time, yielding products more comparable to their fresh counterparts (Hilder 1930). Today, these same fundamental processes are still widely used to preserve the integrity of a food’s nutrients, microbial stability, as well as flavor and texture (Lemelson-MIT).

Frozen foods deliver important benefits to both the food industry and consumers. For the food industry, freezing preservation provides an outlet for crops, such as broccoli,
cauliflower, and Brussels sprouts, that are almost exclusively commercially processed, while other crops, such as potatoes, corn, and peas, depend on this technology for considerable portions of their market share (Joslyn 1961). Consumers, on the other hand, benefit from convenient and affordable food options that have limited quality damage in comparison to other processed food counterparts.

However, there are also downsides to the frozen food process. In terms of energy usage, several studies have found frozen foods to be more energy intensive than their fresh or canned counterparts (Hendrickson 1994). Although aluminum and steel can production are extremely energy intensive, the electricity needed to run freezers for long periods of time adds up to even higher energy demands (Hendrickson 1994). In fact, frozen food warehouses are known to be one of the largest consumers of energy in the food industry (Prakash and Singh 2008).

While there have been significant advancements in the field of freezing research, there are still major gaps in the scientific literature. Continued success and growth of the frozen food market depends on the delivery of high quality products. For frozen foods, texture is considered one of the most critical parameters contributing to quality that influence sales. The rate of freezing and the process of ice crystallization directly impacts product quality (Joslyn 1961, Hilder 1930). Slow freezing methods lead to the formation of large ice crystals, which can damage product quality. Rapid freezing methods, in comparison, lead to the formation of small ice crystals, which can help

Textural properties are related to the deformation, disintegration and the flow of food under force (Erickson and Hung 1997). For frozen food, textural hardness is one of the most commonly properties used to determine damage. This property can be measured objectively and correlated to sensory textural attributes by doing texture profile analysis (Erickson and Hung 1997). Currently, there is sizeable interest in the development of methods to predict and control the texture of frozen foods (Waldron and others 1997). This study provides the food industry insight into the relationship between texture and quality. Understanding the tradeoffs between freezing rate and textural quality allows companies to better select an optimal strategy for their needs.
Chapter 2: Objectives

The objective of this investigation was to quantify the impact of freezing and thawing on food textural quality for a typical food structure:

- To develop and test a protocol to determine the impact of the time to freeze on textural damage of potato over a wide range of times to freeze.
- To develop and test a protocol to compare the influence of thawing method on textural damage of frozen potato.
- To evaluate the impact of time to freeze within potato structure on the texture after thawing
- To present recommendations on freezing process changes needed to improve textural quality in frozen food
Chapter 3:  **Review of Literature**

Freezing food is touted as one of the best ways to preserve product quality (Fennema 1996; Li 2002). Lowering both the temperature and water activity contributes to longer shelf life by impeding microbiological growth and chemical reactions, without significant sensory or nutritional quality loss (Kyureghian G, Stratton J, Bianchini A and Albrecht J. 2010, Fellows 2000, Joslyn 1961).

### 3.1 The Mechanism of Freezing

The freezing process involves three main phases. In the first or “chilling” phase, sensible heat is removed, lowering the product’s temperature. During this phase, the product becomes super-cooled when the temperature is temporarily below the product freeze point without triggering crystallization. In the sec phase, the latent heat of crystallization is released, resulting in the formation of ice crystals until most of the water has turned to ice. During this phase, the temperature remains fairly constant. The temperature at which this occurs depends largely on the composition of the food, with total water content and water that can be separated as ice by freezing being most important. Less time in this phase results in more uniform ice formation (Joslyn 1961). Lastly, in the third phase, more sensible heat is removed from the product until it reaches a final storage temperature. Some ice formation occurs in this range, but much less than in the sec phase (Joslyn 1961).
3.2 Freezing Systems

In choosing a freezing system, it is important to consider the rate of freezing required; the product size, shape, and packaging requirements; whether the operation will be batch or continuous, the scale of production, the range of products to be processed and capital and operating costs (Fellows 2000). The general mechanism of freezing systems involves the evaporation and compression of refrigerant in a continuous cycle and the use of cooled air, cooled liquid or cooled surfaces to remove heat from products. This includes systems that use cold air with varied parameters for freezing, systems that freeze on a refrigerated surface, and cryogenic systems (Fellows 2000).

3.2.1 Cold Air Systems

In still air systems (still air freezer), the air temperature ranges from -20 to -30°C. Other than cyclic temperature fluctuations and scheduled defrost cycles, the temperature remains fairly constant. This results in low freezing rates (Fellows 2000). This type of system is commonly used for storage. With mid air movement systems (cold store freezers), the fans circulate cold air. This increases the uniformity of temperature distribution; however the heat transfer coefficients are still low. This method facilitates freezing carcasses, storing pre-frozen foods, and hardening ice cream (Fellows 2000).

With air blast systems (blast freezers), -30 to -40°C, air is circulated at a velocity of 1.5–6.0 m s−1. The air velocity significantly improves the surface heat transfer coefficient. This method is used for products such as peas. This is the most common method of freezing for the food industry. Then there are fluidized-bed systems. In these systems air
between -25 and -35°C is circulated at an even higher velocity, ranging from 2 - 6 m s\(^{-1}\), through a porous bed of food. This results in even higher heat transfer coefficients. This method is used for particulate foods such as corn kernels, and berries (Fellows 2000).

### 3.2.2 Cooled Surface Systems

In cooled surface systems, refrigerant is pumped directly under the surface that comes in contact with the product. Product can be placed between refrigerated plates (plate freezer) or liquids or semi-solid foods such as ice cream are scraped off of refrigerated surfaces (scraped-surface freezers) (Fellows 2000).

### 3.2.3 Cryogenic Systems

In cryogenic systems, refrigerant comes in direct contact with the product. Cryogenic systems use solid or liquid carbon dioxide, or liquid nitrogen. These liquefied gases have low boiling points allowing for faster freezing than most conventional methods. This close contact with the food leads to high heat transfer coefficients and rapid freezing (Fellows 2000). Liquid nitrogen boils at a temperature of -196°C and is commonly used for fish fillets, seafood, fruits and berries. Liquid carbon dioxide is -70°C when released into the atmosphere. It is used in a variety of freeze systems as well as a pre-treatment for a liquid nitrogen spray (Barbosa-Cánovas and others 2005).

### 3.3 Microwaved Food Market and Technology

The introduction of the microwave led to another milestone for the frozen food industry, with a new high in sales for frozen meals. Initially invented in 1945, microwave usage
only became wide spread in the late 1970s and 80s. Microwaves provided a new level of convenience, with food that could be prepared in record time (NFRA 2009). It also provided a solution to households with two working parents, where there was less time and more cooking illiteracy or unwillingness to cook (NFRA 2009; Ryynänen S. 2002). Today, the biggest category within frozen food is the $6.1 billion industry of Frozen Dinners/Entrees (MarketResearch.com 2011; NFRA 2009). Consumer research shows that microwave ovens are the most widely owned appliance (Mintel 2009). The modern fast paced lifestyle has resulted in growing demand for rapid and “ready-to-eat” meals, for which microwave ovens fit the bill. Microwave ovens are considered an essential appliance in most kitchens today, recognized for their quick, easy and energy efficient ability to heat frozen foods (Barbosa-Cánovas and others 2005). There is also the benefit of decreased nutritional loss in comparison to conventional heating methods (The Harvard Medical School 2008).

Simultaneously, the food industry is the largest consumer of microwave energy (Ayappa and others 1992; Venkatesh and Raghavan 2004). While in homes, microwaves are touted for their ability to save time, and achieve a higher level of automation, in industry microwaves serve to accelerate processes, improve quality, reduce costs and increase yield (Datta and Rakesh 2013). Some of the other applications for microwave heating include drying, cooking, pasteurization, and preservation of food (Chandrasekaran and others 2013).
3.4 The Mechanism of Microwave Heating

Microwaves operate using electromagnetic waves with frequencies ranging from 300 MHz to 300GHz. Microwave heating affects polar molecules in a material causing alternating electromagnetic field energy to be transformed into thermal energy (Vadivambal and Jayas 2008). One of the most important characteristics of microwave heating is that it is a direct method of heating that generates volumetric heat. Volumetric heat generation allows material to heat up much more rapidly than conventional methods. In conventional methods, heat diffuses in from the surface of a material by convection followed by conduction. With microwave heating, the whole material absorbs microwave energy and converts it into heat from within the product. Since thermal damage to a material is directly proportional to time and temperature, faster processing times accomplished with volumetric microwave heating can be important to quality preservation (Vadivambal and Jayas 2008).

When microwaves penetrate food, some of the waves are absorbed and converted into heat. The degree and rate of heating is dependent largely on the dielectric and ionic properties of a product. Dielectric energy, a form of electromagnetic energy, causes molecular friction in molecules, which produce heat, whereas ionic properties of a product are related to the types of bonds present in a substance. Dielectric properties determine how a material interacts with electromagnetic waves (Soproni and others 2008). The dielectric properties include relative dielectric constant ($\varepsilon'$), relative dielectric loss constant ($\varepsilon''$) and the loss tangent ($\delta$) (Singh and Heldman 2009). The relative dielectric constant ($\varepsilon'$) indicates the ability of a material to store electrical energy, the
dielectric loss constant (ε’’) indicates the ability of a material to absorb microwaves and dissipate energy into heat, and the loss tangent indicates how well the microwave field is converted to heat (Balasubramaniam 2013).

Microwave properties of foods are dependent on temperature change, which means that changes in temperature during the microwave process cause dynamic changes in microwave absorbance. Some of these changes can be determined using Maxwell’s equation and Lambert’s law. Maxwell’s equation determines the propagation of microwave radiation in a dielectric medium. Lambert’s law is based on the exponential decay of microwave absorption within a product (Chandrasekaran and others 2013). However, often more complicated mathematical equations and modeling are used when studying a specific food.

3.5 Preserving Food Quality

For food, texture is one of the three major acceptability factors along with appearance and flavor. When it fails to meet consumer expectations, a product may not get eaten and will not likely get purchased again. While the mouth predominantly senses texture, some textural notes are perceived by other parts of the body, such as the hands. Texture is a key quality characteristic in almost all foods, and even more so in bland foods that rely on characteristics of crispiness or crunchiness for their acceptance (Bourne 2005). The International Organization for Standardization defines texture as “all the mechanical, geometrical and surface attributes of a food product perceptible by means of mechanical, tactile, and, where appropriate, visual and auditory receptors” (Bourne 2005).
The mechanical structure of food governs the textural properties perceived in biting and chewing. This structure is the biggest determinant of its behavior during processing. At the same time, preservation or manipulation of this structure depends on the processing parameters (Raeuber and Nikolaus 1980).

Sometimes textural modifications during food processing are deliberate. A chief goal in processing is altering the structure of food to make it more acceptable to the consumer. For example, whole grains can be ground into flour and made into bread, which is softer and easier to chew than straight whole grains. However, when textural modifications during processing are not deliberate, they can be highly undesirable. For example in canning, processing can cause undesirable softening of food materials. While there is no universal “ideal” product texture, there are some textures that are generally favored and others that are less favored. Some of the generally favored ones include firmness, crispness, creaminess, juiciness, tenderness, crunchiness, and chewiness. Whereas, some of the less favored ones include greasiness, sliminess, hardness, dryness, coarseness, crumbliness, stickiness, gumminess, and mealiness (Bourne 2005). While hardness is not considered favorable for all products, with frozen produce, it can be a favorable parameter corresponding to a lack of structural cellular damage.

3.6 Frozen Food Quality

While freezing is touted as one of the best ways to preserve food, freezing can also have detrimental effects on food quality. Physical disruption of cells, cell components and macromolecule structure induced during freezing can affect the water-holding capability
and textural quality (Alvarez and others 2005). Both freezing and microwave processing introduce factors that can affect the end product quality. For the freezing process, many of these issues relate back to ice crystal formation, and for the microwave process, many of these issues relate to uneven heating.

3.6.1 Ice Crystal Formation

The role of structure in the development of texture cannot be overstressed (Raeuber and Nikolaus 1980; Bourne 2005). For plant-based foods, texture depends highly on the physical and chemical properties of the cell wall (Waldron and others 1997). Most fruits and vegetables are between 74-96% water (University of Kentucky College of Agriculture 1997). This water is contained within rigid cell walls, which provide support structure and texture. During freezing, ice crystals can rupture cell walls, resulting in textural damage (Schafer and Munson 2012). Clarence Birdseye was one of the first people to observe this relationship when he coined the individual quick freeze process (Hilder 1930).

When water freezes, it expands 9% in volume, forming ice crystals that vary both in size and quantity depending on the conditions (Foster 2011). Previous literature establishes an accepted link between freeze rate and quality. Slow freezing leads to the formation of large ice crystals that can damage cell walls, having an adverse effect on product quality. In comparison, rapid freezing (up to a point), results in the formation of small ice crystals that are less likely to break through cell walls, resulting in better end product quality (Joslyn 1961). On a microscopic level, during the freezing process, water within plant
tissue gradually fills intercellular space eventually forcing cells apart. Large enough ice crystals will rupture cell walls (Joslyn 1961). On a macroscopic level, the breakage of cell walls results in drip loss and mushy texture. Undesirable quality changes can be attributed to concentration of solutes, dehydration, and mechanical factors (Joslyn 1961).

### 3.6.2 Freeze Concentration and Dehydration

Ice crystals are composed of virtually pure water regardless of the solution from which it is formed. So, as the ice crystals grow in size, the remaining solution becomes more and more concentrated (Moridon and Hartel 2007). Chemical reactions can still occur among the soluble solids within the concentrated solution. These concentrated solutions will also then draw water out of the cell leading to dehydration (Meryman 1960).

### 3.6.3 Chemical Changes during Freezing

Fruits and vegetables continue to undergo numerous chemical changes due to enzymatic activity after harvest (Schafer and Munson 2012). Blanching is an accepted practice used to inactivate enzymes prior to freezing. Briefly exposing produce to boiling water inactivates the enzymes, after which products must be cooled rapidly in ice water to prevent cooking. In addition, the blanching process can remove some surface microorganisms and help prevent loss of vitamin C (Clemson 2011).

### 3.6.4 Undesirable Mechanical Changes During Freezing

There are also many undesirable mechanical changes occurring during the freeze process. As mentioned above, in slow-freeze methods, after thawing natural moisture can drain...
out from damaged or broken cells (Hilder 1930). These issues as less prevalent in rapid-freeze methods where the ice crystals formed are smaller, and thus less likely to tear cell wall tissue causing textural damage. However, in very rapid freeze methods, such as cryogenic freezing, irreversible mechanical damage called freeze cracking can occur (Buggenhout and others 2006, Kim and Hung 1994). Freeze cracking is a phenomenon where products may crack or shatter at very high freezing rates as a result on internal stress (Hung 1997, Kim and Hung 1994).

3.6.5 Run-Away Heating

One of the biggest challenges with microwaving frozen foods is localized overheating, known as run-away heating (Balasubramaniam 2013). The non-uniform distribution of hot and cold spots not only lead to potential quality issues, but can also raise an issue of food safety when microorganisms are not destroyed in the cold spots (Vadivambal and Jayas 2008, Chandrasekaran and others 2013). Uneven heating can be due to several factors. Some of the challenges directly associated with the microwave oven are product placement, oven size and geometry, use of turntables, oven power, use of power cycling, use of mode stirrer, and effect of feed location. Some of the factors that influence even microwave heating are the dielectric properties of the material being heated, the power output of the system, the shape and density of the material, and the food composition (Singh and Heldman 2009). Dielectric properties have arguably the most significant effect (Soproni and others 2008).
The speed of heating dielectric material is directly proportional to the power output of the system, which is dictated by the power outlet (Singh and Heldman 2009). In the microwave heating of frozen foods, the effect of the dielectric properties is exceptionally significant because of the large difference in values for ice and water. The dielectric constant ($\varepsilon'$) of water is 80, in comparison to the dielectric constant ($\varepsilon'$) of ice is 3, and the relative dielectric loss constant ($\varepsilon''$) of water is 10, in comparison to the relative dielectric loss constant ($\varepsilon''$) of ice is 0.003. So, when the ice begins to melt to water, the water components heat at a much more rapid rate than the remaining ice components, thus leading to runaway heating (Singh and Heldman 2009).

The shape and density of material is also important in maintaining uniformity in heating. Non-uniform shapes and sharp edges and corners can cause non-uniform heating (Singh and Heldman 2009). Lastly, composition is important in how food material heats in the microwave field. Properties such as moisture content, salt content, and air pockets can all influence the distribution of heat within a product (Singh and Heldman 2009).

### 3.7 Texture Analysis

While the complete texture of food can be complex, specific textural characteristics can be evaluated with standard texture analysis instruments. The food industry uses the TA.XT2 Texture Analyzer as one of the main instruments to evaluate a wide range of products (Phadungath 2002). The Texture Profile Analysis (TPA) test was designed to imitate the chewing action of the mouth. For this test, a sample with a fixed size and shape is placed on the instrument’s base then compressed and decompressed twice using
an appropriate probe. The parameter values required to perform this test are collected and graphed. The result is a force-time curve (Figure 3.1) that objectively determines textural parameters that have been correlated to corresponding textural sensory attributes (Table 3.1)

Figure 3.1 Texture Profile Analysis Curve Obtained from TA. XT2 Texture Analyzer (Source: Texture Technologies Corp 2003)
TABLE 3.1 DEFINITIONS OF STANDARD TPA TERMS (SOURCE: TEXTURE TECHNOLOGIES CORP 2003)

<table>
<thead>
<tr>
<th>Standard terms</th>
<th>Definitions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hardness</td>
<td>The hardness value is the peak force of the first compression of the product. The hardness need not occur at the point of deepest compression, although it typically does for most products.</td>
</tr>
<tr>
<td>Fracturability</td>
<td>The fracturability point occurs where the plot has its first significant peak (where the force falls off) during the probe’s first compression of the product.</td>
</tr>
<tr>
<td>Cohesiveness</td>
<td>Cohesiveness is how well the product withstands a second deformation relative to how it behaved under the first deformation, measured as the area of work during the second compression divided by the area of work during the first compression. (Refer to Area 2/Area 1 in Figure 5)</td>
</tr>
<tr>
<td>Springiness</td>
<td>Springiness is how well a product physically springs back after it has been deformed during the first compression. The springback is measured at the downstroke of the second compression.</td>
</tr>
<tr>
<td>Chewiness</td>
<td>Chewiness only applies for solid products and is calculated as Gumminess X Springiness (which is Length 1/Length 2).</td>
</tr>
<tr>
<td>Gumminess</td>
<td>Gumminess only applies to semi-solid products and is Hardness X Cohesiveness (which is Area 2/Area 1).</td>
</tr>
<tr>
<td>Resilience</td>
<td>Resilience is how well a product &quot;fights to regain its original position&quot;. The calculation is the area during the withdrawal of the first compression, divided by the area of the first compression (Area 5/Area 4 on the Figure 5).</td>
</tr>
</tbody>
</table>

For frozen foods, hardness is most closely correlated to cell wall damage. Popular nomenclature for the textural parameter of Hardness is Soft→Firm→Hard (Szczesniak 1963). Boyd and Sherman (1975) provide a thorough explanation of the oral sensory evaluation of hardness by panelists (Table 3.2).
<table>
<thead>
<tr>
<th>Mechanism of oral evaluation of hardness by panelists</th>
</tr>
</thead>
<tbody>
<tr>
<td>Soft</td>
</tr>
<tr>
<td>Resistance offered to teeth</td>
</tr>
<tr>
<td>Type of breakdown in mouth</td>
</tr>
<tr>
<td>Shape retention</td>
</tr>
<tr>
<td>Actions involved in sensory evaluation</td>
</tr>
<tr>
<td>Stimuli associated with sensory evaluation</td>
</tr>
<tr>
<td></td>
</tr>
</tbody>
</table>

3.8 Opportunities to Solve Quality Issues During Freezing

There are many opportunities to address quality issues of frozen foods. Some of these opportunities involve improvements in the freezing process, while others may involve improvements in the thawing process.

3.8.1 High Pressure Shift Freezing

In the standard freezing process, there is a volume increase of around 9-13%, dependent on the amount of water in the product and temperature at which the product is frozen. In standard ice formation, this expansion results in freezing damage. This expansion can damage product quality by damaging the tissues. Under high-pressure (>200Mpa), low density ice is formed, which does not expand during ice formation. These lower density ice crystals formed are different shapes than the standard hexagonal ice crystals, allowing them to yield a more compact ice. As a result, instantaneous and homogeneous ice
crystals are formed across the product (Li and Sun 2002). Numerous studies have been published, illustrating the benefit of high pressure freezing on texture and structure of plant food products such as carrots, strawberries, tofu, mangos, eggplants, and peaches (Fuchigami and others 1997, Buggenhout and others 2006, Sun and Lee 2003).

3.8.2 Jet Impingement Cooling

Jet impingement is a cooling method that involves cooling air jets that are directed on a product at high velocities to enhance heat transfer (Dirita and others 2007; Datta and Rakesh 2013). This technology improves the heat transfer from solid and compact foods leading to reduced process times and improved food quality (Dirita and others 2007).

3.8.3 Dehydrofreezing

Dehydrofreezing is an alternative freezing method especially advantageous to high moisture products. High moisture foods, such as fresh produce, are exceptionally susceptible to the formation of large ice crystals during freezing. Dehydrofreezing, overcomes this obstacle by dehydrating food to a desirable moisture level prior to freezing. This process generates smaller, more uniform ice crystals, resulting in improved frozen food quality. This also reduces the refrigeration load since less energy is needed to bring the temperature down below freezing. Additional benefits include lower packaging costs, distribution and storage, and maintenance of product quality in comparison to the conventional method. Taste panel results also indicated that dehydrofrozen foods were comparable in consumer acceptance to their conventionally frozen counterparts (Li and Sun 2002).
3.8.4 Antifreeze Protein Additives

Natural antifreeze protein additives, found in fish native to polar and northern coastal waters, have been shown to depress the freezing points of these fish so they can successfully inhabit these areas. Similar antifreeze proteins have also been found in insects, plants, bacteria and fungi. These proteins have become a source of experimentation in manipulating the freezing process of foods. In these experiments, these natural antifreeze proteins have been added in order to lower freezing temperatures and suppress the growth of ice nuclei.

There are potential uses of this for ice cream, where it is very undesirable to have recrystallization. The fine ice crystal structures, characteristic of smooth and creamy ice cream texture are often altered as a result to temperature fluctuations in storage or in transmit, yielding diminished quality. In 1992 Warren, Mueller, and Mckow patented the process of adding antifreeze proteins to foods. In this process, foods are initially frozen well below their freezing point and then they can be stored longer term at higher temperatures without experiencing adverse effects of increased ice crystal growth.

This process might also have applications in the meat industry where large ice crystals can result in drip and loss of nutrients during thawing. Studies where meat was soaked in a solution containing antifreeze proteins prior to freezing, demonstrated evidence of reduced ice crystal size. In another study, lambs were injected intravenously with antifreeze proteins from Antarctic Cod prior to slaughter, which also resulted in smaller overall ice crystal size when frozen (Li and Sun 2002).
Currently antifreeze proteins are mostly being used in research or special uses due to prohibitive costs. Future chemical synthesis or genetic modification of these proteins could serve as a feasible solution to extend usage in the future (Li and Sun 2002).

### 3.8.5 Nucleation Protein Additives

Nucleation protein additives are proteins that serve to elevate the temperature of ice nucleation. These ice nucleation proteins are known as Ina+ (Li and Sun 2002). They have potential use for frozen foods with their ability to elevate the temperature of ice nucleation, shorten freezing times, and alter product texture, resulting in decreased energy cost and improved quality. Research has shown that these proteins can effect the formation of ice patterns introducing new product textures such as large and long ice crystals with a flake like texture and harder textures that are easily fractured (Li and Sun 2002). Such textures can be desirable and can be use to manipulate sensory qualities of a food product.

The one major concern with using these bacteria is that they must not introduce any food safety concerns. It is imperative that they are safe, non-toxic and non-pathogenic. Any inedible bacteria added to a product, must be completely destroyed prior to consumption (Li and Sun 2002).

### 3.8.6 Acoustic Thawing

Acoustic thawing is a novel thawing method that uses sound energy. Brody and Antenecich first investigated it in 1959, but negative aspects of poor penetration,
localized heating and high power requirements hindered its success. More recently, this method was revisited and demonstrated a more promising outcome when using a frequency in the relaxation range of ice crystals. Acoustic thawing methods exhibited higher thawing rates than using standard conduction methods. In 1981 experiments using blocks of cod, acoustically assisted water immersion thawing required 71% less time than samples solely using water immersion thawing. For these experiments 1500 Hz acoustic energy at 60 watts was applied. In 1999, more experiments were conducted using high power ultrasound to thaw meat and fish. This work illustrated acceptable ultrasonic thawing at frequencies around 500 kHz. Although this is done mostly for research applications as of now, these results show a promising method of thawing for food industry (Li and Sun 2002).

3.8.7 Ultrasound for Improved Crystallization

Deora and others (2013) discuss a method in which ultrasound energy is used to control the nucleation of the crystallization process in frozen food called sonocrystallisation. The main mechanism of action for this process is cavitation, the formation of vapor cavities. This cavitation causes nucleation to occur at higher temperatures or in shorter times, resulting in smaller and more uniform ice crystal formation (Deora and others 2013). Sun and Li (2002) similarly used this sonocrystallisation technique on potato samples. They observed that potato samples that had ultrasound applied for 2 min at power levels of 15.85 and 25.89W while the samples were decreasing in temperature from -1 to 7°C, exhibited significant (P<0.05) improvement in their freezing rate, as compared to the untreated samples (Sun and Li 2002).
3.9 Opportunities to Solve Quality Issues in Microwave Heating

Some of methods to improve uneven microwave heating include the use of a turntable, power cycling, microwave specific packaging, product formulation and use of a mode stirrer (Datta and Rakesh 2013). Many of these technologies are already present in commercial microwaves today.

3.9.1 Commercial Microwaves

In home microwaves, a greater knowledge of the microwave oven power functions is one of the simplest methods for an average person to improve the quality of their frozen food experience. The power function of a microwave allows for a lot of flexibility in microwave oven usage. While often overlooked, adjusting the power setting can prevent overcooking, overheating, and burning. Most microwave oven power settings are directly proportional to the frequency and duration of the magnetron cycle over time. In other words, the magnetron tube cycles on and off in pulses in order to obtain a range of power levels (General Electric 2014). The default setting is generally the highest power where the magnetron tube constantly generates microwaves 100% of the time (General Electric 2014). Most consumers stick to this setting. While the highest power setting will result in food heating up the most quickly, this is not always the ideal setting. In Table 3.3, General Electric describes some of the best uses for the power settings.
**Table 3.3 Microwave Power Settings (Adapted from General Electric 2014)**

<table>
<thead>
<tr>
<th>Level 10 – High</th>
<th>Best for browning, boiling liquids, cooking or heating fish, ground meats, bacon and zapping foods</th>
<th>100% on time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Level 9</td>
<td></td>
<td>90% on time</td>
</tr>
<tr>
<td>Level 8</td>
<td></td>
<td>80% on time</td>
</tr>
<tr>
<td>Level 7 – Medium High</td>
<td>Best for roasting meats and poultry, baking casseroles and convenience foods, sauté and re-heating food</td>
<td>70% on time</td>
</tr>
<tr>
<td>Level 6</td>
<td></td>
<td>60% on time</td>
</tr>
<tr>
<td>Level 5 – Medium</td>
<td>Best for slow-cooking and braise food</td>
<td>50% on time</td>
</tr>
<tr>
<td>Level 4</td>
<td></td>
<td>40% on time</td>
</tr>
<tr>
<td>Level 3 – Low</td>
<td>Best for defrosting, simmering, delicate sauces</td>
<td>30% defrost</td>
</tr>
<tr>
<td>Level 2</td>
<td></td>
<td>20% defrost</td>
</tr>
<tr>
<td>Level 1 – Warm</td>
<td>Best for heating breads, keeping food warm for a short period of time, soften butter, cheese and chocolate</td>
<td>10% warm</td>
</tr>
</tbody>
</table>

One advancement in home microwave technology is inverter technology. This technology allows for delivery of consistent levels of microwave energy at the various power levels instead of the standard pulsing method. These consistent levels of microwave energy help minimize runaway heat (Panasonic Microwave 2014).

**3.9.2 New Microwave Research and Technology**

In research and industry, some new strategies to minimize runaway heat include combining microwave heating with other heating modalities for a synergistic effect. These combination methods can also improve automation and quality. The three main goals for microwave combination heating are to achieve rapid heating, a desired
temperature profile, and a desired moisture profile. In microwave-jet impingement heating, for example, microwaves supply volume heating while jet impingement heats surfaces. Such heating combinations have been shown to generate more uniform temperature profiles (Datta and Rakesh 2013). Other combination microwave heating methods in current research include, infrared heating, steam heating and induction heating.

3.9.3 Combination Heating - Jet Impingement Heating

Jet impingement heating is a type of hot air heating in which jets of hot air are directed on a product at high velocities to enhance heat transfer. This technique introduces airflow to the microwave process, increasing the rates of heat and moisture transport (Datta and Rakesh 2013). This method can cause rapid evaporation at the surface of food products resulting in desired sensory characteristics such as crispness. As a result, this combined method introduces new baking and frying applications. Sometimes these jet impingement microwave combination systems also include a source of infrared or a grill (Datta and Rakesh 2013). Figure 3.2, shows that this combined heating system delivers a substantial temperature rise after two min in comparison to the individual methods on their own. This data also shows that with the combination heating system, the non-uniformity of heating is also less than the individual methods. Currently these types of combination microwaves are available commercially in the high-end microwave market (Balasubramaniam 2013).
Figure 3.2 Average Temperature Rise (Patterns) and Non-Uniformity (Solid Regions) in Heating After 2 min of Heating Under Different Heating Modes in the Microwave-Hot Air Jet Combination Oven (Datta and Rakesh 2013)

3.9.4 Combination Heating - Infrared Heating

The applications of infrared heating include drying, baking, roasting, grilling and reheating. This form of combination heating involves introducing halogen lamps or heated rods as a source of the infrared into the microwave cavity (Datta and Rakesh 2013).

3.9.5 Combination Heating - Steam

Another microwave heating combination system introduces the use of steam. Several patents exist using applications that produce secary steam from within a food container or package, however there is little scientific research literature on introducing steam into the microwave cavity simultaneously with microwave heating. In the secary steam forms, this steam can surround food items in the microwave, leading to a more even heat distribution (Datta and Rakesh 2013). Currently there are also ovens that have a
steaming function that introduce the use of steam into the microwave cavity, although this steaming function is used independently of microwave energy (Datta and Rakesh 2013). Considering these systems are already in place, it would be interesting to see research on the effectiveness of introducing steam and microwave heating simultaneously.

3.9.6 Combination Heating - Induction

Induction heating involves using cookware that heats up, thus cooking the product. This induction cookware is often made using magnetic material such as steal, iron, nickel or alloys. In this technology, induction coils that produce a high-frequency alternating magnetic field are placed below glass to heat the cookware. The cookware is stimulated by the alternating magnetic fields resulting in it becoming hot and cooking the food (Datta and Rakesh 2013). Combined induction heating and microwave heating systems are not yet available commercially or discussed extensively in scientific studies.

3.9.7 Metallic Packaging

Packaging has been one very successful solution to many of the uneven heating issues. In 1952, Moffett patented the use of a metallic shield to prevent one part of a multi-food package from being heated in an “electric cooker”. Three years later, Welch took this idea and adapted it for the microwave by patenting a multicomponent microwave tray with metallic lidding material that would be selectively placed depending on the desired heat distribution (McCormick 1991). This technology is also present in the use of susceptors, a microwave specific package, which causes metallic components to come in
contact with the product in order to induce browning. This technology is familiar to most people from the product Hot Pockets.

3.10 Future Research and Gaps

Some of the research on improvements in freezing and microwave technology is done privately by the food industry, so there is most likely much more information on improved methodologies that is not publically available. Some of the gaps in information where available scientific studies are lacking include packaging, product formulation, steam combination heating and induction microwave combination heating (Datta and Rakesh 2013).

Considering strides made with freezing and microwave technology in the last century, it will be interesting to see how these technologies continue to evolve. As demonstrated in Datta and Rakesh 2013, combination-heating methods have immense potential to improve microwave heating. As mentioned previously, combined methods such as introducing steam with microwave heating could be one successful way to improve uniform heat distribution that has not been thoroughly researched. Acoustic thawing in combination with microwave heating is another specific area that needs more study. Datta and Rakesh 2013, describe combination of heating modes as the “holy grail of customized quality that can be automated,” explaining that there are infinite possibilities for microwave heating combination methods in the future. On the other hand, the future of freezing research studies could involve the introduction of new food product additives that can affect freezing rates and freezing quality, as well as new freezing techniques that
effect sensory characteristics. Lastly, improvements in temperature modeling for both freezing and especially microwave technology could vastly advance and ease research in these fields.
For all experiments, the following methods were used. The chapter begins with the generic methods with all variants listed. Following the generic description in this section, specific experiments and respective details are explained in the next section.

4.1 Sample Preparation

Russet potatoes and daikon radishes, purchased from Giant Eagle supermarket in Columbus, Ohio were used in this study. Care was taken to assure that the products were not visibly damaged and large in size (approximately 300 g for the potatoes). Slices ranging 8-12 cm in diameter and 6mm or 4.5mm thick were cut using a V-blade mandolin (OXO International, Ltd., New York, NY). These dimensions provided infinite slab geometry, where the width was more than three times the height, so that temperature exchange would only occur from the top and the bottom.

Raw sample slices were either run immediately for texture analysis to provide a baseline, prepped for freezing, or frozen immediately depending on experimental design. Freezing prep involved lightly blanching the slices, individually blotting them with paper towels, placing them on a sheet of plastic wrap, covering them with a sec sheet of plastic wrap, and bringing them to ambient temperature (~23°C±2).
4.2 Freezing

All samples started at ambient temperature (~23°C±2) prior to freezing. Time to freeze was defined as the time it took for the center of the slice to reach -20°C. Characteristic freeze time, a measure of the amount of time from when the initial ice crystals appear to when approximately 95% of the product is frozen, was experimentally defined as the time it took to get from -0.5 to -16°C (Heldman 2007). All temperatures of samples during freezing were measured using a Multilogger Thermometer (Model # HHH506RA Omega Engineering Inc. Stamford, CT) using grounded copper-constantan T-type probes with a sheath diameters of 0.032 in [0.082 cm] and lengths of 6 in [15.24 cm] (Part # TMQSS-032G-6, Omega Engineering, Inc., Stamford, CT). This will be referred to as the ‘primary temperature logger’ going forward. The probes were inserted into the center of the potato slices through the radius prior to freezing.

Due to the limited data capacity of the primary temperature logger, for longer-term temperature logging of air inside freezers, a stainless steel waterproof temperature data logger was used (model # HT220 Dickson, Addison, IL). Going forward this will be referred to as the ‘alternate temperature logger’.

For the slower range of freezes (1,080 sec to 25,860 sec), slices were placed on an upside down pizza insert (Figure 4.1) in order to maximize airflow to the sample and thus provide more uniform freezing.
Plastic wrap was kept on the potato slices in order to prevent moisture loss. For these freezes, the following equipment was used:

### Table 4.1 Freezer Details

<table>
<thead>
<tr>
<th>Name as Used/Freezer Type</th>
<th>Freezer Details</th>
</tr>
</thead>
<tbody>
<tr>
<td>Blast Freezer</td>
<td>Model: BCF- 65 Blast Chiller/Freezer Single phase 208V, 60Hz, 15A, Henny Penny®, Eaton, OH</td>
</tr>
<tr>
<td>Manual Defrost Freezer</td>
<td>Model#253.28042807, 115V, 60Hz, 5.0A, Kenmore, Beverly Road Hoffman Estates, IL</td>
</tr>
<tr>
<td>Auto-defrost Freezer</td>
<td>Model#2F, 115V, 60Hz, Continental, Bensalam, PA</td>
</tr>
<tr>
<td>Walk in Deep Freezer</td>
<td>Bally Refrigerated Boxes, Inc., Morehead City, NC</td>
</tr>
<tr>
<td>(not used in final experiments)</td>
<td></td>
</tr>
<tr>
<td>Refrigerator Freezer Compartment [with auto-defrost cycle] (not used in final experiments)</td>
<td>Model# FFTR1814LWA, 115 V, 60 Hz, Frigidaire Electrolux Home Products, Charlotte, NC</td>
</tr>
</tbody>
</table>

For the rapid freezing (60 to 488 sec), slices were frozen in a dry ice ethanol bath. For these samples, the cellophane was removed and slices were placed in zip lock bags to prevent moisture loss and direct contact with the dry ice ethanol fluid. The dry ice ethanol bath consisted of approximately 400 liters of 200 proof ethanol (Decon Laboratories, Inc. King of Prussia, PA) and between 450- 2,300 g of fully dissolved 5/8 in [1.59 cm] diameter dry ice pellets (Continental Carbonic Products Inc. Decatur, IL). The dry ice bath was contained in a 4300 ml shielded Dewar liquid nitrogen flask (Pope
Lab Grade Wide Mouth, Pope Scientific Inc. Saukville, WI). The amount of dry ice was adjusted in order to provide dry ice-ethanol bath temperatures ranging from -22°C to -71°C. The primary temperature logger was used to monitor the bath temperature.

After freezing, all frozen slices were then stored in a Styrofoam box (dimensions 4.5” [11.43 cm] x 4.5” [11.43 cm] x 5.5” [13.97 cm]) in the manual defrost freezer to equilibrate for 12-24 hours.

**Table 4.2 Summary of Established Freezing Conditions and Mean Times to Freeze**

<table>
<thead>
<tr>
<th>Freezing Condition</th>
<th>Mean Times to Freeze Established</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dry Ice Ethanol Bath</td>
<td>60 to 488 sec</td>
</tr>
<tr>
<td>Blast Freezer</td>
<td>1,080 sec</td>
</tr>
<tr>
<td>Manual Freezer Setting 7</td>
<td>4,860 sec</td>
</tr>
<tr>
<td>Auto Defrost Freezer with Small Styrofoam Box Nested in Medium Styrofoam box</td>
<td>25,860 sec</td>
</tr>
</tbody>
</table>

### 4.3 Thawing

Individual and stacked frozen slices were thawed to ambient temperature (~23°C±2) either at ambient temperature or using a conventional microwave oven. The individual slices thawed at ambient temperature were kept covered in either plastic wrap (slower freezes) or in zip lock bags (rapid freezes), and the individual microwave-thawed slices were all in zip lock bags to prevent moisture loss. The plastic wrap on individual slices that were to be compiled in stacks was removed, and these stacks were carefully rewrapped in plastic wrap as a stack prior to thawing. This was done to prevent moisture
loss and to keep the slices in the stacks flush. The plastic wrap on the stacks frozen as stacks remained on the stacks during thawing.

The microwave process was conducted using a 1,250-watt full-size microwave oven (Model: NN-SD987S, SKU: 8285232, Panasonic Osaka, Osaka Prefecture, Japan). The dimensions and features were as follows: height: 14” [35.56 cm], width: 23-7/8” [60.6425 cm], depth 19-4/7” [49.7114 cm], and capacity: 2.2 cubic ft [67.056 cubic cm]. A 16.5 in [41.91 cm] diameter Styrofoam plate was placed on the turntable to limit heat transfer between the samples and the glass plate. This microwave also featured inverter technology known to minimize runaway heat (Panasonic Microwave 2014). A fiber optic temperature sensor (Micronor, Inc. Newbury Park, CA) was used to measure the core temperature of the samples in the microwave oven in order to determine optimal microwave power and thaw times. All the acquisitions were made using FoTemp Assistant software (Optocon AG. Dresden, Germany), in a Windows environment.

While thawing at ambient temperature, the stack samples were balanced on empty open yogurt cups (Figure 4.2) so that the counter would not affect the thawing of the bottom slice.
4.4 Objective Texture Analysis

Each sample was quantitatively analyzed using TAxT2i Texture Analyzer (Stable Micro System, Godalming, Surrey, UK) equipped with a TA-90 aluminum platform, a TA-25 2”diameter 20mm tall aluminum cylinder probe, and a 25 Kg load cell. The TAxT2i is a microprocessor that measures force, distance and time, providing a three-dimensional product analysis (TA.XT2i and TA.HDi Texture Analyser User Guide 2006). All samples were tested at ambient temperature 25°C±3 using the double compression non-fracture Texture Profile Analysis (TPA) test. TAxT2i Texture Analyzer and Texture Expert Exceed software create texture profile analysis curves used to analyze the textural qualities of the potato. The standard double compression test generates a curve as previously shown in Figure 3.1.

The TPA curve is characterized by three height measures, four time measures, and five areas measures. The TAxT2i Texture Analyzer and Texture Expert Exceed software use these measures to provide the following objective textural parameters: TPA hardness (g), TPA springiness (s/s), TPA cohesiveness (g s/g s), TPA resilience (g s/g s), TPA chewiness (g) and TPA gumminess (g).
The following experimental parameters were selected for each TPA test: pre-test speed (2.0mm/s), test speed (5.0mm/s), post test speed (5.0mm/s), rupture test distance (4.0%), distance (35.0%), force (100g), Time (1.00sec), and count (5). All the acquisitions were made using Texture Expert Exceed software version 2.64 working in Windows environment.

4.5 Statistics

Early models for curves used the trend line capability in Microsoft ® Excel® for Mac 2011 Version 14.4.1 (Microsoft Corporation, 2010). Final statistical analysis was completed with the help of the The Ohio State University Statistical Counseling Services (subsequently, “stats counselor”).

4.6 Influence of Thaw Methods on Textural Quality of Frozen Foods

In this experimental series, 6mm individual potato slices were frozen for times to freeze ranging from 65 to 4,860 sec. Individual frozen slices were either thawed at ambient temperature or using the microwave oven at power 1 for 180 sec. The primary textural parameter (TPA hardness) was plotted against time to freeze for both the slices thawed at ambient temperature and the slices thawed in the microwave oven. A stats counselor was used to fit an enhanced exponential function to the TPA parameter data using SAS. A regression analysis was conducted to compare the slices thawed at ambient temperature and the slices thawed in the microwave oven. This was done by combining the sets of data in the presence of a dummy variable and an indicator to determine significant differences in the plots.
4.7 Influences of Freezing on Potato Texture – Best Fit Model

In this experimental series, 6 mm potato slices were frozen for times to freeze ranging from 65 to 4,860 sec. The primary textural parameter (TPA hardness) was plotted against times to freeze. Excel was used to evaluate the following trend lines: linear, logarithmic, polynomial, moving average, exponential and power. The stats counselor helped fit an enhanced power function and enhanced exponential function using SAS.

To further validate these findings, micrographs were taken using a scanning electron microscope. The four types of photographs taken were of the following: raw, just blanched (using blanching method mentioned previously), a time to freeze of 70 sec, and a time to freeze of 1,080 sec. A sample of these potatoes was acquired for the microscope using a razor blade. These photographs were taken at three levels of magnification x40, x250 and x1.5k.

4.8 Analysis of TPA Parameters

In this experimental series, 6 mm potato slices were frozen for times to freeze ranging from 65 to 4,860 sec. TPA textural parameters were plotted against times to freeze, compared and evaluated. These parameters and their equations were as follows:

- TPA Hardness = Force 2
- TPA Gumminess = Force 2 * Area 2 / Area 1
- TPA Chewiness = Gumminess * Length 2 / Length 1
- TPA Cohesiveness = Area 2 / Area 1
• TPA Springiness = Length 2 / Length 1
• TPA resilience = Area 5 / Area 4

4.9 Impact of Freezing on Structure

In this experimental series, a limited sample of raw 6 mm daikon radish slices were frozen with times to freeze of 75 to 5,340 sec. All frozen slices were thawed at ambient temperature. The primary textural parameter (TPA hardness) was plotted against time to freeze. A stats counselor was used to fit an enhanced exponential function to the data using SAS. A regression analysis was conducted to compare the daikon radish data to the potato data. This was done by combining the sets of data in the presence of a dummy variable and an indicator to determine significant differences in the plots.

4.10 The Influence of Slice Thickness on Freezing Impact

In this experimental series, a limited sample of blanched 4.5 mm potato slices were frozen with times to freeze of 28 to 5,700 sec. All frozen slices were thawed at ambient temperature. The primary textural parameter (TPA hardness) was plotted against time to freeze. A stats counselor was used to fit an enhanced exponential function to the TPA hardness data using SAS. A regression analysis was conducted to compare the TPA hardness data for the 4.5 mm potato to the TPA hardness data for the 6 mm potato. This was done by combining the two sets of TPA hardness data in the presence of a dummy variable and an indicator to determine significant differences in the plots.
4.11 TPA Hardness versus Characteristic Freeze Time Relationship

The Industrial-Scale Food Freezing-Thawing Simulation (Version 3.0.0, Department of Biological and Agricultural Engineering, University of California Davis, CA) was used to further analyze the TPA hardness data by providing temperature history distributions. Brine and/or air temperatures were altered to achieve the target times to freeze that were covered in the range of the freeze-thaw experimental data. The time and temperature data from the program were used to provide characteristic freeze times within the 4.5 mm and 6 mm slices and mass average characteristic freeze times that would correlate with experimental TPA hardness data.

4.12 Influence of Location During Thawing on TPA Texture of Frozen Potatoes

In this experimental series, individual 6 mm thick slices were frozen either for 65 sec (dry ice ethanol bath) or 1,080 sec (blast freezer). After equilibrating the samples, slices were arranged into uniform or mixed freeze type stacks of five as indicated in Figure 4.3. Stacks were either thawed at ambient temperature or in the microwave oven for 420 sec at power 1. The results were averaged for each treatment condition, and the Tukey Test was used to determine statistical differences in the TPA hardness of the individual slices.

![Figure 4.3 Uniform and Mixed Freeze Type Stacks of Five Potato Slices Showing the Positions for the Freeze Types](image-url)
Chapter 5: Development of Experimental Process

There were many challenges in developing the experimental protocol for this study. Some of the issues related to product choice and preparation (sample uniformity, undesirable chemical and physical changes), the freezing process (equipment and instrumentation limitations, temperature fluctuations, establishing varied times to freeze, maintaining repeatable freeze conditions), the microwave process (instrumentation and equipment limitations, moisture loss, temperature fluctuations, establishing uniform thaws), and the texture analysis. Experiments were conducted to optimize conditions for the final methods.

5.1 Product Choice and Preparation

For product choice and preparation, some of the challenges faced were ensuring uniform samples, preventing undesirable chemical changes, and preventing undesirable physical changes.

5.1.1 Sample Uniformity

In determining what kind of samples to use for this study, a challenge faced was finding a sample that would provide a freezing model for a typical food structure. It was important that the samples were accessible year round, had limited seasonal variation, were easy to handle, and had relatively consistent radial texture. Russet potatoes provided the most
ideal fit of the requirements for these core experiments, as well as being relatively shelf stable and inexpensive. In addition, frozen potatoes account for 50% of total US potato exports and are even considered one of the six main categories of frozen food in the global frozen food market (United States Potato Board 2014; Transparency Market Research 2013).

In subsequent experiments, daikon radishes were also used. Daikon radishes, which are root vegetables like potatoes, were chosen because they too had a relatively consistent radial texture as well as a similar size and shape, but had a large difference in %water with potatoes at about 79% water and daikon radishes at about 95% (Water Content of Fruits and Vegetables 1997).

In order to ensure consistent slice sizes, a mandolin was used and care was taken to ensure slices were large enough in length and width to exhibit infinite slab geometry. As a result, the potato slices were cut to either 4.5mm or 6mm thick, approximately 8-9 cm in length and approximately 5-7 cm in width.

5.1.2 Preventing Undesirable Chemical and Physical Changes

One of the first challenges with the potato slices samples was enzymatic browning. Blanching potatoes is commonly done prior to cooking or processing in order to limit enzymatic activity that can result in loss of flavor, color, texture and nutrients (Joslyn 1961). This practice also removes air from the tissue, increases flexibility and controls firmness after cooking (Tijskens and others 1997).
To determine optimal blanching times, samples of potato slices were blanched at 15-sec intervals from 30 sec to 120 sec, cooled in an ice bath and then the samples were stored in plastic containers with their replicates for 24 hours at ambient temperature and evaluated based on color and texture.

<table>
<thead>
<tr>
<th>30 Sec</th>
<th>45 Sec</th>
<th>60 Sec</th>
<th>75 Sec</th>
<th>90 Sec</th>
<th>105 Sec</th>
<th>120 Sec</th>
</tr>
</thead>
</table>

**Figure 5.1 Blanching Visual Test**

Visual analysis of the samples indicated that visible enzymatic browning occurred at 30 and 45 sec, and after 60 sec no enzymatic browning was visibly present (Figure 5.1).
Texture analysis of the blanched samples indicated little change in TPA hardness until 75 sec Figure 5.2. After 75 sec, there was a large decrease in TPA hardness of about 70%. Based on the results from these two tests, a blanch time of 60 sec in boiling water was established. Slices were then moved to an ice bath for 90 sec, which provided enough time to stop the blanching, without overly chilling the slices. This time was selected because slices were warm to the touch at the first attempt of 60 sec. During this test, it was also observed that when stored together, the samples on top had more browning than the ones underneath, so in subsequent experiments, slices were stored individually to decrease sample variance.

Internal and external moisture presented another challenge. In order to remove excess water, slices were individually blotted dry with paper towels after being removed from
the ice bath. This was done so that excess water was not reabsorbed by the potato, and thus influence the freeze experiment. Each slice was then covered in cellophane to minimize moisture loss as a variable during freezing experiments.

5.2 The Freezing Process

During the freezing process, some of the challenges included managing temperature fluctuations, dealing with instrumentation and equipment limitations, establishing varied times to freeze, and maintaining uniform repeatable freeze conditions.

5.2.1 Managing Temperature Fluctuations with Freezing Equipment

Freezers are known to have defrost cycles and regular temperature fluctuations. It was important to minimize these fluctuations in order to limit the experimental variables. In addition, temperature fluctuations tend to decrease the number of ice crystals and increase the overall size of the ice crystals, resulting in a loss of quality (Erickson and Hung 1997), which make managing these fluctuations even more critical.

The first step in developing the freezing process involved evaluating various freezers. The first freezer evaluated was the walk in deep freezer. (Please refer to chapter 4 for details on equipment used in this chapter.)
A deep freeze walk in freezer was evaluated using the alternate temperature loggers for a three-day period. One temperature logger was placed near the door “by door” and one temperature data logger was placed away from the door “away from door”, to get a range of the air temperatures. The data from two complete days were recorded and plotted. Figure 5.3 shows twenty spikes in temperature during the 24-hour period including two defrost cycles daily. The defrost cycles ran at 11AM and 11PM daily causing the temperature “by door” to reach as high as -13.6°C and “away from door” to reach -9.5°C. The temperature “by door” reached a minimum value of -33.1°C and the temperature “away from door” reached a minimum temperature value of -33.3°C. The mean temperature was -28°C for both locations with a standard deviation of 4.2 for “by door” and 4.8 for “away from door”.

Figure 5.3 Temperature Profile of Air for 24 Hours over 2 Days in Walk in Deep Freezer
The biggest problem encountered with this freezer was that while the primary temperature logger, used to measure the temperature of samples, could measure temperatures from -200 to 400°C, the device itself was not equipped to be in -30°C temperature. As a result, during the freezing process, the device would begin to dysfunction. Unfortunately, even with a longer thermocouple probe wire, leaving the primary logger in the hallway during freezing was not ideal. In addition, people walking in and out of public freezers could not be easily controlled adding another variable to the freezing experiments.

The next freezer evaluated was the auto-defrost freezer. The same alternate temperature logger was used for a three-day period to measure the air temperature in four different places within the freezer: top, top below the fan, middle, and bottom. Figure 5.4 shows the temperature profile over a 24-hour period. In this data there are twenty spikes in temperature during the 24-hour period including two defrost cycles. The defrost cycles ran at 11AM and 11PM daily causing the probe in the freezer to reach as high as -6.4°C, which occurred in the top section. Overall, the top section had the highest mean temperature of -25.9°C with a standard deviation of 4.5°C and the bottom section had the lowest mean temperature of -28°C with the lowest standard deviation of 4.1°C.
One issue that became apparent during this experiment was that there was a wide range of time in the beginning of the data that exhibited highly instable temperature fluctuations. In the first nine hours, highlighted in Figure 5.5, there was visible instability in the temperature fluctuations, which raised concern.
The next freezer evaluated was the refrigerator freezer compartment (Figure 5.6). This data showed 24 fluctuations in temperature approximately every hour, with a range from -9 to -22.5°C. The defrost cycle occurred every 30 hrs causing the temperature to go as high as 34.6°C. It was not expected that the defrost cycle would go so high. These were not acceptable figures for experimental freezing conditions.
The next freezer evaluated was the manual defrost freezer (Figure 5.7). At its lowest setting, there were 20 temperature fluctuations and no defrost cycle. The soft freeze section had the least variability with a mean temperature of -20.4°C and a standard deviation of 2.0°C, however this area was not large enough for sample freezing. The top section was the next least variable, with a mean temperature of -16.5 and a standard deviation of 3.9°C. However, even at the coldest setting (setting 7), this section did not stay below -20°C, which was mandatory for these experiments. The middle and bottom section had mean temperatures of -22.4°C, -23.8°C and standard deviations of 4.5°C, 5.0°C respectively, which were both suitable for these experiments.
Next the process of freezing the potatoes at different temperature in order to achieve multiple times to freeze was explored (Figure 5.8). At settings 1 and 2, the target end point of -20°C was not reached. At settings 3, 4, and 5, the samples responded to temperature fluctuations starting at -7°C, while settings 6 and 7 provided relatively clean curves. Setting 7 was chosen as the primary setting to use because it reached the end point of -20°C most consistently, as well as being the least likely to be effected by the normal freezer temperature fluctuations. While the other setting provided slight variations in the times to freeze, the differences were not large enough to produce a significant enough change of condition for comparison.
The temperature over 24 hours was recorded for the same manual defrost freezer at the
coldest setting (setting 7) to confirm this decision (Figure 5.9).

**Figure 5.8 Temperature Profile of Single Potato Slices Frozen at 7 Different Settings in Manual Defrost Freezer**
Figure 5.9 Temperature Profile of Air at Setting 7 in Manual Defrost Freezer

At this setting the mean temperature was -33°C and the standard deviation was 5.6°C, which was appropriate for these experiments. In fifteen independent trials, setting 7 produced a mean time to freeze of 81 min (8,460 sec) with a standard deviation of 9 min (540 sec). This was the setting used for the majority of the experiments. An example of a freeze curve in this condition is presented in Figure 5.10.
Potato slices were also frozen at settings 6 and 4 in preliminary experiments. For setting 6, the mean time to freeze was 89 min (5,880 sec) with a standard deviation of 11.2 min (672 sec). For setting 5, the mean time to freeze was 94 min (5,640 sec) with a standard deviation of 4.2 min (252 sec). For setting 4, the mean time to freeze was 128 min (7,680 sec) with a standard deviation of 14.6 min (876 sec).

Next a blast freezer was used to provide a more rapid freeze condition. The freeze curve for the blast freezer is shown in Figure 5.11. The mean temperature was -34.4°C with a standard deviation of 0.4°C. It was evident from the curve that the constant air circulation in the blast freezer was able to keep the temperature in a much more stable state than standard freezers. In seven independent trials, the mean time to freeze of 18 min (1,080 sec) was established with a standard deviation of 3.1 min (186 sec).
5.2.2 Establishing Extra Long Freezes

In order to establish an extra long time to freeze, Styrofoam was used to create layers of insulation. In the first experiment, a potato slice was frozen between two pieces of Styrofoam. This freeze took 582 min (34,920 sec). The problem with this freeze was that most of the cooling would be occurring from the sides as opposed to from the top and bottom.

Next, a single potato slice was frozen in a small Styrofoam box with the dimensions 8” [20.32cm] x 4.5” [11.43cm] x 6” [15.24cm] in the manual defrost freezer at setting 7 (Figure 5.12). This freeze took 161 min (9,660 sec) with the temperature of the slice fluctuating repeatedly from -21 to -27°C about every 70 min (4,000 sec).
To further increase the time to freeze and dampen the fluctuations, experiments were conducted nesting a small Styrofoam box inside a larger one. Since the manual defrost freezer racks were not large enough to contain larger Styrofoam boxes, these experiments were conducted in the auto defrost freezer. In one trial, a small Styrofoam box was nested in a medium Styrofoam box with the dimensions 13” (33.02 cm) x 13” (33.02 cm) x 11” (27.94 cm), and in another, a small Styrofoam box was nested inside a large Styrofoam box with the dimensions 17” (43.18 cm) x 14” (35.56 cm) x 12.5” (31.75 cm) (Figure 5.13). The times to freeze of both cases were very similar, 365 min (21,900 sec) for the small in medium and 372 min (22,320 sec) for the small in large. While there were some fluctuation in both cases around the time of the defrost cycle, the addition of
another layer of Styrofoam box significantly increased the time to freeze and muted the effect of freezer temperature fluctuations in comparison to the single Styrofoam box in the manual defrost freezer.

![Figure 5.13 Freezing Curve for Single Potato Slices Frozen in Styrofoam Boxes (Auto Defrost Freezer)](image)

**Figure 5.13 Freezing Curve for Single Potato Slices Frozen in Styrofoam Boxes (Auto Defrost Freezer)**

Figure 5.14 compares the effect of each condition, as well as the single slice frozen in just a single small Styrofoam box against the freezer air temperature.
In twelve independent trials, the small Styrofoam box nested in a medium Styrofoam box had a mean time to freeze of 409 min (25,540 sec) with a standard deviation 61 min (3,660 sec). In twelve independent trials the small Styrofoam box nested in a large Styrofoam box had a mean time to freeze of 388 min (17,280 sec) with a standard deviation of 89 min (5,340 sec). As a result, it was decided to use the small Styrofoam box nested in a medium Styrofoam box for the extra long freezes. In thirteen independent trials with this condition, the mean time to freeze was 431 min (25,860 sec) with a standard deviation of 49 min (2,940 sec).
5.2.3 Establishing Short Freezes

After establishing the long freeze, the next challenge was establishing short freezes. The first attempt at this was to freeze a potato slice on a slab of dried ice. In four trials (Figure 5.15), there was a mean time to freeze of 15.5 min (930 sec) with a standard deviation of 3 min (180 sec). This variation seemed unacceptable for trials done in the same series. In addition, irregularities in the freeze curve indicated that the slices were not being cooled at a consistent rate. This was likely because it was difficult to ensure that the slices were completely flush to the slab of dry ice. As a result, air pockets could lead to insulation that would cool the slices unevenly as well as effect the times to freeze. With this type of freeze, the cooling was also only occurring from the side where the slice was adjacent to the dry ice. This was problematic since this was a different cooling mechanism than the other freezes where the cooling was occurring evenly around the whole slice.
The next attempt for a rapid freeze was using a dry ice ethanol bath. In the first set of experiments, six slices were frozen in a dry ice ethanol bath with a mean time to freeze of 68 sec and a standard deviation of 10.6 sec, while this standard deviation was high, the first trial with a time to freeze of 89 sec appeared to be an outlier. Once this data point was removed, the mean time to freeze was 65 sec with a standard deviation of 2.8 sec. This initially appeared to be a very reliable method for repeatable freezes. So, at this point a time to freeze of 65 sec was established for the dry ice bath.
In an effort to establish times to freeze between the 65 sec freeze and the 1080 sec freeze, in further experimentation, the temperature of the dry ice bath varied by adjusting the amount of dry ice pellets used. This process resulted in dry ice bath temperatures ranging from -22°C to -71°C. In attempts to establish very rapid times to freeze, the variability in the short times to freeze between trials became a big issue. It was determined that the best way to manage rapid times to freeze was to freeze two slices at a time in a plastic bag, with one slice containing a thermocouple probe. The plastic bag was then pulled out of the dry ice bath when the slice with the thermocouple reached -20°C. The pair of slices was then associated with an exact time to freeze recorded, as opposed to experimentally determining set times to freeze for different bath temperatures. In addition, the type of plastic bag used was also varied in order to obtain a larger range of times to freeze. This process resulted in times to freeze from 60 to 488 sec.

Lastly, a bath of liquid nitrogen was used to establish rapid times to freeze. This resulted in times to freeze of 39 to 44 sec. However, with this process, the slices were fracturing during or after the freeze process (Figure 5.16), so this was not an acceptable method of freezing the slices. Often products that are frozen in liquid nitrogen are pretreated with dry ice to prevent freeze cracking. This was not an option, since it was desired that the samples be frozen in a uniform condition. Another method used to prevent freeze cracking is spraying liquid nitrogen on the slices. Spraying liquid nitrogen has been known to prevent cracking (Brown 1977), and for this reason, it is the method in which most commercially manufactured liquid nitrogen freezers use (Brown 1977). However,
using a spray method in the lab would make it difficult to control for an end temperature of -20°C.

**Figure 5.16 Freeze Cracking with Liquid Nitrogen Freeze**

The freezing conditions and mean times to freeze established in this section are summarized in the following table.

<table>
<thead>
<tr>
<th>Freezing Condition</th>
<th>Established Mean Times to Freeze (sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dry Ice Ethanol Bath</td>
<td>60 to 488</td>
</tr>
<tr>
<td>Blast Freezer</td>
<td>1,080</td>
</tr>
<tr>
<td>Manual Defrost Freezer Setting 4</td>
<td>7,680</td>
</tr>
<tr>
<td>Manual Defrost Freezer Setting 5</td>
<td>5,640</td>
</tr>
<tr>
<td>Manual Defrost Freezer Setting 6</td>
<td>5,340</td>
</tr>
<tr>
<td>Manual Defrost Freezer Setting 7</td>
<td>4,860</td>
</tr>
<tr>
<td>Auto Defrost Freezer with Small Styrofoam Box Nested in Medium Styrofoam box</td>
<td>25,860</td>
</tr>
</tbody>
</table>

The three primary freezing methods (dry ice ethanol bath, blast freezer, and manual defrost freezer) were further validated using Industrial-Scale Food Freezing-Thawing Simulation (Version 3.0.0, Department of Biological and Agricultural Engineering,
University of California Davis, CA). This simulation program used the imperial system, and then all figures were converted to the metric system for analysis. This comparison is exhibited in Table 5.2.

**Table 5.2 Validation of Experimental Times to Freeze Using Industrial-Scale Food Freeze-Thaw Simulation**

<table>
<thead>
<tr>
<th>Freeze Condition</th>
<th>Mean Times to Freeze</th>
<th>Simulation Conditions and Times to Freeze</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dry Ice Ethanol Bath (coldest)</td>
<td>65 sec</td>
<td>Temperature: -68°C</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Rate of Heat Transfer: 403 W/m²K</td>
</tr>
<tr>
<td>Dry Ice Ethanol Bath (warmest)</td>
<td>488 sec</td>
<td>Temperature: -23°C</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Rate of Heat Transfer: 135 W/m²K</td>
</tr>
<tr>
<td>Blast Freezer</td>
<td>1,080 sec</td>
<td>Temperature: -30°C</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Velocity: 0.905 m/sec</td>
</tr>
<tr>
<td>Manual Defrost Freezer - Setting 7</td>
<td>4,860 sec</td>
<td>Temperature: -23°C</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Rate of Heat Transfer: 13.5 W/m²K</td>
</tr>
</tbody>
</table>

5.2.4 Keeping Slices in a Stack Flush

In the initial stack experiments, it was observed that the slices were not always flush. It was important that this issue be minimized as much as possible because air pockets between slices could affect the results. To do this, care was taken to number slices so that the stack slices would be in the same order as in the original potato. In addition, slices did not always freeze completely flat; so in all cases many extra slices were frozen in order to account for irregularly frozen slices.
5.3  Thaw Process

Thaws were completed both at ambient temperature and in a microwave oven. Some of the challenges encountered during these steps related to the instrumentation and equipment limitations, moisture loss, temperature fluctuations, and overall establishing uniform thaws.

5.3.1 Moisture Loss

The first challenge was that the microwave cavity became more humid as the experiments took place as moisture evaporated out from the potato slices. To control the microwave cavity humidity, samples were placed in zip lock bags.

5.3.2 Managing Temperature Fluctuations

In initial experiments aimed to examine the heating capabilities of the microwave on potato slices (Table 5.3), it was apparent there were challenges with even heating. In the first experiment, 6mm potato slices were blanched and then microwaved in 5-sec increments from 5 to 30 sec at the default setting, power 10 (highest setting). Temperature measurements were made using the primary temperature logger.

Since the temperature often continued to increase after the microwave treatment, the temperature recorded for early experiments was recorded as the highest temperature reached by the probe after the completed microwave treatment.
Next, it was observed that the glass plate in the microwave would absorb heat from the potato slices as the experiments were performed altering the experiments initial conditions. To limit this effect going forward, the glass plate with a thermal conductivity of 0.8W/m, °K (NDT Education Resource Center 2012), was replaced with a Styrofoam disk with the much lower thermal conductivity of 0.01W/m, °K (NDT Education Resource Center 2012).

5.3.3 Limitations in Equipment and Instrumentation

While in freezing experiments, temperature measurements were made by inserting the thermocouple probe of the primary temperature logger prior to freezing, it was not possible to microwave slices with metal probes inside them. In addition, the standard method of inserting the thermocouple probes radially was also difficult. This would involve physically touching the slices, which could affect the temperature and textural data. As a result, in these experiments, the probe was inserted into the center of the slice from the top once the microwave treatment was completed.

While these were just blanched slices, the results of these experiments indicated that at low microwave times (5-30 sec), the slices were heating up very quickly (as high as 84.5°C), so there could be value in examining lower power levels.
TABLE 5.3 FINAL TEMPERATURES OF BLANCHED SLICES MICROWAVED FROM 5 TO 30 SEC AT DEFAULT POWER SETTING (10) IN THE MICROWAVE OVEN (N=36)

<table>
<thead>
<tr>
<th>Time in Microwave (sec)</th>
<th>Mean Temperature (°C)</th>
<th>Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>30.6</td>
<td>4.5</td>
</tr>
<tr>
<td>10</td>
<td>46.8</td>
<td>3.3</td>
</tr>
<tr>
<td>15</td>
<td>57.0</td>
<td>4.4</td>
</tr>
<tr>
<td>20</td>
<td>70.7</td>
<td>4.8</td>
</tr>
<tr>
<td>25</td>
<td>84.5</td>
<td>9.9</td>
</tr>
<tr>
<td>30</td>
<td>84.6</td>
<td>8.9</td>
</tr>
</tbody>
</table>

In the next set of experiments (Table 5.4), the differences in the power setting on the microwave oven were explored. For these experiments, blanched potato slices were heated up at either power 1 or power 10 for 5 to 480 sec. The final temperature of three trials at each time was recorded using the same method as the prior experiment.

The first observation was that the slices were heating up too fast at power 10. At 5 sec, the temperature of the slice had already reached 30.5°C. In comparison, it took 20 sec at power 1 to reach 30.6°C. While the final experiments would be dealing with frozen slices, these results made it clear that at the lower power setting there would be more control in reaching a target end temperature. Lower power resulted in slower heating, which helped get the slices to more precise end temperatures. Lastly, while not definitive, it appeared that the lower power gave more consistent results, as observed in comparing the standard deviations at power 1 and power 10 (see Table 5.4).
Further experiments were performed using a fiber optic temperature sensor (Micronor, Inc. Newbury Park, CA). With this sensor, the temperature of the potato slices could be measured during microwave heating. However, in order to do this, the probe needed to be in the slices. Since the probe could not readily be inserted into the frozen potato slices, a small drill bit was used to allow for the probe to be inserted.

To test the reliability of drilling holes in frozen potatoes with the fiber optic temperature sensor potato samples were frozen, half with thermocouple probes frozen inside them, half without. Pairs of slices were removed from the freezer one at a time, with one slice
containing a thermocouple probe. The slice without the thermocouple probe then had a hole drilled in it. The temperature of both slices was then simultaneously measured and recorded in Table 5.5.

These results demonstrated that the drilling did not significantly distort the data and therefore this method could be confidently used in subsequent analysis to determine thaw times in a microwave oven for the various freeze conditions.

**Table 5.5 Reliability of Drilling Potato Slices for Measuring Internal Temperature of Potato Slices Using Fiber Optic Temperature Sensor**

<table>
<thead>
<tr>
<th>Trial</th>
<th>Fiber Optic Temperature Sensor (°C)</th>
<th>Primary Temperature Logger (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>#1</td>
<td>-2.5</td>
<td>-5.7</td>
</tr>
<tr>
<td>#2</td>
<td>-5.2</td>
<td>-6.4</td>
</tr>
<tr>
<td>#3</td>
<td>-7.5</td>
<td>-6.3</td>
</tr>
<tr>
<td>#4</td>
<td>-6.8</td>
<td>-4.8</td>
</tr>
<tr>
<td>#5</td>
<td>-4.9</td>
<td>-6.4</td>
</tr>
<tr>
<td>#6</td>
<td>-4.6</td>
<td>-4.4</td>
</tr>
<tr>
<td>#7</td>
<td>-4.9</td>
<td>-4.3</td>
</tr>
<tr>
<td>#8</td>
<td>-4.4</td>
<td>0.8</td>
</tr>
</tbody>
</table>

One of the first experiments done using the fiber optic temperature sensor compared end temperature with time in microwave at power 1 (Figure 5.17). These results demonstrated a large variation of end temperatures over a wide range of microwave times. One interpretation of the data is that even with the fiber optic temperature sensor, measuring one point in the slice did not seem adequate, possibly due to runaway heat.
The next set of experiments was aimed to evaluate the effect of microwave time on stacks of five slices. First, the time in the microwave at power 1 to reach 20°C was recorded for each position of individually frozen slices, thawed as stacks of five (Table 5.6). These times ranged from 380 to 710 sec with a mean of 556.2 sec.
**Table 5.6 Time in Microwave at Power 1 to Reach 20°C for Each Position in Stacks of Five Frozen Slices Thawed as a Stack for Five Different Stacks**

<table>
<thead>
<tr>
<th>Slice Order in Stacks of Five Potato Slices Frozen Individually at Time to Freeze of 65 Sec</th>
<th>Time in Microwave at Power 1 to Reach 20°C (sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1st Slice</td>
<td>380</td>
</tr>
<tr>
<td>2nd Slice</td>
<td>382</td>
</tr>
<tr>
<td>3rd Slice</td>
<td>694</td>
</tr>
<tr>
<td>4th Slice</td>
<td>710</td>
</tr>
<tr>
<td>5th Slice</td>
<td>665</td>
</tr>
</tbody>
</table>

The TPA hardness of these slices is shown in Figure 5.18.
This figure shows that the TPA hardness of the individual slices is not significantly different. This observation is explored in more detail in the results section.

5.4 Textural Analysis

For this study, TAxT2i Texture Analyzer and Texture Expert Exceed software were used to analyze the textural qualities of the potato. The TAxT2i Texture Analyzer was chosen since it is an industry standard and has been proven to correspond to sensory test data (Meullenet and others 1998). Some of the challenges during this part related to sample size, probe size and sample uniformity.
5.4.1 Sample and Probe Size

In initial experiments, whole potato slices were being used. While during freezing slight differences in the slice sizes were not significant due to the infinite slab geometry, these differences presented a challenge in textural measurement. As illustrated in Figure 5.19, with slices larger than the probe, the strain from the compression test would sometimes result in stress release causing the slices to buckle. As a result of this observation, a coring tool was used to ensure that all slices were exactly the same size (2.5 cm diameter).

![Figure 5.19 Potato Slice on TPA Platform](image)

From this point forward, these 2.5 cm samples were made and used for texture analysis, and a larger 2” (50.8mm) diameter 20mm tall aluminum cylinder probe was used.

5.4.2 Sample Uniformity

During these trials, the textural differences within the potato slice also became apparent. From visual observations it appeared that the center texture of the slices was different than the areas around the periphery. To test this observation, core samples from the
center of blanched potato slices were compared with core samples from the remaining areas (Table 5.7). The standard deviation of the center cores (1001.76 g) was less than half the standard deviation of the multiple cores (2069.70 g), so it was decided to go ahead with just the center cores. This observation was validated in literature that demonstrated the variance of texture within the potato slice (Figure 5.20). The outer skin consists of a layer of dead cells called “corky periderm”. These cells are thicker than the main parenchyma cells and do not contain starch of protein grains (Andersson and others 1994). So as a result, it was decided to go forward with center core measurements to improve sample consistency.

**Table 5.7 Textural Hardness of Center Cores Versus Multiple Cores**

<table>
<thead>
<tr>
<th></th>
<th>Mean (g)</th>
<th>Standard Deviation (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Blanched Center Cores</td>
<td>22186.64</td>
<td>1001.76</td>
</tr>
<tr>
<td>Blanched Multiple Cores</td>
<td>20831.49</td>
<td>2069.70</td>
</tr>
</tbody>
</table>

**Figure 5.20 Longitudinal Cross-Section of the Potato Tuber (Andersson and others 1994)**
Before presenting and discussing the analysis of the TPA parameter data, it is important to provide background on steps followed during the collection and assembly of the data.

In general, each TPA parameter has been measured at several times to freeze and over a range of times. When the times to freeze were between 60 and 488 sec, the potato slices were frozen in a dry ice ethanol bath. An air blast freezer provided a time of freeze of 1,080 sec, and a manual defrost freezer provided a time to freeze of 4,860 sec.

As mentioned in section 4.2, these procedures did not allow for collection of TPA parameter data at less than 60 sec, between 488 and 1,080 sec or between 1,080 and 4,860 seconds:

- **Times to Freeze at less than 60 sec**: The shortest time to freeze using the dry ice ethanol bath was ~60 sec, using the dry ice ethanol bath at the coldest temperature achievable (~80°C).

- **Times to Freeze Between 488 and 1,080 sec**: For longer times to freeze using the dry ice ethanol bath (~488 sec), the dry ice ethanol bath temperature was very close to -20°C, or the warmest temperature that could be used to reduce the
product temperature to -20°C. The blast freezer provided mean times to freeze of 1,080 sec, and no alternative settings of the blast freezer were available.

- *Times to Freeze Between 1,080 and 4,860 sec:* The blast freezer provided the mean time to freeze of 1,080 sec, and the manual defrost freezer provided the mean time to freeze of 4,860 sec.

The TPA hardness time to freeze data for the potato slices did not include the TPA hardness for the blanched potato slices. These TPA hardness points were included in the illustrations as a reference for comparison. The y-intercept from the regression of the established model would predict the TPA hardness of an unfrozen slice.

6.1 **Influence of Thaw Methods on Textural Quality of Frozen Foods**

Preliminary data comparing the TPA hardness of potato slices after freezing and thawing at ambient temperature to similar data for slices thawed in a microwave oven suggested that the thawing method might impact potato texture (Appendix E). Thawing in the microwave oven seemed to cause slightly higher TPA hardness values than thawing at ambient temperature. However, after the experimental methods were standardized, the results did not illustrate the same differences as the preliminary results. The primary change in methods was limiting the temperature of the potato slices during microwave heating to a temperature no higher than the ambient temperature used for ambient temperature thawing.
Figure 6.1 presents TPA hardness data and times to freeze for slices thawed at ambient temperature and slices thawed in a microwave oven.

These plots demonstrate that the relationship between TPA hardness and time to freeze for thawing in the microwave oven is very similar to the relationship for TPA hardness and time to freeze for thawing at ambient temperature. For both relationships, times to freeze ranged from 69 to 4,860 sec, and TPA hardness decreased exponentially as time to freeze increased. At the most rapid time to freeze in this range (69 sec), the potato slice thawed in the microwave oven retained a TPA hardness of 19,511 g as compared to the potato slice thawed at ambient temperature at 20,679 g. When the time to freeze was much longer (488 sec), the TPA hardness had decreased to 8,213 g for the potato slice.
thawed in the microwave oven as compared to 8,474 g for the potato slice thawed at ambient temperature. When the times to freeze are increased to 1,080 sec, the TPA hardness of the potato slice thawed in the microwave oven decreased to a mean of 2,516 g, while the slices thawed at ambient temperature had slightly higher mean TPA hardness of 3,913 g. Lastly, when the time to freeze was 4,860 sec, the TPA hardness was 4,428 g after the thawing in the microwave oven as compared to 3,436 g after thawing at ambient temperature.

A regression analysis was conducted on the combined sets of data in the presence of a dummy variables (Da, Db, and Dc) and an indicator (i) in order to determine whether the conditions had an impact on the value of the parameters. Each parameter was paired with a corresponding indicator that showed whether or not the parameter differed in a statistically significant way between the conditions. All three parameters were tested simultaneously using the following equation:

\[
TPA \text{ hardness} = (a + Da \times i) \times e^{(b+Db\times i) \times time} + (c + Dc \times i)
\]

Table 6.1 shows the best-fit values for the six parameters.
Table 6.1 Parameter Estimates with Dummy Variable for Condition (Microwave versus Ambient Thaw)

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Estimate</th>
<th>Approximate Std Error</th>
<th>Approximate 95% Confidence Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>a</td>
<td>23295.3</td>
<td>1434.3</td>
<td>20452.9 26137.6</td>
</tr>
<tr>
<td>b</td>
<td>-0.0031</td>
<td>0.0005</td>
<td>-0.0040 -0.0022</td>
</tr>
<tr>
<td>c</td>
<td>3215.8</td>
<td>585.1</td>
<td>2056.3 4375.3</td>
</tr>
<tr>
<td>Da</td>
<td>-661.1</td>
<td>2165.1</td>
<td>-4951.9 3629.7</td>
</tr>
<tr>
<td>Db</td>
<td>0.0003</td>
<td>0.0007</td>
<td>-0.0010 0.0016</td>
</tr>
<tr>
<td>Dc</td>
<td>-53.4991</td>
<td>1122.6</td>
<td>-2278.2 2171.2</td>
</tr>
</tbody>
</table>

Because the 95% confidence interval for all three D parameters includes the null hypothesis (D=0), the thawing condition had no statistically significant impact on any parameter. Therefore, it could be concluded that there was no statistical difference between the TPA hardness of potato slices after thawing at ambient temperature as compared to slices after thawing in the microwave oven. As a result, during presentation of all subsequent results (except for stack experiments) the data for experiments with potato slices thawed in a microwave oven have been pooled with data for potato slices thawed at ambient temperatures.

6.2 Influence of Freezing on Potato Texture – Best Fit Model

Once the data was pooled, in order to identify the most appropriate regression model for the relationship between the TPA parameters and time to freeze, several models were explored. The following models were evaluated: linear, logarithmic, polynomial, moving average, exponential and power. The analysis and plots leading up to the best-fit model can be found in Appendix B. Based on this analysis, an updated exponential model with a non-zero asymptote provided the best fit to the data. This was chosen as a best fit
because it provided a logical y-intercept, it reasonably modeled times to freeze and it resulted in an asymptote in line with minimum TPA hardness seen in data.

For this model, the following equation was fit to the data:

\[ hardness = a \times e^{b \times \text{time}} + c \]

This model can be interpreted as follows:

- \( c \) is the asymptote; the lowest average TPA hardness of the potato slice as a function of time to freeze.
- \( a+c \) is TPA hardness at time=0, as predicted by the model.
- \( b \) represents a rate of change in TPA hardness as a function of time to freeze.

Table 6.2 presents the \( a, b \) and \( c \) parameter estimates for TPA hardness data and times to freeze for thawed 6 mm potato slices.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Estimate</th>
<th>Approximate Standard Error</th>
<th>Approximate 95% Confidence Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>( c )</td>
<td>3159</td>
<td>498.5</td>
<td>2172</td>
</tr>
<tr>
<td>( a )</td>
<td>22929</td>
<td>1028</td>
<td>20892</td>
</tr>
<tr>
<td>( b )</td>
<td>-0.00294</td>
<td>0.000320</td>
<td>-0.00357</td>
</tr>
</tbody>
</table>

These figures estimated the rate of change in TPA hardness as a function of time to freeze as \(-0.00294 \, 1/s\), the asymptote, the lowest TPA hardness of the potato slice as a function
of time to freeze, as 3,159 g, and the y-intercept as 26,088 g. The non-zero asymptote was an important factor in choosing this model as a best fit. The regression model arising from these parameters is plotted against actual data in Figure 6.2.

![Figure 6.2 TPA Hardness of Frozen and Thawed Potato Slices Versus Time to Freeze](image)

\[ \text{hardness} = 22929 \times e^{-0.00294 \times \text{time}} + 3159 \]

**Figure 6.2 TPA Hardness of Frozen and Thawed Potato Slices Versus Time to Freeze**

When the curve is plotted, first there is a rapid exponential decline with increasing time to freeze. This is followed by a constant level of TPA hardness, representing maximum damage or minimum TPA hardness, as represented by the asymptote.

The model does a good job of fitting the actual data. The predicted y-intercept value at 26,088 g, was in line with the non-frozen blanched slices that ranged from 23,099 – 26,769 g with an average of 24,483 g. This also fell within the range established by the
95% confidence limits for the regression model of 23,064 to 29,133 g. The model also successfully captured the asymptotic behavior of the data, with an asymptote value at 3,159 g. This value fell within the range of the TPA hardness values at 1,080 sec from 2,482 - 5,367 g with a mean of 3,602 g and of the TPA hardness values at 4,860 sec from 2,215 – 5,377 g with a mean of 3436 g.

The significance of this relationship for frozen food manufacturers is the trade off between time to freeze and textural quality. According to the established model, a time to freeze of 200 sec would result in TPA hardness of about 16,000 g, whereas reducing the time to freeze to 100 sec would result in much higher TPA hardness of about 20,000 g. On the other hand, a time to freeze of 2,400 sec would provide a TPA hardness of around 3,200 g, and reducing the time to freeze to 1,080 sec would not provide a noticeable difference in TPA hardness, with a value of about 4,000 g.

This relationship was further validated with a visual confirmation of scanning electron microscope micrographs of potato slices under different conditions. Figure 6.3 shows a slice of a raw potato, a just blanched potato, a potato frozen and thawed with a time to freeze of 70 sec, and a potato frozen and thawed with a time to freeze of 1,080 sec, all at x250.
The raw slice appears to have very smooth and straight cell wall structure. In the just blanched slice, a modest impact of heating is apparent, where there is slight wrinkling, but the cell wall structure still appears to be very much intact. The slice frozen with a time to freeze of 70 sec looks very similar to the blanched slice, which is expected since it was at a similar TPA hardness level to the blanched counterpart. Lastly, in the slice with a time to freeze of 1,080 sec, considerable damage is apparent. The cell walls appear visibly deflated and disruptions of the cell walls are visible. This was also congruent with the TPA data showing minimum TPA hardness retention at this point, having reached the asymptote of 3,519 g.

6.3 **Analysis of TPA Parameters**

While TPA hardness is the most commonly used parameter to evaluate product quality in scientific literature (Buggenhout and others 2006, Chandrasekaran and others 2013, Schmidt and Ahmed 1971), data on TPA chewiness, TPA springiness, TPA cohesiveness, TPA resilience and TPA gumminess were also collected during this study. In this section, all TPA parameters were measured for 6 mm potato slices.
TPA cohesiveness has been defined as a measure of how well a sample responds to a second deformation relative to how it behaved during the first deformation (Can-Am Instruments Ltd. n.d.). The data for the parameter of TPA cohesiveness were fit to a linear curve.

\[ TPA \, Cohesiveness = 3 \times 10^{-6}x + 0.7024 \]

This model predicted a y-intercept value at 0.7024 and a rate of increase in TPA cohesiveness as an increase time to freeze of $3 \times 10^{-6}$. The results in Figure 6.4 present the plot for TPA cohesiveness versus time to freeze.

![Figure 6.4 TPA Cohesiveness Versus Time to Freeze](image)

With a $R^2$ value of 0.00849, there appeared to be no obvious trend with this data. This indicated that time to freeze had little impact on the relationship between the first and
second compression. Overall, TPA cohesiveness did not appear to be affected during freezing.

The next parameter evaluated was TPA gumminess. TPA gumminess has been defined as a measure of energy required to disintegrate semi-solid food (Can-Am Instruments Ltd. n.d.). The data for the parameter of TPA gumminess was fit to an exponential fit adjusted with a non-zero asymptote.

\[
TPA \text{ Gumminess} = 17,517 \times e^{-0.00345 \times \text{time}} + 2,316
\]

The results in Figure 6.5 present TPA gumminess versus time to freeze.

**FIGURE 6.5 TPA GUMMINESS VERSUS TIME TO FREEZE**

This model predicted a y-intercept value at 19,833 g, which was in line with the non-frozen blanched slices that range from 15,854 – 18,585 g, with an average of 16,968 g.
The rate of decrease in TPA gumminess as a function of time to freeze was \(-0.00345 \text{ l/s}\), and the asymptote, the lowest TPA gumminess of the potato slice as a function of time to freeze, was 2,316 g. This indicated that there was a clear relationship between TPA gumminess and time to freeze, where the TPA gumminess decreased as time to freeze increased until it hit an asymptote where maximum textural damage had occurred. This was very similar to the trend with TPA hardness and time to freeze.

The next parameter evaluated was TPA springiness. TPA springiness did not follow in the same trend as the TPA hardness and TPA gumminess. TPA springiness has been defined as a measure of how well a sample springs back after the first compression (Can-Am Instruments Ltd. n.d). This parameter was fit to the following linear curve.

\[
TPA \text{ Springiness} = 2 \times 10^{-5}x + 0.7733
\]

The results in Figure 6.6 present TPA gumminess versus time to freeze.
The model predicts a y-intercept value at 0.7733, which was in line with the non-frozen blanched slices that range from 0.74 – 0.82, with an average of 0.78. The rate of increase in TPA springiness as a function of time to freeze was $2 \times 10^{-5}$. Overall, the plot illustrated a slight increase in TPA springiness with time to freeze. This could indicate that with increased damage caused by longer times to freeze, the potato slices more easily returned to their original damaged form. However, with an $R^2$ value of 0.22172, this was not a reliable trend.

The next parameter evaluated was TPA chewiness. TPA chewiness has been defined as the amount of energy needed to chew solid food before swallowing (Can-Am Instruments Ltd. n.d.). The TPA chewiness data followed a similar trend to the TPA hardness and

**Figure 6.6 TPA Springiness Versus Time to Freeze**

![Graph showing TPA Springiness versus Time to Freeze with data points and a linear fit line. The equation for the linear fit is $Springiness = 2E-05x + 0.7733$.](image)
TPA gumminess data. The data for the parameter of TPA chewiness was fit to an exponential fit adjusted with a non-zero asymptote.

\[ TPA \text{ Chewiness} = 13,050 \times e^{-0.00348 \times \text{time}} + 1,989 \]

The results in Figure 6.7 present TPA chewiness versus time to freeze.

![Figure 6.7 TPA Chewiness versus Time to Freeze](image)

This model predicted a y-intercept value at 15,039 g, which was only slightly higher than the TPA hardness values for the non-frozen blanched slices, which range from 11,971 – 14,868 g and have an average of 13,319 g. The rate of decrease in TPA chewiness as a function of time to freeze was -0.00348 1/s, and the asymptote, the lowest TPA chewiness of the potato slice as a function of time to freeze, was 1,989 g. This indicated
that there was a clear relationship between TPA chewiness and time to freeze, where the TPA chewiness decreased as time to freeze increased until it hit an asymptote where maximum textural damage had occurred. This was very similar to the trend with TPA hardness and TPA gumminess.

The next parameter evaluated was TPA resilience. TPA resilience has been defined as how well a product recovers after deformation (Can-Am Instruments Ltd. n.d.). The TPA resilience data followed a similar trend to the TPA hardness, TPA gumminess, and TPA chewiness data. The data for the parameter of TPA resilience was fit to an exponential fit adjusted with a non-zero asymptote.

\[
TPA\ Resilience = 0.3131 \times e^{-0.00214 \times time} + 0.3484
\]

The results in Figure 6.8 present TPA resilience versus time to freeze.
This model predicted a y-intercept value at 0.6615, which was slightly higher the non-frozen blanched slices that range from 0.53 – 0.58, with an average of 0.56. The rate of decrease in TPA resilience as a function of time to freeze was -0.00214 and the asymptote, the lowest TPA resilience of the potato slice as a function of time to freeze, was 0.3484. Overall, the plot illustrated a decrease in TPA resilience with time to freeze. This indicated that there was a clear relationship between TPA resilience and time to freeze, where the TPA resilience decreased as time to freeze increased until it hit an asymptote where maximum textural damage had occurred. This indicates that with increased times to freeze, the samples have a more difficult time recovering after

**Figure 6.8 TPA Resilience Versus Time to Freeze**

![Graph showing TPA resilience versus time to freeze with equation: $Resilience = 0.3131 \cdot e^{-0.00214 \cdot time} + 0.3484$.]
deformation. This was very similar to the trend with TPA hardness, TPA gumminess, and TPA chewiness.

Since TPA hardness, TPA gumminess, TPA chewiness, and TPA resilience exhibited the most obvious trends with the data, the characteristics of these plots were compared in the following table.

**Table 6.3 Comparison of Fit Equation Characteristics for TPA Hardness, TPA Chewiness and TPA Gumminess**

<table>
<thead>
<tr>
<th></th>
<th>Y-Intercept</th>
<th>Rate of Hardness Retention as a Function of Time to Freeze</th>
<th>Asymptote</th>
</tr>
</thead>
<tbody>
<tr>
<td>TPA Hardness</td>
<td>26,088 (g)</td>
<td>-0.00294 (1/s)</td>
<td>3,159 (g)</td>
</tr>
<tr>
<td>TPA Chewiness</td>
<td>15,039 (g)</td>
<td>-0.00348 (1/s)</td>
<td>1,989 (g)</td>
</tr>
<tr>
<td>TPA Gumminess</td>
<td>19,833 (g)</td>
<td>-0.00345 (1/s)</td>
<td>2,316 (g)</td>
</tr>
<tr>
<td>TPA Resilience</td>
<td>0.6615 (gs/gs)</td>
<td>-0.00214 (1/s)</td>
<td>0.3484 (gs/gs)</td>
</tr>
</tbody>
</table>

Table 6.3 indicates that TPA hardness had the highest y-intercept value at 26,088 g with TPA gumminess following second at 19,833 g, then TPA chewiness at 15,039 g and lastly TPA resilience at 0.6615 gs/gs. At these values, TPA gumminess is approximately 75% the size of TPA hardness, TPA chewiness is approximately 58%, and TPA resilience is negligible at 2.5x10^{-6} %. The same trend was apparent with the asymptote levels, with TPA hardness at 3,159 g, TPA chewiness at 1,989 g, TPA gumminess at 2,316 g and TPA resilience at 0.3484 gs/gs. This indicated that the parameter of TPA hardness would provide the highest level of granularity in comparing different samples. The other notable difference were the differences between the rate of TPA parameter retention as a function of time to freeze. Hardness had the smallest value at -0.00294 1/s.
TPA chewiness and TPA gumminess had very similar values that were slightly larger, -0.00384 \(1/s\) and -0.00345 \(1/s\) respectively, and TPA resilience had the lowest value at -0.00214 \(1/s\).

After analyzing the six textural parameters, TPA hardness remained the best way to represent the data for several reasons. First, all the TPA parameters that exhibited the clearest trends (TPA hardness, TPA chewiness, TPA gumminess and TPA resilience), used force 2 (TPA hardness) in their equations, so it is no surprise that these parameters had similar trends. In addition, TPA hardness provided the highest level of granularity with the largest scale making it easier to compare different sample values. Next, TPA hardness could be closely correlated to cell wall damage, the predominant mechanism of freezing damage. The collapse of the cell wall, would directly result in decreased TPA hardness retention. Lastly, as mentioned previously, it is the most commonly used parameter to evaluate produce quality in scientific literature.

6.4 Impact of Freezing on Structure

In subsequent experiments, daikon radish samples were frozen using the same protocol. It is important to note, that unlike the potato samples, daikon radish samples were not blanched. For the daikon radish samples, raw samples were frozen and then thawed at ambient temperature.
The daikon radish data was fit to the same exponential model with non-zero asymptote previously described for potatoes. Table 6.4 presents the a, b and c parameter estimates for TPA hardness data and times to freeze for thawed 6 mm daikon radish slices.

**Table 6.4 Exponential Model Fit (6 mm Daikon Radish Slices)**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Estimate</th>
<th>Std. Error</th>
<th>95% Confidence Interval</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Lower Bound Upper Bound</td>
</tr>
<tr>
<td>a</td>
<td>16858</td>
<td>1304.886</td>
<td>14218.876</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>19497.640</td>
</tr>
<tr>
<td>b</td>
<td>-0.00132</td>
<td>0.0002705</td>
<td>-0.0018680</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>-0.0007739</td>
</tr>
<tr>
<td>c</td>
<td>3475</td>
<td>1161.269</td>
<td>1126.522</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>5824.299</td>
</tr>
</tbody>
</table>

These figures estimated the rate of change in TPA hardness as a function of time to freeze as \(-0.00132 \, 1/s\), the asymptote, the lowest TPA hardness of the potato slice as a function of time to freeze, as 3,475 g, and the y-intercept as 20,333 g. The regression model arising from these parameters is plotted against actual data in Figure 5.9.
In comparison to the potato slices, daikon radish slices were damaged far more when comparing frozen to unfrozen TPA hardness. At a time to freeze of 75 sec (the shortest freezing time conducted), from a raw slice at a TPA hardness level of about 32,000 g to 18,000 g, whereas for the potatoes, the TPA hardness level started lower, at about 24,500 g, but only decreased to 20,000 g.

A regression analysis was conducted on the combined sets of data in the presence of dummy variables (Da, Db, and Dc) and an indicator (i) in order to determine whether the sample type had an impact on the value of the parameters. Each parameter was paired with a corresponding indicator that showed whether or not the parameter differed in a
statistically significant way between the sample types. All three parameters were tested simultaneously using the following equation:

$$TPA\ hardness = (a + Da \cdot i) \cdot e^{(b + Db \cdot i) \cdot time} + (c + Dc \cdot i)$$

Table 6.5 shows the best-fit values for the six parameters.

**Table 6.5 Parameter Estimates With Dummy Variable For Condition (6 mm Potato versus 6 mm Daikon Radish)**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Estimate</th>
<th>Approximate Std Error</th>
<th>Approximate 95% Confidence Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>a</td>
<td>23029.1</td>
<td>1012.1</td>
<td>21029.1 – 25029</td>
</tr>
<tr>
<td>b</td>
<td>-0.0028</td>
<td>0.0003</td>
<td>-0.0035 – -0.0022</td>
</tr>
<tr>
<td>c</td>
<td>2868.2</td>
<td>574.5</td>
<td>1732.8 – 4003.5</td>
</tr>
<tr>
<td>Da</td>
<td>-6170.8</td>
<td>1630</td>
<td>-9392 – -2949.6</td>
</tr>
<tr>
<td>Db</td>
<td>0.0015</td>
<td>0.0004</td>
<td>0.0007 – 0.0023</td>
</tr>
<tr>
<td>Dc</td>
<td>607.2</td>
<td>1274.1</td>
<td>-1910.5 – 3125</td>
</tr>
</tbody>
</table>

Variable Da, Db and Dc, in Table 6.5 show the significance of the thaw condition on the parameters a, b and c. The ‘a’ and ‘b’ parameters were deemed significant because the 95% confidence interval did not include the null hypothesis, D=0. The ‘c’ parameter was deemed insignificant because the 95% confidence interval included the null hypothesis, D=0. This analysis indicated that the y-interval for both sets of data were statistically different. For the potatoes, the y-intercept was 26,088 g and for the daikon radishes, the y-intercept was higher at 27,489 g. This was likely because the raw daikon radishes also started off at a higher TPA hardness than the raw and blanched potatoes. This analysis also indicated that for both sets of data the rate of TPA hardness retention as a function of time to freeze ‘b’ was statistically different. This seemed apparent in the values, with
potatoes at -0.00294 and daikon radishes at less than half this rate at -0.00132. This suggested that with the daikon radishes, a large portion of the damage occurs with the initial freezing. However, once frozen, the damage occurs less quickly for the daikon radishes in comparison to the potatoes. Lastly, the asymptotes (‘c’) or the lowest average TPA hardness as a function of time to freeze, were not considered to be statistically different, with potatoes at 3,159 g and 3,475 for daikon radishes.

In general, the overall relationship between TPA hardness and time to freeze for daikon radishes generated a very similar curve to what was seen for the potato. These results illustrate that the same relationship between TPA hardness and time to freeze is apparent in samples other than potatoes.

6.5 Influence of Slice Thickness on Freezing Impact

To explore the influence of thickness, 4.5 mm slices were frozen using the same protocol. A new set of data, at a different slice thicknesses, provided a mechanism to further study the effect of freezing damage on the cellular structure of the potato. The hypothesis would be that slices with the same time to freeze would have the same level of damage as indicated by the same TPA hardness value regardless of slice thickness.

Differences in slice thickness presented a challenge to direct comparisons. Table 6.6 presents data on slice thickness versus TPA hardness for raw potato slices.
Table 6.6 Slice Thickness Versus TPA Hardness for Raw Potato Slices

<table>
<thead>
<tr>
<th>Slice Thickness (mm)</th>
<th>Mean Hardness (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>19,467</td>
</tr>
<tr>
<td>4.5</td>
<td>25,726</td>
</tr>
<tr>
<td>6</td>
<td>28,544</td>
</tr>
</tbody>
</table>

Tests on 3 mm, 4.5 mm and 6 mm raw unfrozen slices indicated a positive correlation between thickness and reported TPA hardness. This means that as the slice thickness increased, the mean TPA hardness increased. The 3 mm slices had a mean TPA hardness of 19,467 g, the 4.5 mm slices had a mean TPA hardness of 25,726 g, and the 6 mm slices had a mean TPA hardness of 28,544 g. This indicates that the TPA was sensitive to slice thickness.

In order to account for the TPA’s sensitivity to slice thickness, the TPA data needed to be normalized. Normalization was done by dividing the TPA hardness values by the mean TPA hardness of the blanched slices for their respective thickness. Figure 6.10 presents the fits for the normalized TPA hardness versus time to freeze for the 4.5 mm and 6 mm potato slices.
As indicated, the 4.5mm data show a very similar shape and trend to the 6mm data. A regression analysis was conducted on the combined sets of data in the presence of dummy variables (Da, Db, and Dc) and an indicator (i) in order to determine whether the different slice thicknesses had an impact on the value of the parameters. Each parameter was paired with a corresponding indicator that showed whether or not the parameter differed in a statistically significant way between the slice thicknesses. All three parameters were tested simultaneously using the following equation:

\[
TPA \text{ Hardness} = (a + Da \ast i) \ast e^{(b+Db\ast i)\ast \text{time}} + (c + Dc \ast i)
\]
Table 6.7 shows the best-fit values for the six parameters.

**Table 6.7 Parameter Estimates with Dummy Variable for Slice Size (4.5 mm versus 6 mm)**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Estimate</th>
<th>Approximate Std Error</th>
<th>Approximate 95% Confidence Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>a</td>
<td>1.1884</td>
<td>0.0326</td>
<td>1.1241 - 1.2526</td>
</tr>
<tr>
<td>b</td>
<td>-0.0024</td>
<td>0.0005</td>
<td>-0.0034 - 0.0015</td>
</tr>
<tr>
<td>c</td>
<td>0.1012</td>
<td>0.0348</td>
<td>0.0324 - 0.1700</td>
</tr>
<tr>
<td>Da</td>
<td>-0.2518</td>
<td>0.0538</td>
<td>-0.3579 - 0.1457</td>
</tr>
<tr>
<td>Db</td>
<td>-0.0005</td>
<td>0.0006</td>
<td>-0.0016 - 0.0006</td>
</tr>
<tr>
<td>Dc</td>
<td>0.0279</td>
<td>0.0406</td>
<td>-0.0522 - 0.1079</td>
</tr>
</tbody>
</table>

Variable Da, Db and Dc, in Table 6.7 show the significance of slice size on the a, b and c parameters. Variables b and c were deemed insignificant because the 95% confidence interval included the null hypothesis, D=0. This means that after normalization, the asymptotes (‘c’) of the 4.5mm (0.1012) and 6mm (0.129) slices were not statistically significant. This indicated that the maximum damage due to freezing was not impacted by the thickness of the slices. The rates of TPA hardness retention as a function of time to freeze (‘b’) were also not considered statistically different for the 6mm (-0.00294) and the 4mm slices (-0.00244). In addition, the 6 mm slices reached maximum TPA hardness damage before the 4.5 mm slices as expected. Lastly, the variable ‘a’, which corresponds closely to the y-intercept value, with a value of 1.29 for the 4.5 mm slices and was deemed significant at a 95% confidence interval. This was likely a result of the normalization factor.
6.6 TPA Hardness versus Characteristic Freeze Time Relationship

In order to gain a better understanding of the influence of freezing on damage to the potato structure, a simulation of the temperature distribution history was used (Mannapperuma and Singh 1989). The simulation parameters for these points are listed in Table 6.8. The rate of heat transfer and air velocity values for the different times to freeze were previously determined in Table 5.2. The brine and/or air temperatures were altered to achieve the target times to freeze. The simulation time was the time it took to get from 22.22°C (ambient temperature) to -20°C.

<table>
<thead>
<tr>
<th>Simulation Time (sec)</th>
<th>121.2</th>
<th>120</th>
<th>508.3</th>
<th>498.6</th>
<th>1099</th>
<th>1103</th>
<th>4074</th>
<th>3516</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness (mm)</td>
<td>4.5</td>
<td>6</td>
<td>4.5</td>
<td>6</td>
<td>4.5</td>
<td>6</td>
<td>4.5</td>
<td>6</td>
</tr>
<tr>
<td>Rate of Heat Transfer W/m²°K</td>
<td>403</td>
<td>403</td>
<td>136</td>
<td>136</td>
<td>3258</td>
<td>3258</td>
<td>183</td>
<td>183</td>
</tr>
</tbody>
</table>

The points for these simulations were chosen to cover the range of times to freeze used in the experiments up until the times to freeze for minimum TPA hardness retention.

Using the parameters for the two 120 sec points from Table 6.8, the simulation provided the temperature profile of eleven points within both the 4.5 mm and 6 mm potato slice from the center (0 mm from center) to the exterior (3 mm from center or 2.25 mm from center). The temperature distribution history for one slice (in this case a 6 mm slice) is illustrated in Figure 6.11.
This illustration demonstrates that the times to freeze are not uniform throughout a sample. The points by the outside of the slice, have steeper initial curve, and reach a temperature of 0°C and -20°C earlier than the points by the center of the slice, which stay at higher temperatures throughout the freezing process and have overall longer times to freeze. This indicates that measuring one point in a sample does not provide a complete representation for a sample. The same trend was visible in the output for the 4.5 mm slice that can be found in Appendix C.

While the simulation provided the total freezing time from ambient temperature to -20°C, a more useful value was the characteristic freezing time, when the initial ice
crystals form to when approximately 95% of the water has frozen (Heldman 2007). For
potatoes, the initial freezing temperature for the potato is -1.2°C and the temperature
when 95% of the water is frozen is about -16°C. This is the portion of the freezing
process where the majority of the damage due to ice crystals is likely to occur. It was
anticipated that simulation data would provide information into ice crystal damage
happening internally during freezing. Literature states that lower characteristic freeze
times translate into smaller ice crystals (Bevilacqua and others 1979). For these
simulations, characteristic freeze time was defined as the time it took to get from -1°C to
-16.4°C. This provided a directly comparable measure for slices of two thicknesses.
Note that, while both slices include measures at 11 points, the points did not represent
equal distances since they are different sized slices. This is indicated by the different
“distances from center of slice” for the 4.5 mm and 6 mm slices. Table 6.9 presents the
characteristic freeze times for 11 points from the center to the outside of a 6 mm potato
frozen with a time to freeze of 120 sec.
Table 6.9 Distribution of Characteristic Freeze Times within 6 mm Slice with Time to Freeze of 120 sec

<table>
<thead>
<tr>
<th>Distance From Center of Slice (mm)</th>
<th>0</th>
<th>0.3</th>
<th>0.6</th>
<th>0.9</th>
<th>1.2</th>
<th>1.5</th>
<th>1.8</th>
<th>2.1</th>
<th>2.4</th>
<th>2.7</th>
<th>3.0</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time to -1°C (sec)</td>
<td>98</td>
<td>91</td>
<td>81</td>
<td>72</td>
<td>63</td>
<td>54</td>
<td>46</td>
<td>37</td>
<td>27</td>
<td>16</td>
<td>7</td>
</tr>
<tr>
<td>Time to -16.4°C (sec)</td>
<td>116</td>
<td>116</td>
<td>116</td>
<td>115</td>
<td>115</td>
<td>114</td>
<td>113</td>
<td>112</td>
<td>110</td>
<td>107</td>
<td></td>
</tr>
<tr>
<td>Characteristic Freeze Time (sec)</td>
<td>18</td>
<td>25</td>
<td>35</td>
<td>44</td>
<td>52</td>
<td>61</td>
<td>68</td>
<td>76</td>
<td>85</td>
<td>94</td>
<td>100</td>
</tr>
</tbody>
</table>

This table demonstrates that the characteristic freeze times increase going from the center (18 sec) to the outside (100 sec).

Table 6.10 presents the characteristic freeze times for 11 points from the center to the outside of a 4.5 mm potato frozen with a time to freeze of 121.2 sec.

Table 6.10 Distribution of Characteristic Freeze Times within 4.5 mm Slice with Time to Freeze of 121.2 sec

<table>
<thead>
<tr>
<th>Distance From Center of Slice (mm)</th>
<th>0</th>
<th>0.225</th>
<th>0.45</th>
<th>0.675</th>
<th>0.9</th>
<th>1.125</th>
<th>1.35</th>
<th>1.575</th>
<th>1.8</th>
<th>2.025</th>
<th>2.25</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time to -1°C (sec)</td>
<td>87</td>
<td>82</td>
<td>74</td>
<td>66</td>
<td>58</td>
<td>50</td>
<td>43</td>
<td>36</td>
<td>29</td>
<td>20</td>
<td>12</td>
</tr>
<tr>
<td>Time to -16.4°C (sec)</td>
<td>115</td>
<td>115</td>
<td>115</td>
<td>114</td>
<td>114</td>
<td>114</td>
<td>113</td>
<td>113</td>
<td>112</td>
<td>111</td>
<td></td>
</tr>
<tr>
<td>Characteristic Freeze Time (sec)</td>
<td>28</td>
<td>33</td>
<td>41</td>
<td>48</td>
<td>56</td>
<td>64</td>
<td>71</td>
<td>77</td>
<td>84</td>
<td>92</td>
<td>99</td>
</tr>
</tbody>
</table>

Once again, this table demonstrates that the characteristic freeze times increase going from the center (28 sec) to the outside (99 sec).
Since characteristic freeze time is correlated with ice crystal size, it would be expected that the larger ice crystals would be towards the outside of the slices and the smaller ice crystals would be towards the center. The characteristic freeze time data from these two tables suggest that the most damage was occurring on the outside of the slices.

Table 6.11 presents summary statistics of characteristic freeze times and TPA hardness for various simulation times.

**Table 6.11 Summary Statistics of Mass Average Characteristic Freeze Times and TPA Hardness for Various Simulation Times**

<table>
<thead>
<tr>
<th>Simulation Time (s)</th>
<th>121.2</th>
<th>120</th>
<th>508.3</th>
<th>498.6</th>
<th>1099</th>
<th>1103</th>
<th>4074</th>
<th>3516</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness (mm)</td>
<td>4.5</td>
<td>4</td>
<td>4.5</td>
<td>6</td>
<td>4.5</td>
<td>6</td>
<td>4.5</td>
<td>6</td>
</tr>
<tr>
<td>Rate of Heat Transfer W/m²K</td>
<td>403</td>
<td>403</td>
<td>136</td>
<td>136</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Air Velocity (m/s)</td>
<td></td>
<td></td>
<td>3258</td>
<td>3258</td>
<td>183</td>
<td>183</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mass Average Characteristic Freeze Time (s)</td>
<td>63.0</td>
<td>59.9</td>
<td>246</td>
<td>266</td>
<td>594</td>
<td>617</td>
<td>2247</td>
<td>1997</td>
</tr>
<tr>
<td>Hardness (Fit)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Normalized as % of Blanched</td>
<td>0.987</td>
<td>0.787</td>
<td>0.445</td>
<td>0.345</td>
<td>0.182</td>
<td>0.166</td>
<td>0.1013</td>
<td>0.1290</td>
</tr>
</tbody>
</table>

The output of the data suggests that the mass average characteristic freeze time seems to be proportionate to the time to freeze, or in this case simulation time, falling in the range of 48-56%. More importantly, within each time to freeze, the values of mass average characteristic freeze of both slices are close. This raises the possibility that the two values are not statistically significant. The same relationship appears to be true with TPA hardness. This supports the hypothesis that two slices with the same characteristic freezing time would have the same level of damage as indicated by the same TPA hardness value regardless of slice thickness.
In order to consider the differences in time required to reach -20°C for the 4.5 mm slices versus the 6 mm slices, an additional set of simulations were run holding the simulation conditions constant for the 4.5 mm and 6 mm slices.

Table 6.12 shows the summary statistics of characteristic freeze times and TPA Hardness for various simulation conditions for 4.5 mm and 6 mm slices.

**Table 6.12 Summary Statistics of Mass Average Characteristic Freeze Times and TPA Hardness for Various Simulation Conditions**

<table>
<thead>
<tr>
<th>Simulation Time (s)</th>
<th>75.5</th>
<th>120</th>
<th>327.6</th>
<th>498.6</th>
<th>614.1</th>
<th>1103.1</th>
<th>1968</th>
<th>3516</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness (mm)</td>
<td>4.5</td>
<td>6</td>
<td>4.5</td>
<td>6</td>
<td>4.5</td>
<td>6</td>
<td>4.5</td>
<td>6</td>
</tr>
<tr>
<td>Brine/Air (°C)</td>
<td>-53.78</td>
<td>-53.78</td>
<td>-29.78</td>
<td>-29.78</td>
<td>-38.78</td>
<td>-38.78</td>
<td>-51.78</td>
<td>-51.78</td>
</tr>
<tr>
<td>Rate of Heat Transfer W/m² K</td>
<td>403</td>
<td>403</td>
<td>136</td>
<td>136</td>
<td>403</td>
<td>403</td>
<td>136</td>
<td>136</td>
</tr>
<tr>
<td>Air Velocity (m/s)</td>
<td>0.905</td>
<td>0.905</td>
<td>0.051</td>
<td>0.051</td>
<td>0.905</td>
<td>0.905</td>
<td>0.051</td>
<td>0.051</td>
</tr>
<tr>
<td>Mass Average Characteristic Freeze Time (s)</td>
<td>37.6</td>
<td>59.9</td>
<td>177</td>
<td>266</td>
<td>345.5</td>
<td>617</td>
<td>1116</td>
<td>1997</td>
</tr>
<tr>
<td>Hardness (Fit)</td>
<td>1.0881</td>
<td>0.7872</td>
<td>0.6356</td>
<td>0.3451</td>
<td>0.3662</td>
<td>0.1656</td>
<td>0.1109</td>
<td>0.1290</td>
</tr>
</tbody>
</table>

In this table, the same conditions (brine/air, rate of heat transfer, and air velocity) were run for the 4.5 mm and 6 mm slices. This indicated that under the same conditions, the reduced slice thickness always resulted in a smaller mass average characteristic freeze time and almost always resulted in a higher TPA hardness retention. The only exception was the last condition (brine/air at -51.78°C, rate of heat transfer at -51.78 W/m²K, and air velocity at 0.051m/s). In this case, the TPA hardness retention was slightly higher for the 6 mm slice (0.1290) than for the 4.5 mm slice (0.1109). However, since these data points are in the region where TPA hardness does not change with time to freeze, it has already been established that the two TPA hardness values for the two slice thicknesses are not statistically different.
6.7 Influence of Location During Thawing on TPA Texture of Frozen Potatoes

When microwaving, the degree and rate of heating is strongly correlated to the dielectric and ionic properties of a product. One of the biggest challenges with microwaving frozen foods is localized overheating, known as run-away heating. Uneven heating can be due to several factors, yet the dielectric properties have arguably the most significant effect (Soproni and others 2008). In the microwave heating of frozen foods, the effect of the dielectric properties is exceptionally significant because of the large difference in values for ice and water. The dielectric constant ($\varepsilon'$) of water is 80, as compared to the dielectric constant ($\varepsilon'$) of ice at 3, and the relative dielectric loss constant ($\varepsilon''$) of water is 10, as compared to the relative dielectric loss constant ($\varepsilon''$) of ice at 0.003 (Singh and Heldman 2009). So, when the ice begins to melt to water, the water components heat at a much more rapid rate than the remaining ice components, thus leading to runaway heating (Singh and Heldman 2009). It is known that shorter times to freeze result in smaller, more uniform ice crystal formation than longer times to freeze that are known to result in larger less uniform ice crystal formation (Joslyn 1961). As a result of these differences, one hypothesis would be that shorter times to freeze would result in more uniform microwave heating and thus less runaway heat than longer times to freeze.

In these experiments, one purpose was to determine whether runaway heat would have an effect on adjacent potato slices. Specifically whether thawing potato slices frozen at short times to freeze adjacent to potato slices frozen at long times to freeze would result in differences in final thaw texture of the individual slices.
The experiments in Figure 6.12 show individual slices frozen with a time to freeze of either 1,080 or 65 sec that were then assembled in stacks of five slices prior to being thawed in the microwave oven. In these experiments, the top, middle and bottom slice were frozen with a time to freeze of 1,080 sec and the second and fourth slice were frozen with a time to freeze of 65 sec.

**FIGURE 6.12 TPA HARDNESS OF FROZEN POTATO SLICES IN STACKS, MIXED WITH TIMES TO FREEZE OF 1,080 SEC AND 65 SEC, MICROWAVE THAWED**

The Tukey test grouped all slices with a time to freeze of 65 sec frozen as ‘a’, and all slices with a time to freeze of 1,080 sec frozen as group ‘b’ with no overlap. Therefore, it could be concluded that there was no statistical difference between the TPA hardness of potato slices frozen with a time to freeze of 1,080 sec in this stack formation, regardless of being in the top, middle or bottom position. It could also be concluded that there was
no statistical difference between the TPA hardness of potato slices frozen with a time to freeze of 65 sec in this stack formation, regardless of being in the second or fourth position.

In the next set of experiments, altering the order of the individual slices within the stacks changed the stack type. The experiments in Figure 6.13 show individual slices frozen with a time to freeze of either 1,080 or 65 sec that were then assembled in stacks of five slices prior to being thawed in a microwave oven. In these experiments, the composition of the stacks was changed so that the top, middle and bottom slice were frozen with a time to freeze of 65 sec and the second and fourth slice were frozen with a time to freeze of 1,080 sec.
In this new arrangement, the Tukey test grouped all slices with a time to freeze of 65 sec frozen as ‘a’, and all slices with a time to freeze of 1,080 sec frozen as group ‘b’ with no overlap. Therefore, it could be concluded that there was no statistical difference between the TPA hardness of potato slices frozen with a time to freeze 65 sec in this stack formation, regardless of being in the top, middle or bottom position. It could also be concluded that there was no statistical difference between the TPA hardness of potato slices frozen with a time to freeze of 1,080 sec in this stack formation, regardless of being in the second or fourth position.
In the next set of experiments, the stack type was changed by altering the order of the individual slices within the stacks, and by using an even longer time to freeze, 4,860 sec instead of a 1,080 sec time to freeze. The experiments in Figure 6.14 show individual slices frozen with a time to freeze of either 65 sec or 4,860 sec that were then assembled in stacks of five slices prior to being thawed in a microwave oven. In these experiments, the top, middle and bottom slice were frozen with a time to freeze of 65 sec and the second and fourth slice were frozen with a time to freeze of 4,860 sec.

![Figure 6.14 TPA Hardness of Frozen Potato Slices in Stacks, Mixed with Times to Freeze of 65 Sec and 4,860 Sec, Microwave Thawed](image)

Even with a longer time to freeze, the Tukey test grouped all slices with a time to freeze of 65 sec frozen as ‘a’, and all slices with a time to freeze of 4,860 sec frozen as group ‘b’ with no overlap. Therefore, it could be concluded that there was no statistical difference
between the TPA hardness of potato slices frozen with a time to freeze of 65 sec in this stack formation, regardless of being in the top, middle or bottom position. It could also be concluded that there was no statistical difference between the TPA hardness of potato slices frozen with a time to freeze of 4,860 sec in this stack formation, regardless of being in the second or fourth position.

In the next set of experiments, altering the order of the individual slices within the stacks changed the stack type. The next set of experiments was conducted by changing the order of the slices within the stacks. The experiments in Figure 6.15 show individual slices frozen with a time to freeze of either 65 sec or 4,860 sec that were then assembled in stacks of five slices prior to being thawed in a microwave oven. In these experiments, the top, middle and bottom slice were frozen with a time to freeze of 4,860 sec and the second and fourth slice were frozen with a time to freeze of 65 sec.
Once again, the Tukey test grouped all slices with a time to freeze of 65 sec frozen as ‘a’, and all slices with a time to freeze of 4,860 sec frozen as group ‘b’ with no overlap. Therefore, it could be concluded that there was no statistical difference between the TPA hardness of potato slices frozen with a time to freeze 4,860 sec in this stack formation, regardless of being in the top, middle or bottom position. It could also be concluded that there was no statistical difference between the TPA hardness of potato slices frozen with a time to freeze of 65 sec in this stack formation, regardless of being in the second or fourth position.

The next set of experiments was conducted by changing the composition of the stacks to include only one type of time to freeze. The experiments in Figure 6.16 show individual
slices frozen with a time to freeze of 65 sec that were then assembled in stacks of five slices prior to being thawed in a microwave oven. In these experiments, all slices were frozen with a time to freeze of 65 sec.

For these experiments, the Tukey test grouped all slices as ‘a’. Therefore, it could be concluded that there was no statistical difference between the TPA hardness of potato slices frozen with a time to freeze 65 sec in this stack formation, regardless of being in the top, second, middle, fourth or bottom position.
The next set of experiments was conducted by changing the type of freeze for the individual slices in stacks that included only one type of time to freeze. The experiments in Figure 6.17 show individual slices frozen with a time to freeze of 1,080 sec that were then assembled in stacks of five slices prior to being thawed in a microwave oven. In these experiments, all slices were frozen with a time to freeze of 1,080 sec.

![Figure 6.17 TPA Hardness of Frozen Potato Slices in Stacks, Uniform with Times to Freeze of 1,080 Sec, Microwave Thawed](image)

For these experiments, the Tukey test grouped all slices as ‘b’. Therefore, it could be concluded that there was no statistical difference between the TPA hardness of potato slices frozen with a time to freeze 1,080 sec in this stack formation, regardless of being in the top, second, middle, fourth or bottom position.
The same outcome was obtained when stacks were thawed at ambient temperature. This indicated that thawing in the microwave using the procedures in these experiments did not affect the end TPA hardness any differently than thawing at ambient temperature. As a result of needing to compare a potato slice after thawing at ambient temperature to a slice after being thawed in a microwave oven, the final temperature of the potato slices after thawing in the microwave oven was carefully limited to ensure both conditions were arriving at the same end temperatures. Consequently, the impact of runaway heating was limited in these experiments. The results of these experiments can be found in Appendix D.

Since there was no effect of position (top, second, middle, fourth, bottom) or type of stack (mixed times to freeze stacks and uniform times to freeze stacks) within the thaw types, all the stack data could be pooled for further analysis. Figure 6.18 shows the Tukey test results done on the pooled data.
The Tukey test grouped all 65 sec slices as ‘a”, and all 1,080 sec slices as group ‘b’ regardless of thaw type. This indicated that there was no effect of the type of thaw (ambient versus microwave thaw). These results confirm that in these experiments, any run-away-heat in the microwave did not affect the final TPA hardness of the slice samples.

Of the four conditions studied (stack type, position in stack, thaw type, time to freeze), time to freeze was the only statistically significant predictor of final texture.

Within time to freeze, the results in Figure 6.18 were also consistent with results discussed previously comparing potato slices thawed in a microwave oven to potato
slices thawed at ambient temperature. When comparing the 1,080 sec times to freeze to the 4,860 sec times to freeze, all 1,080 sec and 4,860 sec slices were grouped as ‘b’ indicating that these treatments were not statistically significant from one another. This confirmed the asymptotic behavior indicating that in this range, close to maximum TPA hardness damage had already occurred.

These results illustrate that the ultimate TPA hardness of a frozen food (after thawing) in these experiments is impacted by the times to freeze within the product structure during the freezing process rather than the position, stack type or thawing method (microwave vs. ambient air). Longer times to freeze (up until the point of minimum TPA hardness retention) can be correlated to collapse of the cell wall, decreasing the end TPA hardness. In contrast, shorter times to freeze, can be correlated to cell walls that are more intact, resulting in higher TPA hardness.
Chapter 7: Conclusion

The results of this investigation provide the basis for the following conclusions:

- Evaluation of potato texture following freezing and thawing requires careful control of sample size and temperature, during and following the freezing process.

- Evaluation of potato texture during TPA measurement also requires careful control of sample size and thickness.

- When evaluating the impact of thawing on frozen potato texture, the use of microwave energy for thawing required careful control of end-point temperature.

- A comparison of thawing at ambient temperature and thawing in a microwave oven indicated the thawing method did not impact potato texture in a significant manner when end point temperatures were the same.

- A statistical relationship between time to freeze and TPA hardness was established for potato slices. A relationship between TPA hardness and time to freeze for potato slices was also illustrated through regression models.

- A model for the relationship between TPA hardness and time-to-freeze for 6mm potato slices was established as $TPA\,Hardness = 22,929 \exp[-0.00294 \times time] + 3,159$, where $a = 22,929$, $b = 0.00294$ and $c = 3,159$. In this model, $c$ is the asymptote, $b$
is the rate of TPA hardness retention as a function of time to freeze and $a+c$ is the y-intercept.

- **TPA Hardness** is the most appropriate parameter to evaluate textural damage caused by the freeze-thaw process as compared to the other TPA parameters.

- The model describing the relationship of TPA hardness and time to freeze includes a non-zero asymptote. For a 6 mm potato slice, maximum structural damage due to freezing and thawing occurs within a time to freeze of 3,513 sec, or a mass average characteristic freeze time of 1,997 sec. For a 4.5 mm potato slice, maximum structural damage due to freezing and thawing occurs within a time to freeze of 4,160 or a mass average characteristic freeze time of 2,247.

- The time to freeze did not impact the texture after thawing when potato slices with significantly different times to freeze were placed in close proximity during thawing.
References


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Appendix A: Preliminary Data on TPA Hardness versus Time to Freeze

Figure A.1 presents early TPA hardness data for 6 mm potato slices thawed in a microwave oven versus time to freeze.

![Graph showing TPA hardness vs. time to freeze]

**Figure A.1 Overall TPA Hardness with Various Times to Freeze (Ambient Thaw)**

This TPA hardness data demonstrated no statistical difference in TPA hardness of times to freeze ranging from 1,080 – 6,900 sec. While the extra long freeze at 25,860 sec appeared to have notably higher levels of TPA hardness, the standard deviation was very
large. Since this data were acquired prior to establishing a complete protocol, there could have been other factors of influence.

The most interesting part of this TPA data was the stark difference in TPA hardness between times to freeze of 65 and 1,080 sec. In further studies, more experiments were done to focus in on this range.
Appendix B: Finding a Best Fit Model

Results of the investigation illustrated a clear relationship between time to freeze and TPA hardness. When the initial TPA data were plotted, several fits were attempted using Excel’s trend line feature. The best-fit curve among the available options was the power fit presented in Figure B.1.

![Figure B.1 TPA Hardness versus Time to Freeze (Excel Power Fit)](image)

This curve generated an $R^2$ value of 0.867. The $R^2$ value close to one indicates a direct relationship with a good regression.
Next, additional data was added at a longer time to freeze of 4,860 sec. Figure B.2 presents the same fit with the added data. A dashed line represents the original fit, and a solid line represents the new fit.

Figure B.2 TPA HARDNESS VERSUS TIME TO FREEZE, INCLUDING LONGER TIMES TO FREEZE (EXCEL POWER FIT)

Once more data were added, it was apparent that the updated power fit with the more data was not ideal because it asymptoted at zero instead of at the value of maximum damage.

Figure B.3 shows this same updated power fit alone, with a dashed line representing the lowest point an asymptote could occur according to the data.
FIGURE B.3 TPA HARDNESS OF THAWED FROZEN POTATO SLICES WITH LONG FREEZE

The dashed line illustrated that this function did a poor job of capturing the asymptotic behavior of TPA hardness at longer times to freeze. Since the blast freeze TPA hardness data were not found to be statistically different than the conventional freeze TPA hardness data, it could be inferred that close to the maximum textural damage occurred by 1,080 sec. The experimental data showed that the potato slices reach a minimum TPA hardness of 2,062 g, while the power fit continued to slope down to zero.

In an effort to better mirror actual behavior, functions with non-zero asymptotes were explored. The most straightforward way to introduce a non-zero asymptote was to add an additional parameter, ‘c’, to the fit to the existing power function:
hardness = a * time^b + c

This resulted in the parameters in Table B.1.

**Table B.1 Power Fit Parameter Estimates**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Estimate</th>
</tr>
</thead>
<tbody>
<tr>
<td>a</td>
<td>116156</td>
</tr>
<tr>
<td>b</td>
<td>-0.4108</td>
</tr>
<tr>
<td>c</td>
<td>-2135</td>
</tr>
</tbody>
</table>

Unfortunately, the optimum fit for this equation, resulted in a negative value for c, the asymptote. Since the data were suggestive of a positive, not negative asymptote, this provided strong indication that this was not the best way to model the data. This is also evident from a visual analysis of the curve as is demonstrated in Figure B.4.
The new non-zero asymptote curve cuts even lower through the existing data, resulting in an even worse fit to the data.

Next, exponential functions were reviewed. Exponential functions also presented an attractive candidate as a model to fit this data. In addition to asymptotic behavior, the exponential function generated a fixed rate of TPA hardness retention as a function of time to freeze. While this model provided an extremely poor fit with an asymptote at zero, it was a strong candidate for a revised model. So, as a final model, the following equation was fit to the data:
\[ hardness = a \cdot e^{b \cdot time} + c \]

This fit provided an asymptote at 3,249 g, which was in line with expectations. The plot and detailed discussion can be found in section 6.1
Appendix C: Temperature Distribution Curves for Simulation Data

The temperature distribution history for a 4.5 mm slice is illustrated in Figure C.1.

**Figure C.1 Temperature Distribution for Half of a 4.5mm Slice with Time To Freeze of 121 sec**

As found with the 6 mm slice temperature distribution, this illustration demonstrated that the times to freeze are not uniform throughout a sample. The points by the outside of the slice, have steeper initial curve, and reach a temperature of 0°C and -20°C earlier than the points by the center of the slice, which stay at higher temperatures throughout the
freezing process and have overall longer times to freeze. This indicates that measuring one point in a sample does not provide a complete representation for a sample. In comparison to the 6 mm slice, these curves were closer together and covered a tighter temperature range.
Appendix D: Stacks Ambient Thaw

The experiments in Figure D.1 show individual slices frozen with a time to freeze of either 1,080 or 65 sec that were then assembled in stacks of five slices prior to being thawed at ambient temperature. In these experiments, the top, middle and bottom slice were frozen with a time to freeze of 1,080 sec and the second and fourth slice were frozen with a time to freeze of 65 sec.
The Tukey test grouped all slices with a time to freeze of 65 sec frozen as ‘a’, and all slices with a time to freeze of 1,080 sec frozen as group ‘b’ with no overlap. Therefore, it could be concluded that there was no statistical difference between the TPA hardness of potato slices frozen with a time to freeze of 1,080 sec in this stack formation, regardless of being in the top, middle or bottom position. It could also be concluded that there was no statistical difference between the TPA hardness of potato slices frozen with a time to freeze of 65 sec in this stack formation, regardless of being in the second or fourth position.
In the next set of experiments, altering the order of the individual slices within the stacks changed the stack type. The experiments in Figure D.2 show individual slices frozen with a time to freeze of either 1,080 or 65 sec that were then assembled in stacks of five slices prior to being thawed at ambient temperature. In these experiments, the top, middle and bottom slice were frozen with a time to freeze of 65 sec and the second and fourth slice were frozen with a time to freeze of 1,080 sec.

**Figure D.2 TPA Hardness of Frozen Potato Slices in Stacks, Mixed, with Times to Freeze of 65 Sec and 1,080 Sec, Ambient Thaw**

The Tukey test grouped all slices with a time to freeze of 65 sec frozen as ‘a’, and all slices with a time to freeze of 1,080 sec frozen as group ‘b’ with no overlap. Therefore, it could be concluded that there was no statistical difference between the TPA hardness of potato slices frozen with a time to freeze of 65 sec in this stack formation, regardless of
being in the top, middle or bottom position. It could also be concluded that there was no statistical difference between the TPA hardness of potato slices frozen with a time to freeze of 1,080 sec in this stack formation, regardless of being in the second or fourth position.

The next set of experiments was conducted by changing the composition of the stacks to include only one type of time to freeze. The experiments in Figure D.3 show individual slices frozen with a time to freeze of 65 sec that were then assembled in stacks of five slices prior to being thawed at ambient temperature. In these experiments, all slices were frozen with a time to freeze of 65 sec.
The Tukey test grouped all these slices as ‘a’. Therefore, it could be concluded that there was no statistical difference between the TPA hardness of potato slices frozen with a time to freeze 65 sec in this stack formation, regardless of being in the top, second, middle, fourth or bottom position.

The next set of experiments was conducted by changing the type of freeze of the stacks that included only one type of time to freeze. The experiments in Figure D.4 show individual slices frozen with a time to freeze of 1,080 sec that were then assembled in stacks of five slices prior to being thawed at ambient temperature. In these experiments, all slices were frozen with a time to freeze of 1,080 sec.
The Tukey test grouped all these slices as ‘b’. Therefore, it could be concluded that there was no statistical difference between the TPA hardness of potato slices frozen with a time to freeze 1,080 sec in this stack formation, regardless of being in the top, second, middle, fourth or bottom position.
Appendix E: Preliminary Data on the Influence of Thaw Methods

Figure E.1 presents early TPA hardness data for 6 mm potato slices thawed in a microwave oven versus time to freeze.

This TPA hardness data suggested that thawing potatoes in a microwave oven resulted in slightly higher TPA hardness levels than thawing potatoes at ambient temperature. This finding was further explored in section 6.1.