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Effects of Transportation Hazards on Package Performance and Food Product Shelf Life

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EFFECTS OF TRANSPORTATION HAZARDS ON PACKAGE PERFORMANCE AND FOOD PRODUCT SHELF LIFE

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by
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ABSTRACT

This research studied the effect of transportation hazards on food product shelf life and package performance. Studies were conducted to determine the effect of package headspace volume, product viscosity and storage temperature on package integrity. Finally, accelerated shelf life testing (ASLT) was utilized to determine how simulated transportation hazards affected the shelf life of a specific food product.

Institutional retort pouches containing either water or 5% starch solution were filled with varying amounts of headspace volume to determine if package headspace volume could aid in package performance during simulated engineering tests for packaged products. Fixed displacement vibration testing and compression testing of the pouches yielded no differences relative to headspace volume. Significant differences were noted during shock testing (free fall drop method) for the water filled pouches, however there was no observed effect for the 5% starch solution pouches. Results indicated that an increase in the headspace volume of a retort pouch does provide increased protection to transport hazards for low viscosity food products versus high viscosity food products.

Clear high-barrier retort pouches were filled with water and gas flushed with nitrogen. Headspace volumes utilized for this study were: 200cc and 400cc. Retort pouches were fitted with an OxyDot® as a non-invasively measurement of package headspace oxygen. The retort pouches were packaged inside regular slotted containers (RSC) and were subjected to laboratory simulated transportation performance tests for small parcel and unit load delivery systems. In addition to these transportation tests,

control samples of each variable were also used for comparison. Headspace oxygen levels were measured for the retort pouches for 63 days. Results indicated there were significant differences in all variables when comparing the transported samples to the control samples. When comparing the different headspace volumes, the 400cc pouches yielded less oxygen ingress into the pouch as compared with the 200cc pouches in the small parcel simulation ($P<0.05$). There were not significant differences for the two headspace volumes in the unit load simulation. Results from this study indicate for individual packaged products being distributed through small parcel supply chains, which are handled more vigorously than those being shipped palletized, the use of increased headspace volume may aid in protection of certain distribution channels.

Kettle cooked potato chips packaged in metallized oriented polypropylene bags were used to evaluate the effects of simulated transportation on product shelf life. Stressing food packages through laboratory simulated transportation hazards can create potential failures in package integrity that would not have normally been observed during typical ASLT test methods. This study showed that the transportation system for food packages has a great affect on package performance and ultimately food product shelf life. Outcomes from this study indicated that food product shelf life can be affected as a result of simulated transportation hazards.

DEDICATION

This dissertation is dedicated to my family, especially…

To Kristen, my best friend and the love of my life;

To Kylie, whose simple question, “Daddy, will you play with me?” is more important than any of the research questions proposed in this dissertation;

To Patrick, thanks for renewing my focus and for your sweet smile that makes me forgot about the long hours of writing;

And finally to my Mom, not a day goes by I do not think about you. Although you passed away during this process, your spirit lives on with me in all that I do. I cannot thank you enough for instilling in me the passion for education and the desire to never stop learning. You were always my biggest supporter, and it is a struggle not having you here to celebrate this great accomplishment with me. I know you are looking down over all of us and still guiding me to be a better father, husband, and person. I still often hear your boisterous laugh and it makes me smile – I love you.

It is with my greatest pleasure that I dedicate this accomplishment to you all!

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Accomplishments like this are never completed and should not be celebrated alone. I would like to share this will all my family and friends who have supported me throughout all the years. A very special thank you to my parents for believing in this journey and in me. A big thank you to my in-laws for the support since I was freshman moving into the dorms. Most importantly I would like to thank my wife and best friend, Kristen. You have been my rock throughout this incredible ride and I can’t wait to see what life has in store for us next. A finally to my children, Kylie and Patrick, I hope in time you come to see from this that if you put your mind to something and work hard, dreams come true.

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CHAPTER ONE

INTRODUCTION

Food science draws from many disciplines in an attempt to better understand food processes and ultimately improve food products for the general public (IFT, 2014). Food chemistry, a major aspect of food science, deals with composition and properties of food and the chemical changes it undergoes during handling, processing, and storage (Fennema, 1996). The approach undertaken by food scientists is to provide an analytical method to the handling, processing, and storage and determine how the properties of the food can be altered by these factors. Food technology is the application of food science to the selection, preservation, processing, packaging, distribution, and use of safe food (IFT, 2014).

Packaging is vital to world in which we live; about half of all packaging is used to package food (Robertson, 2006). Therefore, it is important to properly select packaging systems that will adequately contain and protect the product from processing through consumer use. To do this, the user must fully understand the capabilities of the package as well as determine how best to select the proper package for the environment in which it will be used and transported.

Packaging for consumer products is an area where supply and demand is constantly changing due to the development of an international food market and adaptation to new consumer, distribution, legal and technological requirements (Coles et al., 2003). Packaging plays a vital role in the preservation of the world's resources through the prevention of product spoilage and waste, and by protecting products until

they have performed their intended function. The fundamental roles of packaging are to contain, protect/preserve food, provide convenience, and inform the consumer. Through these fundamental roles of packaging, food waste can be minimized and the health of the consumer protected.

The understanding of food properties and packaging are well established in theory and research. Ideally, product formulation, package selection, and the distribution of the packaged product should be considered during the conception stage, but this is rarely the case in large corporate and manufacturing environments (Coles, et al., 2003). In most cases, the food product is matched with the proper packaging through material and product analysis, but the distribution of the final packaged product is not thoroughly understood or evaluated which can lead to product and package failures once it is launched for production and distribution to consumers. Packaged products traveling through the supply chain will be handled, dropped, vibrated, and compressed multiple times before they reach the consumer. Figure 1.1 shows potential food deterioration as a function of product distribution. As a result, packages can fail and lose their integrity, which can increase certain deterioration factors of the food product and ultimately adversely affect the product's predicted shelf life.

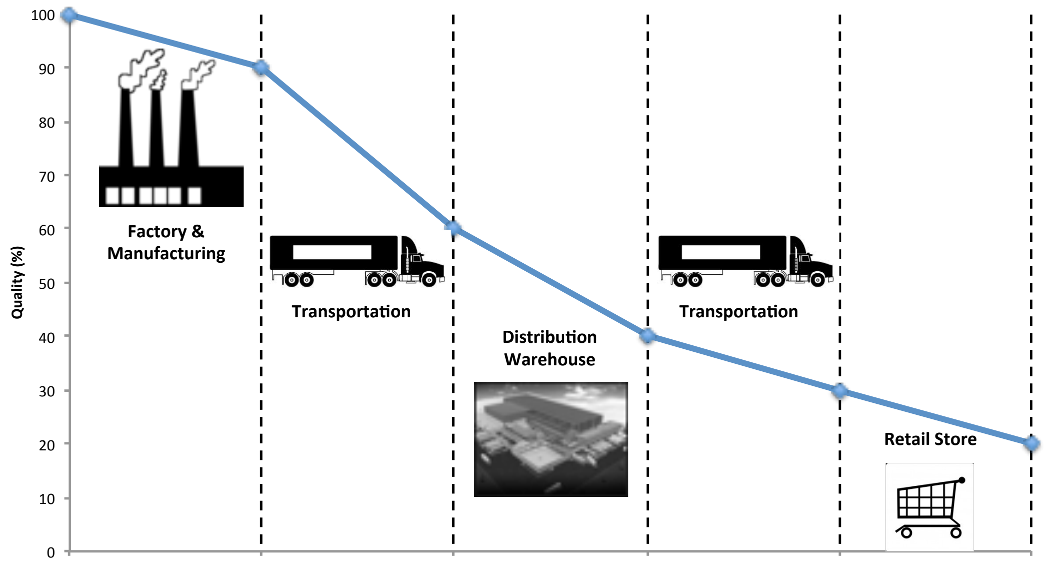

Figure 1.1 Estimated food deterioration through the supply chain (Heap et al., 1998)

This research aims to study packaged food products being transported through the supply chain and how the affect the quality of the food product and package can be altered as a result of the hazards they will experience while being distributed. In one study this research will evaluate the use of increased headspace volume inside retort pouches and determine if the increase in headspace volume can aid in protecting the package and product against known distribution hazards. Another area of this research study takes a look at the effects of simulated transport hazards on package structures and food attributes being evaluated under accelerated shelf life studies.

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CHAPTER TWO

REVIEW OF LITERATURE

Food Packaging

The food industry with its numerous and varied products utilize all the basic principles of packaging, which are to protect the contents, promote the product, inform the consumer, and produce convenience (Robertson, 2006). From the onset, the goal of food packaging was to provide preservation to seasonal food products. As the farm-oriented society began to urbanize, it became necessary to move and distribute food products from the location of harvesting and cultivating to where they were to be consumed, and the package had to offer protection during this process. The distribution process lengthened the time it took to get the product to the consumers and thus increasing the shelf life of food products was essential.

In general there are four main functions of a package: containment, protection, convenience, and communication. While these functions are often discussed independently, all four functions are interconnected and all must be assessed and considered simultaneously in the package development process (Robertson, 2006). Containment is a function so obvious it can often go unnoticed, however all products must be contained in order to function properly throughout the packages intended use. Protection can be summarized as the prevention of mechanical damage due to the hazards of distribution (Coles et al., 2003). Convenience refers to the packages intended use such as single serve or multi-use, or can be interpreted as assisting in the handling or use of a

package through the logistical movement of goods. Communication has a wide interpretation, but can largely refer to the packages ability to sell itself (brand recognition) or communicate important information to the consumer such as nutritional information, quantities, or other promotions (Coles et al., 2003).

Shelf Stable Foods and Packaging

The storage conditions food packages can be grouped into three categories: frozen, refrigerated, and shelf stable. Foods packages that are frozen or refrigerated require minimal processing, usually by hot water bath or blanched. This minimalizes the amount of entrapped gasses within the food and prevents further enzymatic activity that can reduce the quality of the food (Gavin & Weddig, 1995).

The Food and Drug Administration (FDA) regulates shelf stable foods according to their water activity (a_w) and acidity (pH). Briefly stated, water activity relates to the amount of moisture available in a food. Water activity will be discussed in more detail later is this review. One example of a food type having a water activity less than 0.85 is potato chips. Heat is applied to prepare the food and the water activity is reduced to a point were microorganisms cannot grow, thus they do not need additional thermal processing (Gavin & Weddig, 1995).

Shelf stable foods having a water activity greater, than 0.85, are regulated by the FDA's CFR part 113 (2012). Figure 2.1 depicts the relationship between the different classifications in shelf stable foods and their respective processing. High water activity is optimal for microorganisms of public health importance to grow (Blackstone & Harper, 1995). These foods must be additionally treated and/or processed to prevent microbial

growth. Shelf stable foods with high water activity greater that 0.85 are broken down according to their pH levels into three subcategories low acid, acidified, and naturally acidic (Gavin & Weddig, 1995).

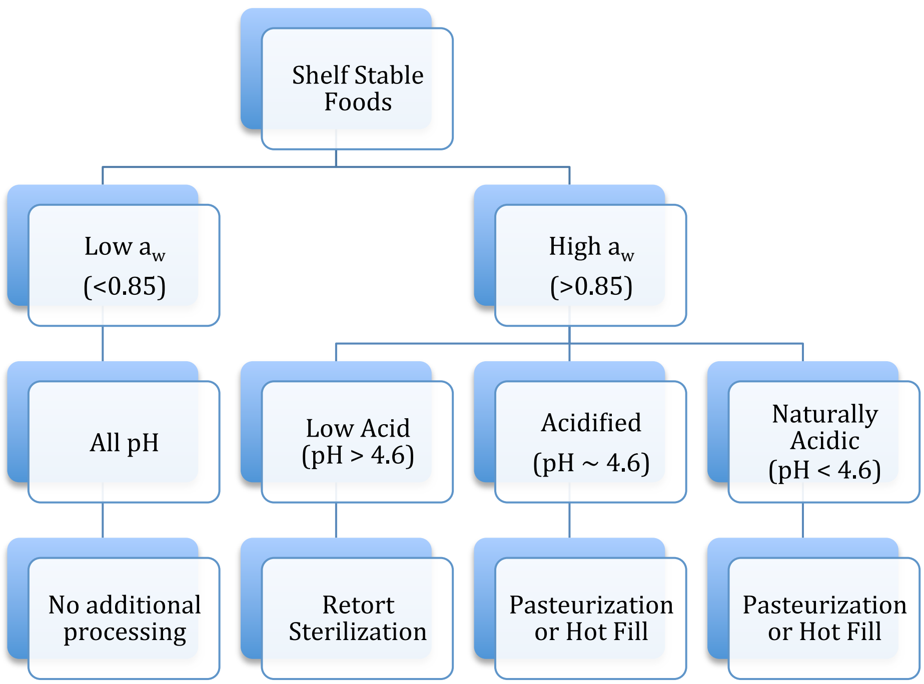

Figure 2.1. Relationship between a_w, pH, and processing

Processing, Packaging and Distribution of Acid, Acidified and Low Acid Foods

According to 21 CFR, foods are classed as acid, low acid or acidified depending on the natural acidity of each product. A product's acidity is measured based on a pH scale. If the raw or initial product has a pH above 4.6 it is considered a low acid food. If the pH is below 4.6 then the food is classified as an acid food. Acidified foods are low acid foods to which acid or acid ingredients are added to produce a final equilibrium pH of 4.6 or below. Equilibrium pH means the final pH measured in the acidified food after

all the components of the food have achieved the same acidity. Figure 2.2 displays the food class as well as regulations required by the FDA.

	And the equilibrium pH of the finished product is:	And/or the water activity (aw) is:	Then the product is:	And the processor needs to comply with:	Need to register and file process?	Need certification training?
If the product contains only naturally acid ingredients*	Equal or less than 4.6	Of any value	ACID	21 CFR 110	NO	NO (but strongly recom-mended)
If the product does contain any low-acid ingredients*	Equal or less than 4.6	Of any value	ACIDIFIED	21 CFR 110, 21 CFR 114	YES	YES
Regardless of product ingredients	Greater than 4.6	Greater than 0.85	LOW-ACID	21 CFR110, 21 CFR113	YES	YES
Regardless of product ingredients	Greater than 4.6	Less than 0.85	EXEMPTED FROM 21 CFR 113, 114 108.35 & 108.25	21 CFR 110	NO	NO (but strongly recom-mended)
Regardless of product ingredients	Of any value	Equal or less than 0.85	EXEMPTED FROM 21 CFR 113, 114 108.35 & 108.25	21 CFR 110	NO	NO (but strongly recom-mended)
If the finished product is refrigerated	Of any value	Of any value	EXEMPTED FROM 21 CFR 113, 114 108.35 & 108.25	21 CFR 110	NO	NO (but strongly recom-mended)

Figure 2.2. Guidelines for classifying food products based on FDA requirements (Clemson, 2000)

The pH value of 4.6 is important because it is the limiting factor for the growth of an extremely dangerous microorganism called *Clostridium botulinum*, which produces a potent toxin that causes the lethal disease botulism. The regulations concerning acidified

foods were established to assure the control and inhibition of the growth of *C. botulinum* by proper acidification and pH control, as this microorganism is very heat resistant and therefore it is not destroyed by pasteurization or cooking temperatures below 212°F.

For acids foods, such as jams and jellies, low pH cannot take the place of proper sanitation and care in manufacturing. Good Manufacturing Practices (GMPs) were designed and instituted by the federal government to assure that foods are manufactured and handled in a safe and sanitary manner to prevent adulteration. The conditions set forth in the regulation (21CFR110) must be met to operate this type of business.

Foods are heated by one of three mechanisms: convection, conduction, or convection/conduction (broken heating). Heat transfer in fluids generally takes place via convection. Convection currents are set up in the fluid because the hotter part of the fluid is not as dense as the cooler part, so there is an upward buoyant force on the hotter fluid, making it rise while the cooler, denser, fluid sinks. This type of heat transfer occurs quickly and refers to thin fluids such as juices, soups, and water. Conduction is the movement of heat by direct transfer of molecular energy within solids (Al-Baali and Farid, 2006). Conduction heating is a slower process utilized by products like pie fillings. Broken heating is a combination of convection and conduction heating where the food product is initially heated rapidly and then slowly. Products heated by this mechanism are products such as stews and gravies. Figure 2.3 graphically illustrates these three heating mechanisms.

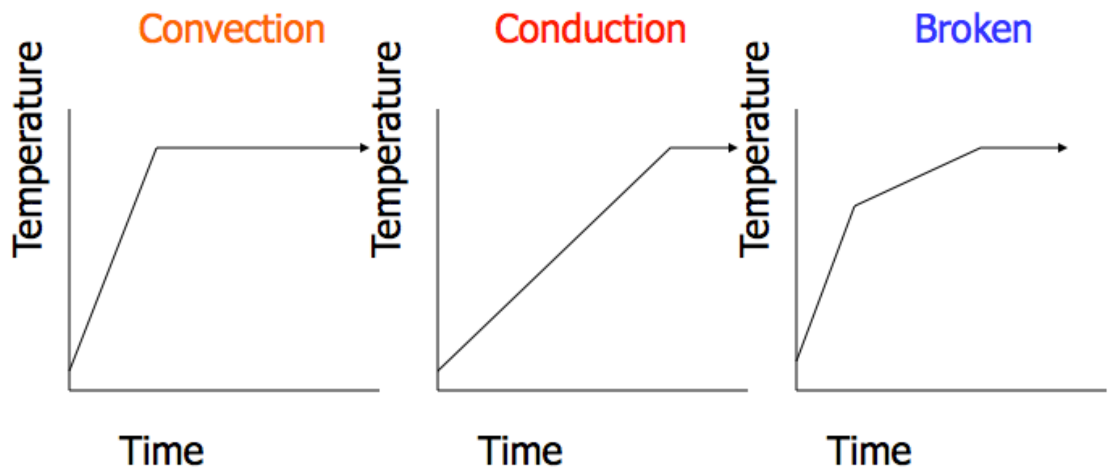

Figure 2.3. Product heating curves

Acidified foods are high-acid foods containing a significant percentage of ingredients that are naturally low-acid. A significant percentage is typically deemed to be 10 percent or more. No matter how the acidification is achieved, all the low-acid components of the food must take up enough acid to lower their pH values below 4.6 within 24 hours (McGlynn, 2010).

Some acidified foods are easy to identify. All pickled vegetables clearly fall into this category. However, some foods, salsas for example, contain amounts of low-acid ingredients such as peppers or onions that make them borderline acidified foods. In these cases, review by a Recognized Process Authority can determine the regulatory status of such foods. A food deemed to be naturally acid rather than acidified is termed a "formulated acid" food (Cornell, 2009).

Acidified foods have a pH 4.6 or lower; therefore, they need only be pasteurized to be safe. However, they are regulated more stringently than formulated acid foods, simply because any misstep in their production that reduces the ratio of acid to low-acid

ingredients in the formula could result in a food with ingredients that are not sufficiently acidified. Some portions of the food could then have a pH greater than 4.6. If this mistake is not caught, the result can be a deadly case of botulism.

For these reasons, producers of acidified foods must register their formula and processing procedures with the FDA, just as canners of low-acid foods do. However, the equipment and record keeping regulations (CFR 21 Part 114) are less involved.

In short, there are four basic requirements for acidified food processors, in addition to the usual requirements for facilities, record keeping and GMPs (McGlynn, 2010).

1. The facility where the food is produced will need to be registered with the FDA.

2. The processing procedures for each product sold need to be registered with the FDA.

3. Certain production records are required to be kept on hand. Simply stated, the processor needs to keep basic records on formulation, processing times and temperatures, pH tests, and container closure evaluation for each batch of product produced.

4. Receive the proper training by attending a "Better Process Control School". Process times and procedures for acidified foods must be reviewed and approved by a Recognized Process Authority.

Containers for acidified foods should be such that a hermetic seal is obtained. The best containers are cans and glass jars/bottles with metal caps lined with plastisol. With

these closures, a good vacuum is obtained. Vacuum is a good indicator of a hermetic seal and helps to maintain the quality of the product (Cornell, 2009).

As mentioned, a low-acid food is defined as a food having a pH of more than 4.6. This value is critical because of one particular bacterium, *C. botulinum*, which produces a dormant form called a spore. These spores are extremely hard to kill and may survive for many years, waiting for a chance to grow. An improperly processed can of food provides an ideal environment for *C. botulinum* spores to germinate, since the bacteria cannot survive in the presence of oxygen. *C. botulinum* produces an extremely potent neurotoxin, among the deadliest poisons known. Trace amounts of this toxin, which causes the food-borne illness known as botulism, are enough to kill. Fortunately, the spores of *C.botulinum* will not grow if the pH of a food is 4.6 or less. For low-acid foods with a pH value greater than 4.6, these spores must be killed by heating during the canning process. Because these spores are very heat resistant, canned low-acid foods must be pressure-cooked at high temperatures (>212°F) for long periods of time. Temperatures of 240°F or greater are commonly used and process times may range from 20 minutes to several hours. Most vegetables, meat and poultry foods fall into the low-acid food category. Because of the necessity of insuring the proper processing of low-acid foods, there are a number of detailed regulations governing their production. To can low-acid foods, the process must be registered with the FDA, use certified equipment, have received proper training at a "Better Process Control School" and keep extensive records as specified by federal regulations (21CFR Part113 for FDA-regulated foods and 9 CFR Part 318 for USDA-regulated foods) (Clayton, et al., 2012).

The canning process must also be reviewed and certified by a Recognized Process Authority. A Recognized Process Authority is any person recognized to have the training, experience and equipment needed to determine or verify the sufficiency of a thermal process. This person serves as an independent information resource for both the processor and regulatory agencies. Recognized Process Authorities may be affiliated with private companies, universities or trade organizations.

The packaging materials used for thermal processing are metal, glass, or rigid plastic containers and retort pouches. Cylindrical metal cans are the most widely used and in the highest production worldwide. Containers made of tin-plated steel are widely used, although lacquered tin-free steels are gradually replacing them (Holdsworth and Simpson, 2008). Glass jars are also widely used for packing foods and beverages. They have the advantage of very low interaction with the contents of the package and provide visibility of the product. However, they require more careful processing, usually in pressurized hot water, and handling. Rigid plastic containers are used in areas where the consumer is able to microwave the contents before consumption. The main requirement for the plastic material is that it must withstand the rigors of the heating and cooling process. The main plastic materials used for the thermal processing of foods are polypropylene (PP) and polyethylene terephthalate (PET) that are fabricated with an oxygen barrier such as ethylene-vinyl acetate (EVA), polyvinylidene chloride (PVdC) and polyamide (PA). Retort pouches are flexible laminated pouches that can withstand thermal processing temperatures and combine the advantages of metal cans and plastic packages. Retorts used in processing pouches can be batch or continuous, agitating or

non-agitating, and they require air overpressure to control pouch integrity (Blakistone, 2003).

Retort pouches have several advantages over traditional cans. The slender pouches are more easily disposed of than metal cans which are bulky. They are considerably lighter than metal cans providing added savings in transit costs. It often requires less heat to achieve commercial sterility, so the contents are generally of higher quality.

The amount of heat required to destroy microorganisms in a product can be determined through thermal death time (TDT) tests. TDT tests involve heating a known amount of microorganisms in a buffer solution or food at several temperatures and for several time intervals at each temperature. The results from the TDT tests are used to calculate D- and z-values. These values are used to define the heat resistance of the microorganisms of concern (Al-Baali and Farid, 2006).

In conducting TDT tests, the thermal characteristics (D- and z-values) of the microorganisms will be determined. The D-value is defined as the time at a particular temperature required to reduce a known number of microorganisms by 90% or to result in a 1-log reduction. The D-value decreases as the temperature increases, since it takes less time to destroy the microorganisms at the higher temperature (Principles, 2005). By determining the D-value at various temperatures, a z-value can be determined from the slope of the line that results from plotting the log of D-values versus temperature. The z-value, indicative of the change in the death rate based on temperature, is the number of

degrees between a 10-fold change (1 log cycle) in an organism's resistance (Principles, 2005). Both the D- and z-values are indirectly used to establish thermal processes.

Traditionally, a 12D process for spores of *C. botulinum* has been used to assure public health protection for low-acid canned foods. This has been based on historical data indicating that a heavy load of *C. botulinum* spores in a canned food product would be 10^{12} spores; therefore, a 12D reduction would provide a one-in-a-billion chance that a spore would survive in a canned food. For all practical purposes the 12D process is very conservative, as it is highly unlikely that spore loads of *C. botulinum* would approach these levels, especially in meat and poultry products. A typical D-value for *C. botulinum* spore destruction in many foods is approximately 0.2 minutes at 250°F; therefore, a 12D destruction would be ~2.4 (=12 x 0.2) minutes at 250°F. However, a value of 3 minutes is sometimes used to incorporate a margin of safety. In some products, the components of a food (or ingredients in a formulated food) can have adverse or beneficial effects on the thermal destruction of spores and will impact the D-values. Processing authorities refer to the times and temperature needed to inactivate *C. botulinum* as "minimum health" processes because this is what is necessary for public health protection (Principles, 2005).

In order to attain commercial sterility, a thermal process more strenuous than that required for public health protection must be provided. Commercial sterility means the condition achieved in a product by the application of heat to render the product free of microorganisms capable of reproducing in the food at normal non-refrigerated conditions of storage and distribution. These microorganisms are heat resistant spoilage organisms.

In order to compare thermal processes calculated for different temperatures, standard F_o value is assigned for each product. This F_o value is the time in minutes (at a reference temperature of 250°F and with a z = 18°F) to provide the appropriate spore destruction (minimum health protection or commercial sterility). As previously noted, using D- and z-values, this reference value at 250°F can be converted to other temperatures. Due to a variety of factors such as the influence of the food on the destruction of spores different foods will have different F_o values. F_o values are already established for many food products (Principles, 2005).

Products exported from foreign countries to the U.S. for marketing in the U.S., including both acidified and low-acid canned foods, are subject to inspection at the time of entry into the United States. Shipments which do not comply with U.S. laws and regulations will be detained at the port of entry. These products must be brought into compliance with the U.S. laws and regulations, destroyed, or re-exported. In addition to low acid food and acidified regulations (21 CFR 108.35, 108.25, 113, and 114), all other requirements of FDA's laws and regulations must be met before the products will be allowed entry into the United States. These include sanitation requirements during processing and storage, labeling, etc (FDA, 2012).

Retort Pouch

The retort pouch is a flexible package that can be sterilized at high temperatures. The retort pouch is a shelf-stable package system that does not require refrigeration. The low profile pouch configuration provides allows for microorganisms at the center of the package to be destroyed quickly. The low profile of the pouch also reduces over cooking

of the outer layer of product while the pouch is processed. The resulting product will have less likelihood of discoloration, flavor loss, texture change, or reduction in nutritional content (Hoddinott, 1975).

Institutional retort pouches are becoming more widely used as a replacement for the large metal cans due to their ease of use and disposal (Brooks, 2010). Retort pouches also help improve the safety for both consumers and employees. There are no sharp edges, as with cans, which eliminates cuts for both employees in the packaging plants and for the consumers opening the packages. It is easier for the consumer to open the package as opposed to cans because pouches do not require a can opener. Retort pouches allow for easy dispensing without utensils, and can even be equipped with re-closable features for repeated usage (Mykytiuk, 2002 and Gazdziak, et al., 2005).

Even though new pouch materials and shapes have appeared on the market, the protocol for defects has remained the same. The National Food Processors Association (NFPA) Bulletin 41-L defined the list of defects into two major classifications: critical and major (NFPA, 1989). Critical defects, which may compromise the seal integrity of the retort pouch include: channels and other leaks, cuts, fracture, non-bonding, notch leakers, puncture, swollen package, and wrinkles. Major defects are defined as those that result in a pouch not showing visible signs of loss of hermetic seal but are of such a magnitude that seal integrity may have been lost. Major defects include: abrasion, blister, compressed seal, contaminated seal, delamination, misaligned seal, seal creep, and wrinkle.

The institutional pouch chosen for this research is a 1.5 kg retort pouch constructed of the following individual polymers: aluminum oxide (AlOx) coated polyester (PET), biaxially oriented nylon (BON) and cast polypropylene (CPP). The individual layers are laminated with solvent-based aliphatic adhesives to produce the following structure: 12µm AlOx coated PET / 15µm BON / 100µm CPP.

Aluminum oxide is a common ceramic material sometimes referred to as one of the traditional ceramics. Traditional ceramics are those composed of clay minerals, as well as cement, and glass (Callister, 2007). Aluminum oxide, commonly referred to as alumina, possesses strong ionic interatomic bonding giving rise to its desirable material characteristics (Accuratus, 2002). Some of the key properties of aluminum oxide are its high strength and stiffness. Typical uses are wear pads, seal rings, or high voltage insulators. Due to its high strength, stiffness, and clarity, aluminum oxide can be used to coat films to increase barrier properties. Transparent thin film barrier materials based on oxides of aluminum, which give similar performance as an aluminization process without the opaque nature of the metal layer, were introduced to the packaging industry in the 1990's and continue to be developed for commercialization (Finson and Felts, 1994). An area where aluminum oxide coated films is seeing a steady growth is in the retort pouch lamination (Weaver, 2011).

British scientists, Whinfield and Dickson, patented polyester (PET) in 1941 as a result of Wallace Carother's incomplete research into investigating the result of mixing ethylene glycol and terephthalic acid (Jezek, 2006). In 1946, DuPont purchased the legal rights from Imperical Chemical Industries (ICI). Polyester was first introduced in the

U.S. in 1951 as fabric needing no ironing (Jezek, 2006). Many of the early applications of polyester were cotton replacements in clothing. One of PET's first uses in packaging came as a result of the Coca-Cola™ and Pepsi-Cola™ companies' interest in using plastic bottles for soft drinks (Robertson, 2006). The Pepsi-Cola™ company initially launched soft drinks in plastic PET bottles followed soon after by Coca-Cola™ and other soft drink producers.

Polyamide is better known under its original trade name, nylon. Nylon is a polymer first produced by Wallace Carothers, head of the DuPont lab, in 1935. Due to its property of thermo-plasticity, nylon was first used in the form of bristles for toothbrushes and later increased in popularity when used for stockings worn by women (Bellis, 2010). Nylon is typically found as the middle layer of a retort pouch structure due to its strength, toughness and burst resistance (Weaver, 2011). These enhanced properties enable flexible pouches to withstand the thermal retort processing.

Phillips Petroleum Company in Bartlesville, Oklahoma, entered the plastics business in 1951, following a discovery by researchers J. Paul Hogan and Robert L. Banks (Phillips, 1983). Hogan and Banks had been looking for ways to convert ethylene and propylene into components for gasoline when they found the catalyst that would transform these products into solid polymers (ACS, 2011). The solid polymers which formed were named polypropylene. Among the very first commercial polypropylene applications were monofilaments for fabrics, carpets, and ropes, as well as clear, biaxially oriented (BOPP) films used as replacements for cellophane in cigarette wrap, bread wrap, and retail packaging for shirts and other garments (Unknown, 2005).

Material Science and Properties of Retort Pouch

Due to the size of the molecules in polymers, they are often referred to as macromolecules. The atoms are bonded together by covalent bonds. For carbon chain polymers, the backbone of each chain is a string of carbon atoms (Callister, 2007). The carbons can be bonded to other carbons, but can also bond with other atoms or radicals in a chain. These long molecules are composed of structural entities called repeat units, which are successively repeated along the chain (Callister, 2007). The term monomer refers to the small molecule from which a polymer is synthesized.

Polyester is a class of polymers containing ester linkages with the following general formula (Selke et al., 2004):

$$H\text{-}O\text{-}(CO\text{-}R\text{-}R\text{-}CO\text{-}O\text{-}R'\text{-}O)_n\text{-}H$$

Polyethylene terephthalate (PET) is condensation polymer produced from para-xylene and ethylene. Figure 2.4 shows the structure of PET.

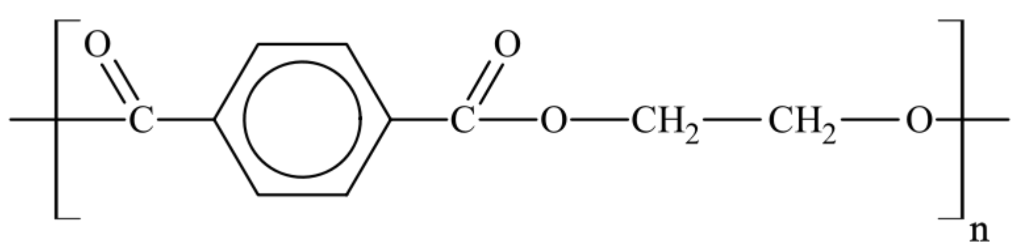

Figure 2.4. Chemical structure of PET (Rohieb, 2007)

PET is semicrystalline. The degree of crystallinity of PET is strongly influenced by processing condition. PET films and bottles typically have a limited degree of crystallinity, with small crystallites and excellent transparency (Selke et al., 2004).

Amorphous PET (APET) are modified to remain amorphous, while crystalline PET (CPET) have agents added which speed up and maximize crystallization. Being amorphous or crystalline has an affect not only on the transparency of the material, but also on the brittleness and its resistance to deformation. CPET is opaque white in appearance while APET is clear. CPET is much less subject to deformation under stress, especially at elevated temperatures, than is APET (Selke et al., 2004). CPET is more brittle at cold temperatures in comparison to APET. Table 2.1 provides typical properties for PET.

Table 2.1. Typical properties of polyethylene terephthalate (Selke et al., 2004)

T_g	73 - 80°C
T_m	245-265°C
Density	1.29-1.40 g/cm^3
Typical yield, 1 mil film	30 m^2/kg
Tensile strength	48.2-72.3 mPa
Tensile modulus	2,756-4,135 mPa
Elongation at break	30-3,000%
Tear strength, film	30 g/25 µm
WVTR	390-510 g µm/m^2 day at 37.8°C, 90% RH
O_2 permeability, 25°C	1.2-2.4 x 10^3 cm^3 µm/m^2 d atm
CO_2 permeability, 25°C	5.9-9.8 x 10^3 cm^3 µm/m^2 d atm
Water absorption, 0.32 cm thick, 24 h	0.1-0.2 %

Nylons are condensation polymers, linear thermoplastic polyamides that contain the amide group as a recurring part of the chain (Selke et al., 2004). Nylons are identified by the number of carbon atoms in the monomer. Figure 2.5 shows the structure of Nylon 6 which is the corresponding nylon being used for this research.

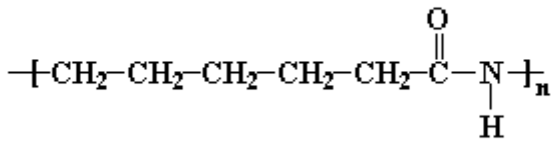

Figure 2.5. Chemical structure of Nylon 6 (Unknown, 2005)

Nylons are semicrystalline polymers with strong intermolecular forces due to the presence of H-bonding between the –C=O and HN- groups of adjacent molecules (Selke et al., 2004). These intermolecular forces are combined with crystallinity to yield tough, high melting thermoplastic materials. Different degrees of crystallinity can be obtained depending on the processing temperature and rate of quenching. An increased cooling rate provides a less crystalline nylon. This is a result of allowing less time for crystals to occur. Nylon films can also be oriented to increase certain properties. Biaxial orientation of nylon film leads to better crack resistance, mechanical properties, and barrier characteristics (Selke et al., 2004). Table 2.2 provides typical properties of Nylon 6.

Table 2.2. Typical properties of Nylon 6 (Selke et al., 2004)

T_g	60°C
T_m	210-220°C
Density	1.13-1.16 g/cm^3
Tensile strength	41.3-165 mPa
Elongation at break	300%
WVTR	3,900-4,300 g µm/m^2 day at 37.8°C, 90% RH
O_2 permeability, 25°C	470-1,020 cm^3 µm/m^2 d atm
CO_2 permeability, 25°C	3,900-4,700cm^3 µm/m^2 d atm
Water absorption, 0.32 cm thick, 24 h	1.3-1.9 %

Polypropylene (PP) is a thermoplastic produced by addition polymerization of propylene (Selke et al., 2004). Figure 2.6 illustrates the structure of PP. Polypropylene is

available as either a homopolymer or random copolymer. The type of polypropylene being utilized in this research project is a random copolymer. Random copolymer PP is similar in structure to isostatic PP (a type of homopolymer) with the addition of random ethylene groups. The addition of the ethylene groups results in lower crystallinity improving clarity and flexibility in comparison to the homopolymers. The increased clarity and flexibility make it a very attractive choice for clear pouch structures. The copolymer PP does not show stresses as visibly as the homopolymer when used in a clear pouch. Table 2.3 provides typical properties for PP.

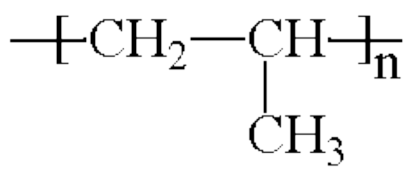

Figure 2.6. Chemical structure of PP (Unknown, 2005)

Table 2.3. Typical properties of polypropylene (Selke et al., 2004)

T_g	-10°C
T_m	160-175°C
Density	0.902 g/cm^3
Typical yield, 1 mil film	30 m^2/kg
Tensile strength	31-42 mPa
Tensile modulus	1,140-1,550 mPa
Elongation at break	30-3,000%
Tear strength, film	50 g/25 µm
Heat Sealability	Yes, 177-232°C
Transparency	Very good
Surface adhesivity	Low
WVTR	590 g µm/m^2 day at 37.8°C, 90% RH
O_2 permeability, 25°C	146,000 cm^3 µm/m^2 d atm
Water absorption, 0.32 cm thick, 24 h	0.01-0.03 %

Ceramics are composed of at least two elements, often times more. Therefore, the crystal structures of ceramics are generally more complex than those of metals (Callister, 2007). Aluminum oxide has predominantly ionic bonding (63%), meaning the structure may be thought of being composed of electrically charged ions. Figure 2.7 represents the structure of aluminum oxide. Table 2.4 illustrates the typical properties of aluminum oxide.

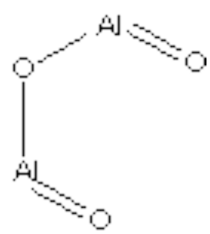

Figure 2.7. Chemical structure of aluminum oxide (ALS, 2014)

Table 2.4. Typical properties of aluminum oxide (Adams, 2002)

Mechanical	**Units of Measure**	**SI/Metric**
Density	gm/cc (lb/ft3)	3.69
Porosity	% (%)	0
Color	—	white
Flexural Strength	MPa (lb/in2x103)	330
Elastic Modulus	GPa (lb/in2x106)	300
Shear Modulus	GPa (lb/in2x106)	124
Bulk Modulus	GPa (lb/in2x106)	165
Poisson's Ratio	—	0.21
Compressive Strength	MPa (lb/in2x103)	2100
Hardness	Kg/mm2	1175
Fracture Toughness KIC	MPa•m1/2	3.5
Maximum Use Temperature (no load)	°C (°F)	1700
Thermal	**Units of Measure**	**SI/Metric**
Thermal Conductivity	W/m•°K (BTU•in/ft2•hr•°F)	18
Coefficient of Thermal Expansion	10–6/°C (10–6/°F)	8.1
Specific Heat	J/Kg•°K (Btu/lb•°F)	880

Manufacturing Process of Polymers

Condensation polymers such as polyester are formed from a monomer with a functional group of –COH. The first step in this reaction is the formation of esters from the diols and diacids (Selke et al., 2004). From these intermediates, the polymerization reaction proceeds. Because the first step is a faster reaction, the monomer is used up quickly (Selke et al., 2004). Condensation polymers generally have non-carbon atoms as part of the main backbone chain. Once polymerization has taken place, pellets are formed which can be used for the production of the PET film. The pellets pass through a drying system and are fed into a hopper. Once through the hopper, the pellets enter the extruder using friction to melt the polymer. Once the polymer is molten it is cast onto a series of rollers. Once the film is has been cast, it is wound and slit for the particular application. Figure 2.8 illustrates a PET film line summarizing the arrival of the pellets to the manufacturing process through the packaging of the rolls of film. Once the PET film has been slit to the correct roll width, it will have a thin layer of aluminum oxide coated on the film. The aluminum oxide is deposited on the films by evaporation using electron beams (Selke et al., 2004). The deposition of the aluminum oxide is still being researched at this time.

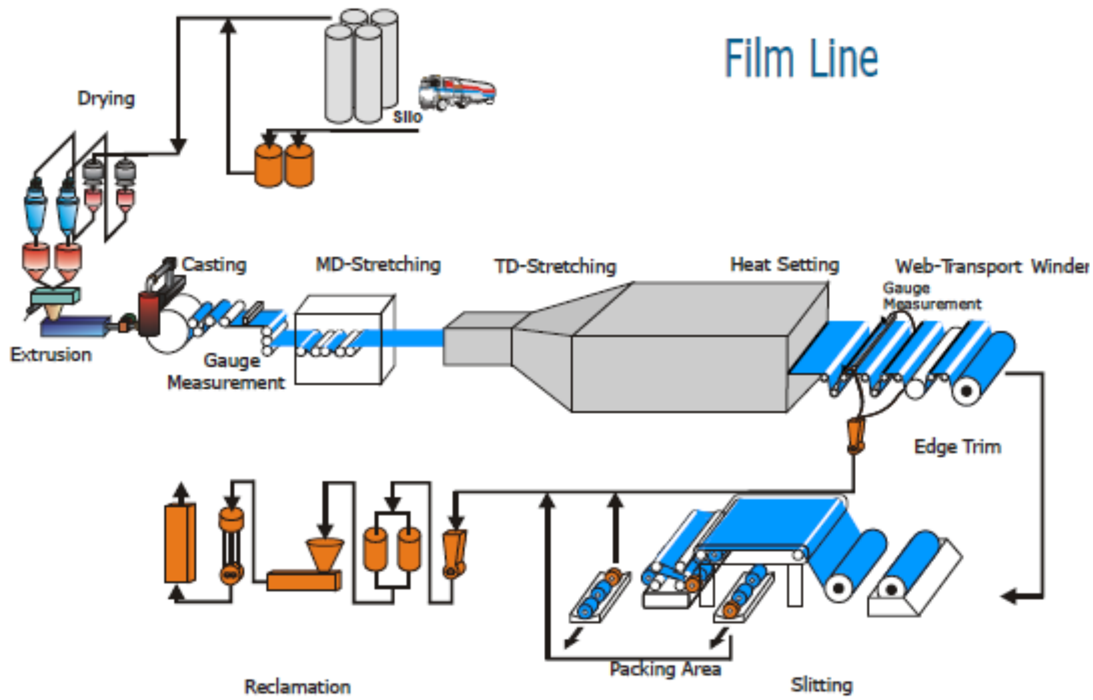

Figure 2.8. PET film line (Mitsubishi, 2011)

Nylon is a condensation polymer formed from a monomer with a functional group of $-NH_2$. The first step in this reaction is the formation of esters from the diacids and diamines (Selke et al., 2004). From these intermediates, the polymerization reaction proceeds. Because the first step is a faster reaction, the monomer is used up quickly (Selke et al., 2004). Condensation polymers generally have non-carbon atoms as part of the main backbone chain. Once polymerization has taken place, pellets are formed which can be used for the production of the nylon film. The pellets pass through a drying system and are fed into a hopper. Once through the hopper, the pellets enter the extruder using friction to melt the polymer. Once the polymer is molten it is cast onto a series of rollers. Once the film is has been cast, it is wound and slit for the particular application.

Polypropylene is an addition polymer formed from a monomer of propylene. Addition polymers are most frequently produced by a free radical polymerization mechanism (Selke et al., 2004). There are three basic steps in the free radical polymerization: initiation, propagation, and termination. Initiation begins the chain growth. Propagation increases the size of the polymer molecule. Termination ends the growth of the molecule. Once polymerization has taken place, pellets are formed which can be used for the production of the polypropylene film. The pellets pass through a drying system and are fed into a hopper. Once through the hopper, the pellets enter the extruder using friction to melt the polymer. Once the polymer is molten it is cast onto a series of rollers. Once the film is has been cast, it is wound and slit for the particular application.

The three layers upon the formation of roll stock are slit to the proper width needed to construct the pouch. Once the correct width is cut, the layers are then laminated together to construct the final structure used to form the retort pouch. The individual layers are laminated together using solvent-based aliphatic adhesive.

Social Aspects, Trends, and New Development of Retort Pouches

Retort pouches are very common in Asia and Europe and high-speed lines have been developed in both geographic regions. There were about 800 million to 1 billion pouches sold in the U.S. and Canada in 2005. In 2005, it was reported that pouch sales in the U.S. were growing at a rate of 13-15% each year (Gazdziak, et. al. 2005). A number of different food products utilize retort pouches including pet food, meat, poultry, seafood, rice and soups. Many companies have recently introduced food products in

retort pouches: Star-Kist, Chicken of the Sea, Bumble Bee, Tyson (whitemeat chicken), Sara Lee (Sweet Sue Kitchens, chicken, ham, turkey and other products in retort pouches) and Masterfoods Inc. (Ready Rice, cooked, shelf-stable rice in pouches) (Demetrakakes, 2004). The advantages of utilizing a pouch versus a can were published by Mykytiuk (2002). Figure 2.9 shows this comparison.

Pouch Vs. Can Advantages

FEATURES	BENEFITS
Reduced cooking time	Improved taste/nutritional value, faster cycle time
No sharp edges	Eliminates cuts and promotes employee safety
Takes up less space	Increased utilization of warehouse/storage space
Package differentiation	Increased sales
Environmentally friendly	Source/energy reduction
Weighs less	Reduced transportation costs
Larger package facing	Better shelf appeal
Rotogravure printing	Improved graphics capabilities
Package durability	No dented cans
Complete product evacuation	Improved yield
Conforms to all FDA guidelines	Market ready

Figure 2.9. Pouch versus can comparison (Mykytiuk, 2002)

There are several advantages of retort pouches. Retort pouches can reduce the processing time of the food product. They can have a shorter cook time because of the high ratio of surface area to volume (Mykytiuk, 2002). The shorter cook time results in better taste, improved nutritional value and less moisture loss (Gazdziak, et. al. 2005). Pouches also take up less space and weigh less meaning increased utilization of warehouse/storage space. The reduced weight and space also saves money because more

unfilled pouches can be delivered at once to the processing facilities as opposed to unfilled rigid metal cans (Mykytiuk, 2002).

New developments in the area of retort pouch technology are in the materials used for the construction of the pouch. Materials such as EVOH are being utilized as barrier layers in the retort pouch as alternative to other barrier films such as metalized films and AlOx and SiOx coated films.

Previous Research on the Transportation of Retort Pouches

Kongcharoenkiat (1980) and Rukspollamuang (1983) both observed how retort pouches behave during a vibration simulation. Both studies observed how retort pouches, which were conditioned at 23°C and 50% RH for 24 hours, performed during sine vibration referencing ASTM D999. The studies utilized the following methodology for determining damage for the retort pouch: Critical Defect – leakers which could be detected by the naked eye, Major Defect – flex cracks, pinholes, and punctures which could be detected by the naked eye, and Minor Defect – flex cracks or pin holes which were visible using a 48X microscope.

Kongcharoenkiat (1980) researched the effect of vibration on retort pouches oriented in horizontal and vertical directions inside corrugated boxes. Retail size retort pouches were filled with Chicken A La King and packed in paperboard cartons placed in corrugated boxes. Stacks of eleven boxes were subjected to vibration for one hour at resonance frequency and at an acceleration of 1 G. The retort pouches had a resonant frequency ranging from 5.8 – 7.9 Hz. The data showed that vertically oriented pouches had more total damage. In both horizontal and vertical orientations, the top box had the

greatest amount of damage (Kongcharoenkiat, 1980). Horizontally oriented pouches were more sensitive to compression from the boxes above. Kongcharoenkiat found that unitization of a stack load reduced damage in both orientations. Unitization reduced the average occurrence of damage per box in horizontally oriented pouches by fifty percent while the average occurrence of damage per box in the vertically oriented pouches was only slightly reduced. Kongcharoenkiat summarized the following possible causes of retort damage: the springiness of the corrugated box, the notched are on side seals, and headspace in upper portion of retort pouches. The size of the pouch in comparison to the size of the carton can result in damage. Vibration can cause the damage due to movement of the pouches if the cartons headspace is too large.

The physical damage and the effect of vibration on the oxygen barrier quality of institutional retort pouches was studied by Rukspollamuang (1983). The retort pouches were made of polyester, aluminum foil, and polypropylene. The outside dimensions of the pouch were 12 x 15 inches, and had a holding capacity of 98 oz. Rukspollamuang packaged water inside institutional retort packages gas flushed with 50 cubic centimeters of nitrogen. The pouches were packed into a regular slotted container (RSC). Stacks of three cases were evaluated. The cases were either non-unitized or unitized. A sine sweep of the cases was completed, and a resonant frequency for both the non-unitized and unitized stacks of products was determined. The resonant frequency of the non-unitized stacks was determined to be 6.6 Hz which increased to 7.9 Hz if the stacks were unitized by stretch wrapping (Rukspollamuang, 1983). Once the resonant frequency was determined, a dwell of 15 minutes at 0.5 G was conducted on the stack of cases. After the

vibration a test, a visual inspection of the pouches was completed to determine if any failures had occurred. In both stacks, the top box contained pouches which had the greatest amount of physical and critical damage. The amount of critical damage in the unitized stacks was 28% lower than in the non-unitized stacks (Rukspollamuang, 1983). An initial headspace analysis was conducted prior to the start of any vibration testing. The headspace was immediately analyzed after the vibration test, and then again every two weeks for thirty days. The results showed that there was an increase of headspace oxygen for each of the samples analyzed, but that the difference was not significant. No further analysis of the oxygen barrier was conducted to determine if there was any interaction with the location of the pouch or location of the box in regards to the oxygen analysis.

The effects of headspace volume inside a retort pouch have been researched to understand the effects of thermal processing time and shelf life storage (Al-Baali and Farid, 2006 and Sepulveda et al., 2003). To date, no published research is available showing the interaction between pouch headspace volume and its effect on hazards encountered in the distribution environment.

Modified Atmosphere Packaging

Air is a gaseous composition primarily made up of nitrogen (N_2) 78.08%, oxygen (O_2) 20.96%, carbon dioxide (CO_2) 0.03%, and variable concentrations of water vapor and traces of inert or noble gases. Many foods spoil rapidly in air due to moisture loss or uptake, reaction with oxygen and the growth of aerobic microorganisms, i.e. bacteria and molds (Coles et al., 2003). Microbial growth can also affect food attributes and qualities

such as texture, flavor, and nutritional content, and can render foods unpleasant and even in some cases unsafe for human consumption. The modification of the gaseous composition inside the package can maintain quality and extend the expected shelf life of food products.

Modified atmosphere packaging (MAP) is defined as the packaging of a perishable product in an atmosphere which has been modified so that its composition is other than that of air (Hintlian and Hotchkiss, 1986). The intended atmosphere for MAP can be achieved in two fundamental ways. These are the mechanical replacement of air with a gas or gas mixture or by generating the atmosphere within the package either passively, as in the case of fruit and vegetables, or actively by using suitable atmosphere modifiers such as oxygen absorbents (Blakistone, 1999). Figure 2.10 provides an example illustration of the gas composition inside packages from both passive and active modification systems.

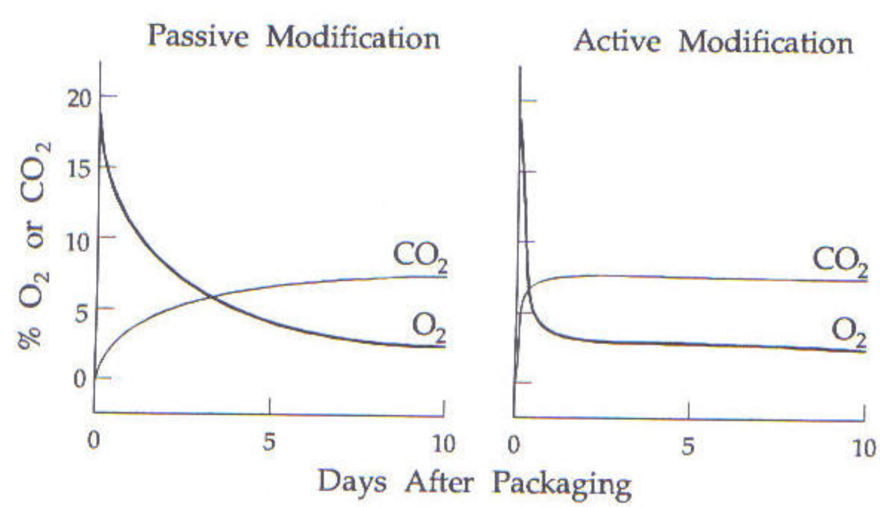

Figure 2.10. Passive and active modification systems employed in MAP (Cooksey, 2013)

Controlled atmosphere packaging (CAP) is the intentional alteration of the natural gaseous environment and maintenance of that atmosphere at a specified condition throughout the distribution cycle, regardless of temperature or other environmental variations (Brody, 1989). CAP is generally recognized as storage or bulk container systems such as pallet bags and paperboard containers (Arvanitoyannis, 2012).

The gaseous composition of fresh MAP foods is constantly changing due to respiration of the product, chemical reactions and microbial activity. Gas exchange between the package headspace and the external environment may also occur because of permeation across the package material. Packaging foods in a modified atmosphere can offer extended shelf life and improved product presentation in a convenient container, making the product more attractive to the retail customer (Blakistone, 1999). However, MAP cannot improve the quality of a poor quality food product. It is therefore essential that the food be of the highest quality prior to packing in order to optimize the benefits of modifying the package atmosphere (Blakistone, 1999). Table 2.5 outlines the advantages and disadvantages of employing the use of MAP.

Table 2.5. Advantages and disadvantages of MAP

Advantages of MAP	**Disadvantages of MAP**
Increased shelf life allows for wider distribution of food	Capital cost of gas packaging machinery and materials (gas and packaging)
Improved presentation – clear view of the product	Temperature control is required
Requires less storage space/time and restocking of retail shelves	Increased package volume increases transport cost and retail display space

In order to maintain the integrity of the MAP generated inside the package it is imperative to have a successful heat seal. Aspects affecting how the seal is formed include film properties, pouch geometry, and production line speed (Lee et al., 2008). The four components from a manufacturing and equipment standpoint factoring into producing a good heat seal are the temperature of the seal bars, dwell time, pressure, and alignment of the seal bars. While manufacturing operations try and push the limitations for packing out, it is essential not to overlook these factors. In doing so, the package will have ineffective seals resulting in the loss of the atmosphere generated.

The three main gases used in modified atmosphere packaging are O_2, CO_2 and N_2. The choice of gas is very dependent upon the food product being packaged. Gases are used individually or in combination. An inert gas, such as nitrogen, has low solubility in both water and lipids, and is useful in applications such as potato chip bags. Experimental use of carbon monoxide (CO) has also been reported.

The research for this project will involve gas flushing with nitrogen, which is a type of mechanical replacement. The gas flushing process uses a continuous stream of gas injected into the package to replace the air. This process dilutes the air in the headspace surrounding the food product. The typical residual oxygen levels in a gas flushed packaged are 2-5% (Blakistone, 1999). Gas flushing with nitrogen is a common industry practice when the intent of the MAP is to extend the shelf life.

While the intention of MAP is to extend the shelf life of a product, few studies have shown how or if the atmosphere inside the package changes as a result of the distribution environment. Margareetta et al. (2009) examined oxygen and carbon dioxide

levels of cold cut meat products packaged in MAP at retail levels. The results of this study showed the atmospheres had been altered from the time the package was sealed to when the packages arrived for retail. This study revealed residual oxygen can be higher than acceptable ranges due to various reasons: minor leaks in structure, leaks formed during sealing, or poor feed of protective gas. Oxygen content can also increase if the packages are handled too roughly during distribution or shelving, and at the point of sale (Margareetta et al., 2009).

Heat Sealing

Sealing is the transition from a flexible web material to the formation of a pouch or bag. Pouch making equipment uses seals to convert a two-dimensional web to a three-dimensional pouch thus being able to contain food or other types of products. The construction and execution of a seal, although in concept is straightforward, is very complex and encompasses several different techniques in order to create the proper seal for the application.

Heat sealing is the process by which two structures containing at least one thermoplastic layer are sealed by the action of heat and pressure (Selke et al., 2004. Figure 2.11 illustrates the two major types of heat seals for packaging applications: lap seal and fin seal. In heat sealing, the thermoplastic layer is heated and pressure is applied to create a bond between the two webs of material. Flexible materials used in heat sealing are grouped into two categories, according to the type of material employed in their construction: supported and unsupported structures (Selke et al., 2004). Supported structures consist of laminations containing one or more non-thermoplastic layers (such

as paper or foil), boded to thermoplastic layers, at least one which is used for sealing (Selke et al., 2004). Unsupported structures consist of one or more thermoplastic layers and do not contain a non-thermoplastic layer.

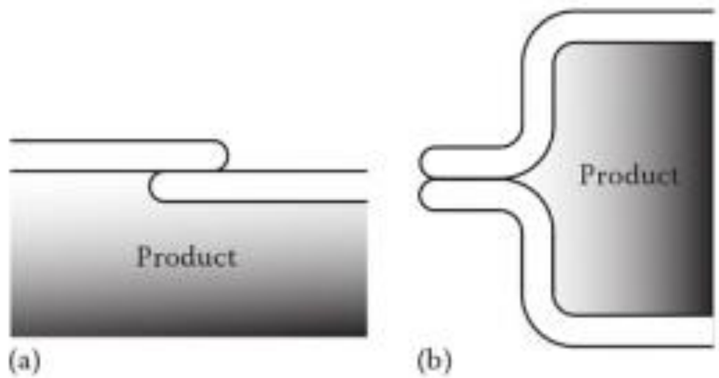

Figure 2.11. Two major types of heat seals: (a) lap seal and (b) fin seal (Robertson, 2006)

Sealing a flexible structure to make a closed package requires the heat seal layer to be located in the interface – typically in contact with another heat seal layer. When heat and pressure are applied to the external surface to make the seal, the heat is transmitted by conduction or radiation to the packaging material, and then is transmitted through the material by radiation as the heat input (Selke et al., 2004). Another key component with heat sealing is referred to as dwell time. This is the time allowed for the two thermoplastic layers to flow and create a bond. This bond is a result of the molecular entanglement within the polymer chains from the two heat sealing layers producing a homogenous layer once after cooling. A good seal is created when enough molecular entanglement has taken place (Selke et al., 2004). Each of the three components of heat sealing, pressure, heat, and dwell time, can be adjusted to create the seal suitable for the particular packaging application. These along with resin factors (density, molecular

weight, and additives) and film factors (thickness, style, or form) all interact with each other in forming the perfect seal for a given application (Robertson, 2006).

Food and Packaging Analysis Techniques

Leak, Burst and Pressure Decay Testing

A simple form of leak detection is through the use of vacuum chamber. The chamber is filled with water and the test package is placed inside the chamber held under the water level. A vacuum is created inside the chamber at either a set rate or to a set pressure and the package is observed for visual leaking. If leaking is present, bubbles will form and exit the package. While this method doesn't provide any quantitative data, it provides a quick and effective method to ensure packages are sealed properly for intended use.

Burst and creep testing entails pressurizing the inside of the package and measuring the pressure required to either cause seals to separate or packaging materials to rupture. Some packages use porous materials, allowing the contents to be gas sterilized through the porous package walls after the package has been sealed; burst testing of these packages can require higher inflow capability in order to achieve sufficient pressure to challenge the package.

Pressure decay testing identifies failure of a package's seal or materials by measuring the package's ability to maintain a constant internal pressure—typically 50% of burst pressure—over a period of time.

Headspace Composition and Analysis

Oxygen promotes many food spoilage reactions. Certain foods can be damaged by exposure to oxygen concentrations of 1-2% (Coles et al., 2003). The level of residual oxygen in the package headspace is therefore of concern to food processors and forms part of the quality procedures for the manufacture and pack of oxygen-sensitive products. An example of the type of equipment which measures the concentration of headspace oxygen is the MOCON PAC CHECK® headspace analyzer. This instrument determines residual oxygen by probing and sampling headspace gases in MAP packs and is suitable for production and laboratory use, with a rapid response time.

Oxygen Transmission Rate

The Oxygen transmission rate (OTR) of packaging materials is often tested with MOCON Ox-Tran 2/21 (Mocon, Minneapolis, MN, USA) oxygen permeation instruments. It precisely controls the temperature and relative humidity and can measure and detect oxygen, carbon dioxide, and nitrogen in minute amounts (parts-per-billion sensitivity). Film samples are removed from packaging material and are securely clamped into a diffusion cell. This is a destructive test and the package cannot be reused. All residual oxygen is then removed from the chamber. When zero percent oxygen is established, pure oxygen is introduced into one side of the chamber opposite to the sensor. The sensor then records the diffusion of oxygen through the material. This process cannot be repeated for the same sample and can take up to 48 hours per sample.

Real time oxygen ingress that is non-invasive and passive can be measured with an OxySense Gen III 300 system (OxySense, Inc., Dallas, TX). This system consists of

two parts, an oxygen sensor, and a master box that evaluates and interprets the findings. The oxygen within an enclosed system is measured without destroying or altering the internal environment, which is an added benefit as the same samples can be repeated (Saini, 2008).

Oxygen concentration measurements are possible based on fluorescence quenching. The OxyDot® (Dot) is comprised of an oxygen sensing dye that is immobilized in polymer that can withstand high temperature and pressure processes yet is permeable to gas. The Dot absorbers blue light emitting diode (LED) light and fluoresces light in the red region. Figure 2.12 represents the fluorescence decay over time. When oxygen is absent the Dot will emit an intense red light for 5μs whereas when oxygen is available the light intensity and emission is decreased to ~1μs. The decrease in intensity and emission can be calculated to accurately provide the amount of oxygen available within 5% accuracy of the reading. The Dot does not consume oxygen in the process and the test can be repeated quickly (5 seconds) and indefinitely (Saini, 2008).

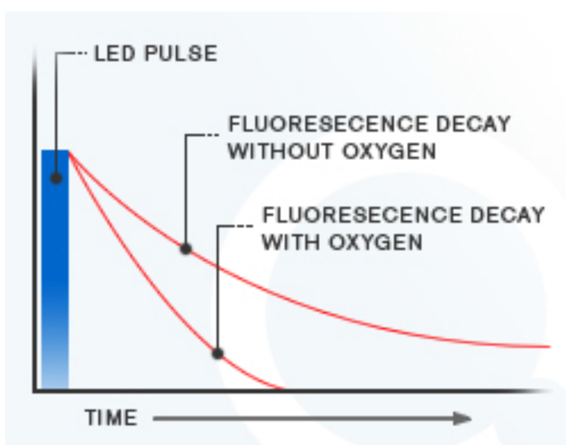

Figure 2.12. Graphical representation of fluorescence decay with and without oxygen, within 5% accuracy of the reading (Saini, 2008)

Oxidation

Food oxidation is an irreversible process causing food to spoil and become rancid. The two types of rancidity are hydrolytic and oxidative. Hydrolytic rancidity occurs with high moisture and heat. Oxidative rancidity occurs when oxygen interacts with unsaturated fatty acids. This is also known as lipid oxidation (Damodaran, Parkin, & Fennema, 2008).

A number of chemical components of food react with oxygen affecting the color, flavor, nutritional status and occasionally the physical characteristics of foods (DeMan, 1980). In some cases, the effects are deleterious and limit shelf life; in others they are essential to achieve the desired product characteristics. Foods containing a high percentage of fats, particularly unsaturated fats, are susceptible to oxidative rancidity and changes in flavor. Saturated fatty acids oxide slowly compared with unsaturated fatty acids. Three different chemical routes can initiate the oxidations of fatty acids: the formation of free radicals in the presence of metal ion catalysts such as iron, or heat, or light – termed the classical free radical route; photo-oxidation in which photo-sensitizers such as myoglobin affect the energetic state of oxygen; or and enzymic route catalyzed by lipoxygenase (Coles et al., 2003). Once oxygen has been introduced into the unsaturated fatty acids to form hydroperoxides by any of these routes, the subsequent breakdown of these colorless, odorless intermediates proceeds along similar routes regardless of how oxidation is initiated. It is the breakdown products of the hydroperoxides – the aldehydes, alcohols, and ketones that are responsible for the

characteristic stale, rancid, and brown colors associated with lipid oxidation (Coles et al., 2003).

The thiobarbituric acid (TBA) test is one of the most extensively used methods to detect oxidative deterioration of fat-containing foods (Kishida et al., 1993). During lipid oxidation, malonaldehyde (MA), a minor component of fatty acids with 3 or more double bonds, is formed as a result of the degradation of polyunsaturated fatty acids. It is usually used as an indicator of the lipid oxidation process, both for the early appearance as oxidation occurs and for the sensitivity of the analytical method (Cesa, 2004). In this assay, the MA is reacted with thiobarbituric acid (TBA) to form a pink MA-TBA complex that is measured spectrophotometrically at its absorption maximum at 530– 535 nm (Figure 2.13) (Shahidi and Zhong, 2005). The extent of oxidation is reported as the TBA value and is expressed as milligrams of MA equivalents per kilogram sample or as micromoles of MA equivalents per gram of sample. It must, however, be noted that alkenals and alkadienals also react with the TBA reagent and produce a pink color. Thus, the term thiobarbituric acid reactive substances (TBARS) is now used instead of MA.

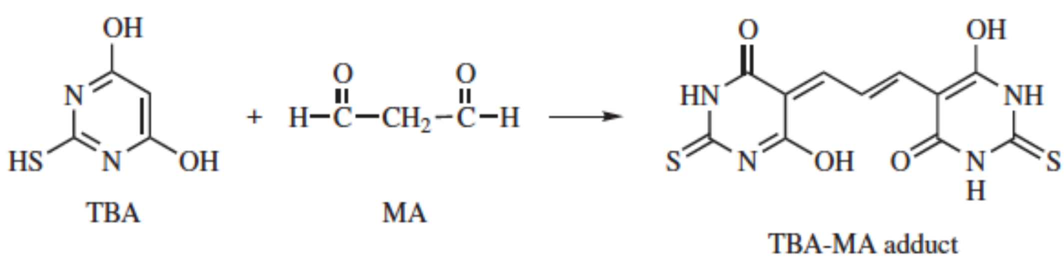

Figure 2.13. Reaction of 2-thiobarbituric acid (TBA) and malonaldehyde (MA) (Shahidi and Zhong, 2005).

The TBA test is used frequently to assess the oxidative state of a variety of food systems, despite its limitations, such as lack of specificity and sensitivity (de las Hera et al., 2003). As already noted, many other substances may react with the TBA reagent and contribute to absorption, causing an overestimation of the intensity of color complex (de las Hera et al., 2003). Despite it limitations, the TBA test is commonly used to evaluate and compare lipid oxidation in foods packaged in different materials.

Texture Measurement

Texture is defined as all the rheological and structural (geometric and surface) attributes of the product perceptible by means of mechanical, tactile and, where appropriate, visual and auditory receptors (Lawless and Heyman 1998). Texture is perceived by the sense of touch and comprises two components: somesthesis, a tactile, surface response from the skin; and kinesthesis, which is a deep response from muscles and tendons (Kilcast and Subramaniam, 2000). While this provides a broad definition of texture, it doesn’t include other factors, such as sound and appearance, and in combination with texture affect the flavor of food products. Dubose et al (1980) found that by increasing color intensity of beverages, there was an increase in the perceived flavor. Likewise, Vickers (1991) determined that the sound produced from chips and other crunchy foods had a large influence on observed flavor.

Quantifying the differences of texture requires the use of instrumentation to analyze and compare different food products. The instrumental methods of texture are based on mechanical tests designed to compress the food and determine the stress or force required to cause a failure. In general texture measuring devices consist of four

basic elements, a probe, a driving mechanism, load (force) cell, and digital output system (Kramer and Szczesniak 1973). Instruments such as the TA.XT*Plus* Texture Analyzer, is capable of measuring virtually any physical product characteristic or materials testing metric.

Water Activity

Water activity (a_w), temperature, and pH are the most important factors that controls rates of deteriorative changes and growth of microorganisms in foods (Eskin and Robinson, 2001). The usefulness of a_w results from its provision of a measure of the osmotic stress experienced by microorganism and also from its ability to take into account water-solute interactions to some extent (Kilcast and Subramanian, 2000). Availability of water for yeast, mold, and bacteria growth are affected by these factors.

Water activity is the ratio of the vapor pressure in a solution or a food material, p, and that of pure water at the same temperature, p_0 (Eskin and Robinson, 2001). This relationship is illustrated in equation 2.1. Therefore, the equilibrium or steady state aw is related to equilibrium relative humidity (ERH) of the surrounding atmosphere by equation 2.2, and water activity can be considered to be a temperature-dependent property of water which is used to characterize the equilibrium or steady state of water within a food material (Eskin and Robinson, 2001).

Equation 2.1 $$a_w = \frac{p}{p_0}$$

Equation 2.2 $$ERH = a_w \times 100\%$$

Water activity is a measure of the energy status of the water in a system. Understanding and measuring the a_w can aid in identifying the relationship of water content to microbial growth, chemical reactivity, and stability as seen in Figure 2.14 Instrument technologies, like AquaLab, have made it easy to measure the water activity of a sample. This type of instrument has the ability for accurate and reliable measurements and can be performed quickly in a laboratory setting.

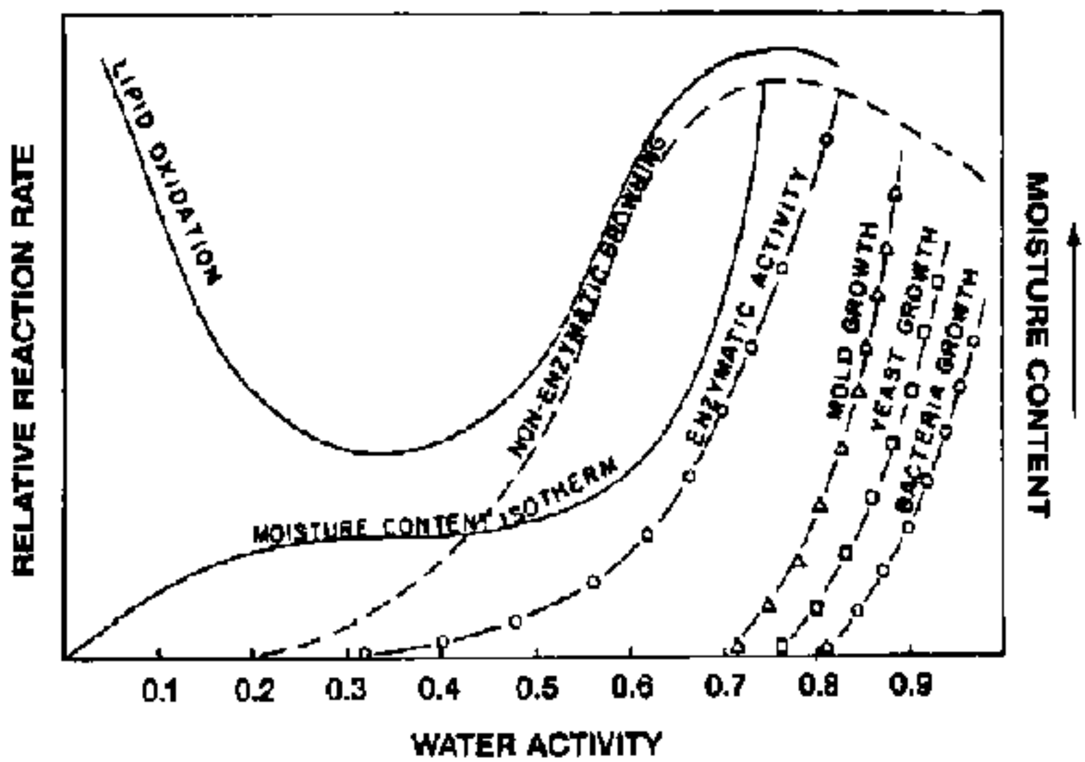

Figure 2.14. Relationship of food deterioration rate as a function of water activity (Barbosa-Canovas, et al., 2003)

Moisture Content

Water is either mixed in or driven off by the process of drying when food items are processed. Many common foods like potato chips absorb water particles when

exposed to increased humidity, resulting in deterioration of quality leaving them unpalatable.

Moisture content of unprocessed foods are generally expressed on a wet basis, but many food products after drying have their moisture content expressed on a dry basis only (Bhuyan, 2007). One of the most commonly used techniques for measuring moisture content of a food product is the dry and weigh technique. A technique providing quicker and more accurate results for moisture content determination utilizes infrared hearting and precision weighing. This method, employed for this research, measures moisture content for a range of products.

Shelf Life Testing

The term shelf life is used throughout the food and other industries, although the concept is largely not understood. Shelf life can be defined as the period of time from the production and packaging of a product to the point at which the product first becomes unacceptable under defined environmental conditions (Lee et al., 2008).

Despite the complexity of food systems, the systematic study of their deteriorative mechanisms can lead to sound methods of determining shelf life (Fennema, 1996). The prediction of shelf life can be performed and carried out in two general ways. The most common way of determining the shelf life involves exposing the food product to a single abuse condition for an extended duration, evaluating quality, generally by sensory methods, and then extrapolating the result to normal storage conditions (Fennema, 1996). The alternative method is to utilize a more elaborate design based on principles of chemical kinetics and to determine the actual temperature dependency of various quality

attributes (Fennema, 1996). The initial method is commonly referred to as accelerated shelf life testing (ASLT) and is the method employed in this research.

ASLT is a method for testing food products under accelerated conditions to decrease the testing window or allowing more evaluations to be conducted in a timely manner. This method is useful for products whose deterioration kinetics is known functions of certain accelerating factors, typically being temperature. This is due in part to the Arrhenius equation (Equation 2.3) which uses an accurate formula displaying the temperature dependence of reaction rates. Essentially for every 10-degree increase in temperature, the reaction rate is doubled. By understanding and employing this concept, it is possible to decrease the testing window as well as extrapolate the data for normal storage conditions.

Equation 2.3 $$k = k_o \exp\left(-\frac{E_a}{RT}\right)$$

Although the use of ASLT is widely used, there are potential problems and errors that can arise during the study. It should be noted during the analysis that ASLT is only an estimate of the actual shelf life of a packaged product, except in the case of very simple chemical reactions (Robertson, 2006). One way to ensure the ASLT is correct is by conducting actual shelf life studies using normal handling and storage conditions. Once this is established for a particular product, then ASLT can be used for that product when evaluating different processes and packaging variables (Robertson, 2006).

One area this research aims to evaluate is the accuracy of ASLT for packaged products having been transported or distributed. In review of this manuscript, it was not clear whether or not this is standard practice for industries to evaluate. ASLT typically

involves the use of temperature, and doesn't subject the package or the food product to any other distribution hazards, such as shock, vibration, or compression, which packages will encounter during the movement of goods. Therefore, part of this research will be evaluating whether the deteriorative rates of certain aspects of the food will be increased as a result of the mechanical and manual abuse the packages will be put through during the distribution to the consumer.

Food Transportation

Logistical Movement of Goods

Throughout history, physical distribution of goods of some kind has been used as a way to move goods from one location to another. Today, logistics involves more than just the movement of goods; it is a key part in the physical distribution of packages and products across the globe.

After World War II, business strategists began to recognize that similar principles, which were applied during the war, could be used in the private industry sector. Peter Drucker, a management theorist, began publishing and writing on this topic about a decade after the war. Drucker described business logistics as the last great frontier for major cost reduction in American manufacturing. An increasing number of managers recognized the advantages of organizing their companies with a physical distribution or logistics department (Ackerman, 1990). These companies saw the value that could be added to the company by cutting cost of moving goods from location to location. Companies began to realize that the large amounts of money that were being lost during

the movement of goods could be significantly decreased if they had skilled logistics professionals employed.

Physical movement or logistics was actually a blending of multiple activities previously separated by most companies. The term logistics management can be defined as the “part of Supply Chain Management that plans, implements, and controls the efficient, effective forward and reverse flow and storage of goods, services and related information between the point of origin and the point of consumption in order to meet customers’ requirements” (CSCMP, 2007). Modern logistics is made possible through the combination of numerous functions working together towards a common goal, delivering a product to the end customer.

Overview of Food Transport

Transportation is an essential link in the modern food supply chain. It exists as a complex matrix, where not only are finished products and fresh good transported, but there is also considerable movement of raw materials and ingredients (Heap et al., 1998). Logistical networks deliver food via complex distribution channels that range from a few hundred miles to across the globe. The movement of food products is sometimes considered to be no more than storage on wheels. However overlooks the fact that these materials are subjected to unique physical and mechanical stresses, and also that the elapsed time and the distances traveled raise the potential for rapid fluctuations in temperature and humidity conditions, which present considerable challenges to the biological status of foods (Heap et al., 1998).

Distribution Performance Testing

Common transportation channels that a packaged product would pass through are over-the-road truck transportation, rail transportation, and aircraft transportation. Throughout the various distribution channels the packaged products are subjected to three major categories of dynamic hazards: shock, vibration, and compression (Brandenburg and Lee, 2001). The protection and containment provided through packaging can be evaluated and compared through laboratory simulations and field studies. Performance testing is used to assess containers in situations that simulate distribution hazards and reproduce damage (Twede and Harte, 2003).

Performance testing differs from engineering testing in that performance testing are tests or simulations performed sequentially in order to replicate a particular shipping environment. Engineering testing are individual tests designed to evaluate a single hazard such as performing a compression test. Performance testing provides feedback directly correlating with data collected from field shipments. To allow for greater repeatability of these performance tests, standards were developed. Two of the most commonly used standard writing organizations are the American Society of Testing and Materials (ASTM) and the International Safe Transit Association (ISTA). Both develop and publish standards designed to simulate a particular shipping mode, such as less-than-truckload (LTL) or small parcel environments. These standards have been used for decades to aid in optimizing package design and protection.

Research Objectives

1. Effect of Headspace Volume on Pouch Performance During Laboratory Simulated Hazards
    a. Evaluate the interactions between headspace volume, storage temperature, and product viscosity on the seal integrity of a retort pouch during engineering tests. The engineering tests used for evaluation are shock, compression, and vibration.
    b. Critical defects will be observed as failure modes for this research objective. The critical defects will result in visible leaking or loss of product. Classification of pouch failure (delamination, seal fracture, channel, etc.) will be recorded.
2. Analysis of Oxygen Levels in Gas Flushed Retort Pouches During Distribution and Storage
    a. Determine if oxygen percentage inside a pouch changes as a result of laboratory simulated distribution hazards.
    b. Evaluate if headspace volume can influence the oxygen percentage changes in packaged products.
    c. Packaged products will be evaluated based on performance test protocols.
        - Performance test protocol will follow current ISTA methodology.
    d. Use non-destructive method to track oxygen changes
        - OxySense® Technology

3. Experimental Evaluation of the Effects of Transportation on Accelerated Shelf Life Testing of Packages Containing Potato Chips
    a. Determine if MAP is effected as a result of laboratory simulated distribution hazards
        - Packaged products will be evaluated based on performance test protocols.
        - Performance test protocol will follow current ISTA methodology.
    b. Evaluate and compare to ASLT to determine if simulated distribution has effect on product

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CHAPTER THREE

EFFECT OF HEADSPACE VOLUME OF RETORT POUCHES ON SIMULATED TRANSPORT HAZARDS

ABSTRACT

Institutional retort pouches containing either water or 5% starch solution were filled with varying amounts of headspace volume to determine if adding gaseous volume to the package can help it survive laboratory simulated engineering tests. Fixed displacement vibration testing and compression testing of the pouches yielded no differences in the amount of headspace volume. Significant differences were noted as a result of the shock testing (free fall drop method) when comparing the water filled pouches, but no headspace volume effect was observed for the 5% starch solution pouches. The results from this study showed increasing the headspace volume of a retort pouch does provide increased protection to transport hazards (shock) for a low viscosity food simulant as compared with a highly viscous product packaged similarly.

INTRODUCTION

Institutional retort pouches are becoming more widely used as a replacement for the #10 can due to its ease of use and disposal [1]. Retort pouches help improve the safety for both consumers and employees. There are no sharp edges as with cans which eliminate cuts for both employees in the packaging plants and for the consumers opening the packages at home [2]. Due to retort pouches weighing significantly less than metal cans, they help lower transportation cost of moving the products throughout the supply chain. Pouches, empty and full, take up less storage space than comparable cans, jars, and trays [3]. Additionally, clear retort pouches, which were employed for this study, are becoming more widely used in applications where the ability to microwave, visibility of the product, and metal detection capabilities are of importance [4]. While all of these features provide advantages to the product manufacturer and consumer, retort pouches do have disadvantages to the metal can such as slower filling speeds and lack of physical durability.

The effects of headspace volume and gas composition inside a retort pouch have been researched to understand the effects on thermal processing time and shelf life storage [5]. Previous research involving the physical durability of retort pouches has shown that pouch orientation and position has an effect on the performance and survival rate during vibration [6] [7]. These studies were conducted utilizing aluminum foil based retort pouches packaged in individual paperboard cartons.

Over-the-road truck transportation, rail transportation, and aircraft transportation are common channels packages pass through. Through these distribution channels

packages are subjected to three major categories of dynamic hazards: shock, vibration, and compression [8]. Another hazard having an adverse effect on package performance are the environmental conditions at which it is transported and stored. A packages ability to withstand these hazards is vital to ensuring the products arrive safely and in usable condition.

The objective of this research was to understand how headspace volume affects the performance of individual retort pouches during laboratory simulated hazards. Product viscosity and storage temperatures were varied and evaluated to determine their effects on the pouches ability to resist critical failures resulting in loss of product. The performance of the pouches through these simulations can determine how they will survive through physical transportation.

MATERIALS AND METHODS

The institutional pouch chosen for this experiment was a 1.5 kg four sided seal retort pouch constructed of aluminum oxide (AlOx) coated polyethylene terephthalate (PET)/biaxially oriented nylon (BON)/cast polypropylene (CPP) (Cryovac® Sealed Air, Duncan, SC). The retort pouches were filled with two food simulants: water and 5% starch solution. The water represented products having a low viscosity, while the 5% starch solution represented a more viscous product.

The headspace volume varied from less than 10 cubic centimeters (cc) to 400cc in 100cc interval and was done for both the water and 5% starch solution pouches. In order to calculate headspace volume, the sealed pouch was submerged in water with a graduated cylinder. The pouch was opened and the pouch was slowly compressed into the

opening of the graduated cylinder. The displaced volume inside the graduated cylinder was recorded. Figure 3.1 provides an illustration on how the headspace volume was captured and recorded [9]. Five samples from each simulant and headspace volume were randomly selected and the average overall gas content was determined for that variable.

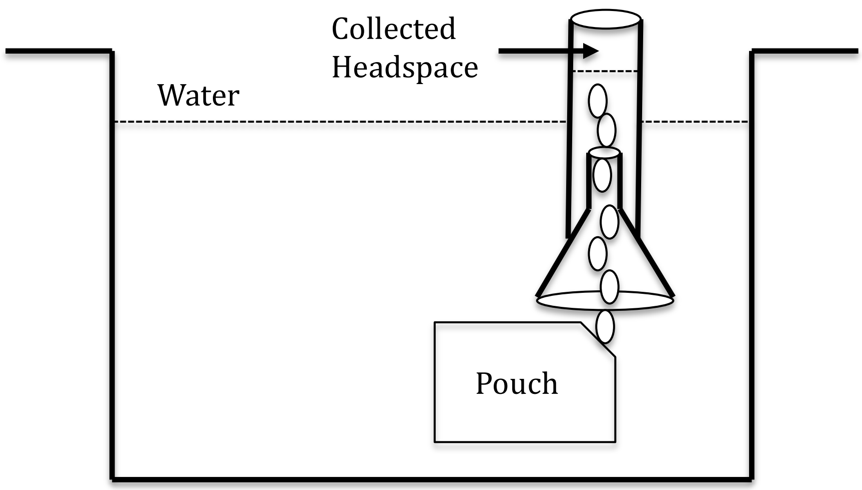

Figure 3.1. Illustration for headspace volume measurement

Storage conditions of the individual pouches also varied. The pouches were stored at 23°C and 50% RH (standard conditions) or at 5°C and 85% RH (refrigerated conditions) per ASTM D4332 [10]. The pouches were held under these conditions for 24 hours prior to testing.

Prior to filling the retort pouches a seal analysis was performed on the pouch to determine the optimal seal temperature for the pouches. The seals were evaluated using a SATEC Universal Tester and following ASTM F88 [10]. The heat seal temperature selected was 350°F with a dwell time of 2.5 seconds. Figure 3.2 illustrates the seal curves of both the pre- and post-retort pouches.

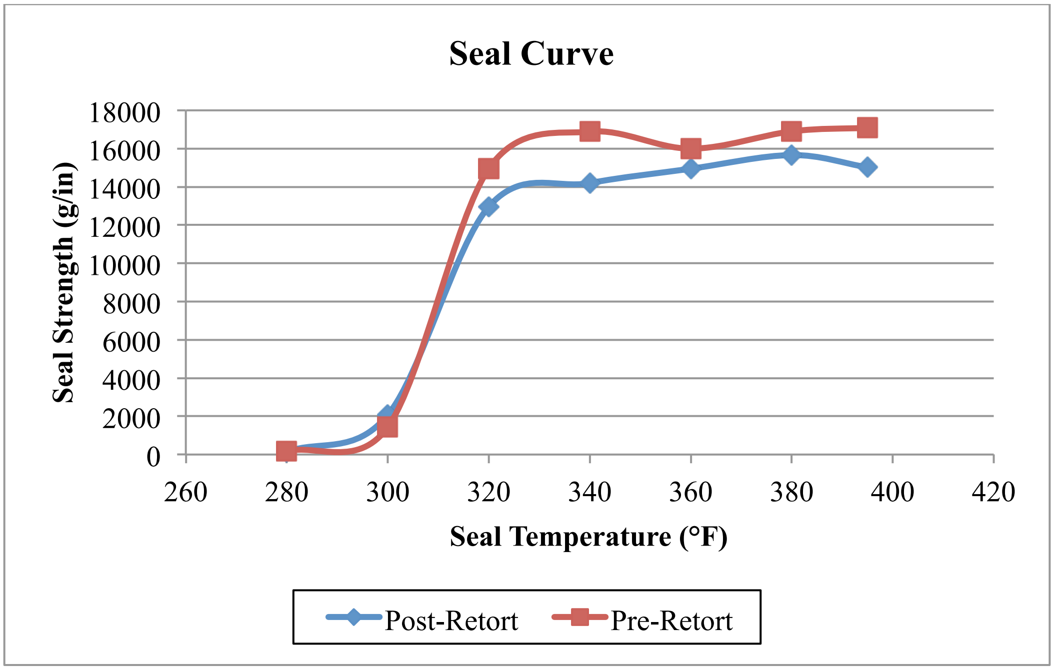

Figure 3.2. Heat seal curve for retort pouch

The retort pouches were filled using a Furakawa/Old Rivers FF-300NU pouch-sealing machine. The heat seal temperature was 350°F with a dwell time of 2.5 seconds. The average filled pouch weight of the water pouches was 1.38 ± 0.10 kg. The average filled pouch weight of the 5% starch solution pouches was 1.36 ± 0.11 kg.

A pilot scale rotary retort was employed for this experiment. The filled pouches were processed for 30 minutes at 250°F (after come-up) and 30 psi using a Surdry Model APR-95 Rotary Pilot Retort (Stock America, Cary, North Carolina). After being processed the viscosity of the two simulants was measured by using a Brookfield Viscometer. The viscosity of the water was 1 cps and the viscosity of the 5% starch solution was 39,000 cps.

Upon conditioning, single pouches were subjected to individual laboratory simulated engineering tests, which included vibration, compression, and shock. Table 3.1

displays the number of samples used for each of the engineering tests. Only critical failures were reported during this research. A critical failure was determined to be a pouch failure that resulted in a visible loss of product.

Table 3.1. Test Protocol Setup and Sample Size

Headspace Volume (cc)	Vibration (Sample Size)		Compression (Sample Size)		Shock (Sample Size)	
	Standard	Refrigerated	Standard	Refrigerated	Standard	Refrigerated
< 10	5	5	20	20	20	20
100 ± 5	5	5	20	20	20	20
200 ± 5	5	5	20	20	20	20
300 ± 5	5	5	20	20	20	20
400 ± 5	5	5	20	20	20	20

Vibration:

Sinusoidal vibration was used to evaluate pouch performance during vibration. Individual pouches were oriented such that the largest surface area was in contact with the vibration table. A Lansmont Vibration Tester Model 1500 was used to perform all vibration tests. ASTM D999 Method A1 (Repetitive Shock Test) was used to perform this experiment [10]. In order to keep refrigerated conditions for those pouches during the vibration test, large insulated coolers were attached directly to vibration table and instrumented with TH10 (Extech® Instruments, Nashua, New Hampshire) temperature data loggers to ensure conditions were maintained.

The test parameters used to drive the vibration table to create enough energy to cause the pouches to oscillate were set to 4.3 Hz and 0.96 G. Test parameters defined by using a 1/16 in. shim that would intermittently pass underneath the pouches. The pouches were vibrated for 60 minutes.

Compression:

Compression testing of the pouches was performed referencing ASTM D642 [10]. An Interlaken Compression Tester (Interlaken Technology, Chaska, MN) was used to compress the individual pouches. Pouches were compressed using a fixed upper platen at a rate of 0.5 in/min. The pouches were compressed to failure recording peak force in pounds (lbs.) and deflection in inches (in.). Twenty pouches from each variable were compressed and the average and standard deviation were calculated for both the maximum force required for critical failure and the corresponding deflection at critical failure.

Shock:

A free fall drop method was used for shock evaluation of the product. Individual pouches were impacted on the face of the pouch with the largest surface area. A Lansmont PDT 56 Drop Tester was used to perform all drop tests. The drop test procedure utilized to perform this experiment was ASTM D5276 [10]. The progressive drop test protocol within the ASTM D5276 standard to determine the critical drop height for each variable being analyzed. The initial drop height for each variable was 48 inches, and the drop height was increased in intervals of 4 inches until critical failure to pouch or a drop height of 72 inches was recorded.

Statistical Analysis:

Data were analyzed as one-way ANOVAs using the general linear model procedure of SAS (version 9.1; SAS Institute, Cary, NC). When significant differences (P

≤ 0.05) occurred among the treatments, the least significant difference test at $P = 0.05$ was used to separate the means.

RESULTS AND DISCUSSION

Vibration:

At the conclusion of each vibration test cycle, the pouches were visually inspected for critical failures (Table 3.2). It was determined that for all of the samples evaluated, no critical failures had been recorded. Critical failures were not observed as a result of the vibration due to the lack of force generated on the pouch seals. Although the product simulants were oscillating on the vibration table, there was not enough force exerted onto the pouch seals to create a critical failure. Because no pouch failures occurred, no statistical comparisons could be made on how headspace volume and product viscosity affect the pouches performance during vibration. Based on these results, it was determined vibration alone was not a critical distribution hazard for individual pouches.

Table 3.2. Vibration Phase Results

Headspace Volume (cc)	Standard Conditions		Refrigerated Conditions	
	Test Samples	Number of Failures	Test Samples	Number of Failures
0	5	0	5	0
100 ± 5	5	0	5	0
200 ± 5	5	0	5	0
300 ± 5	5	0	5	0
400 ± 5	5	0	5	0

Compression:

The compression results displayed in Figures 3.3 and 3.4 illustrate the average maximum force required to create a critical failure. Statistical analysis was performed

independently on the data sets comparing storage temperature and force required for pouch failure on both the water and 5% starch solution using ANOVA. It was concluded that for both the water and the 5% starch solution data sets there was a difference between the mean maximum force required for pouch failure at $P<0.05$. One general trend observed in both of these figures was the refrigerated pouches had a lower compressive force when compared to the standard pouches of the same headspace volume. This was due to the CPP having a glass transition temperature (Tg) near the refrigeration storage conditions resulting in the seals becoming more brittle at lower temperatures causing the seals to fail at a lower force [11].

The deflection results displayed in Figures 3.5 and 3.6 illustrate the maximum deflection required to create a critical failure at the corresponding maximum force. Statistical analysis was performed independently on the data sets for both the water and 5% starch solution using ANOVA. For both the water and the 5% starch solution data sets there was not a statistical difference between the means at $P=0.05$.

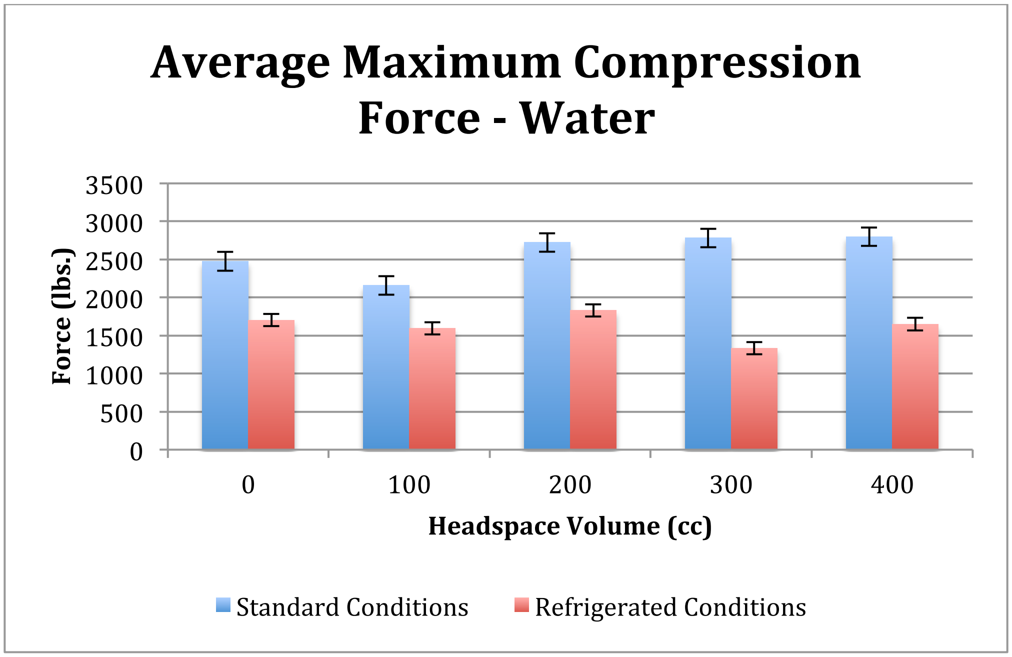

Figure 3.3. Average maximum compression force for pouches filled with water

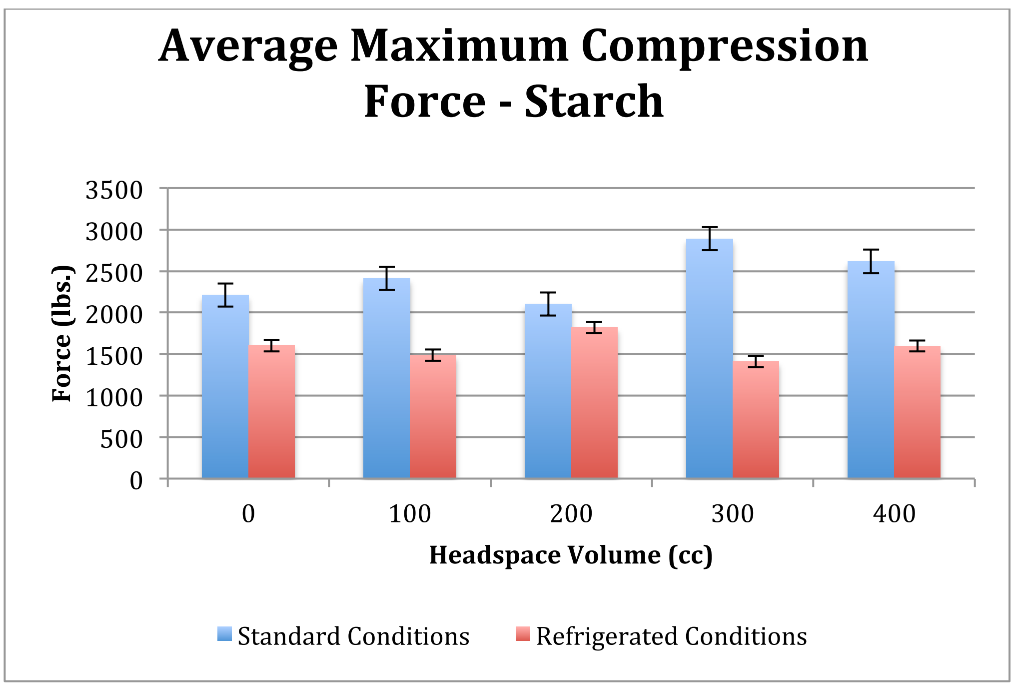

Figure 3.4. Average maximum compression force for pouches filled with 5% starch solution

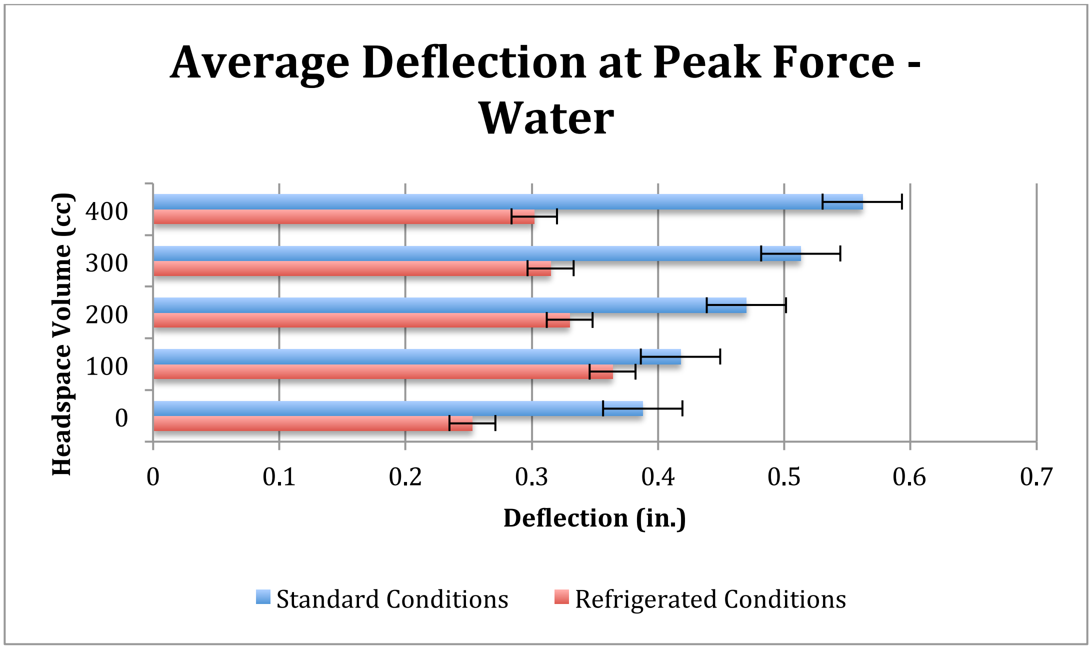

Figure 3.5. Average peak deflection at critical failure for pouches filled with water

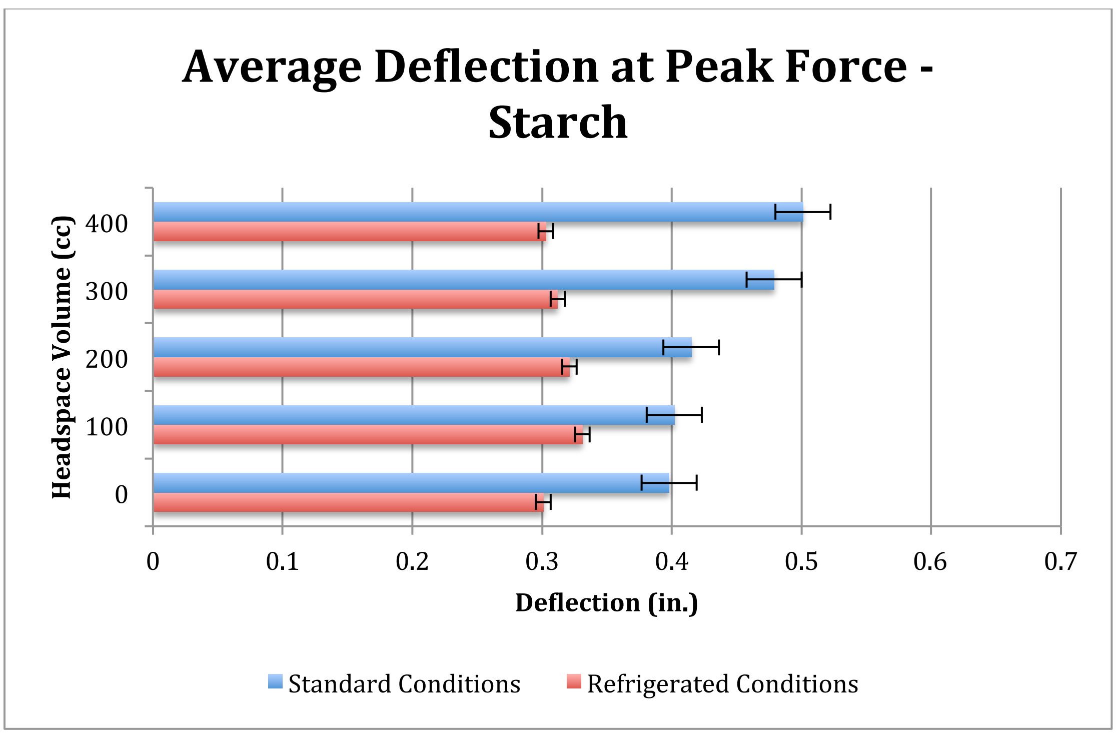

Figure 3.6. Average peak deflection at critical failure for pouches filled with 5% starch solution

Shock:

The critical drop heights displayed in Figure 3.7 show averaged drop height required for a critical failure of the pouches filled with water. A similar trend, noted previously during the compression phase, showed the refrigerated pouches generally had a lower critical drop height than when compared to the standard pouches. An ANOVA was performed on the data set for the water filled pouches and it was concluded at $P<0.05$ there was a difference between the mean critical drop height and pouch headspace volume. Further analysis was conducted comparing the mean drop heights of the standard and refrigerated pouches to the headspace volume. Statistical differences between headspace volumes of 0cc and 100cc were reported, but there was no statistical difference between headspace volumes of 200cc, 300cc, and 400cc at $P=0.05$ when comparing the mean drop height of the standard to the refrigerated pouches. Analysis of the refrigerated pouches revealed there was a difference in the mean critical drop height and pouch headspace volume ($P<0.05$). This trend showed for the refrigerated retort pouches filled with water that increased headspace volume inside the pouch resulted in a greater critical drop height prior to critical failure. The increased headspace volume inside the retort pouch appeared to act as a cushion or shock absorber for the refrigerated pouches allowing them to be dropped from greater heights before failure.

Figure 3.8 displays the averaged critical drop heights required for a critical pouch failure for the pouches filled with 5% starch solution. An ANOVA was performed on the data set for the 5% starch solution filled pouches and at $P<0.05$ there was not a statistical difference between the means. When comparing the two product simulants, the

headspace volume has less affect on pouch performance with the 5% starch solution (more viscous product) than it does with the water filled pouches. The pouches filled with the starch solution were more viscous and did not flow as fluidly as the water filled pouches. Because of this, the force to the seals on the retort pouches were not as great with the starch filled pouches as they were with the water filled pouches. For the water filled pouches the headspace volume appears to aid in cushioning the pouches during the drop whereas this was not required for a more viscous product.

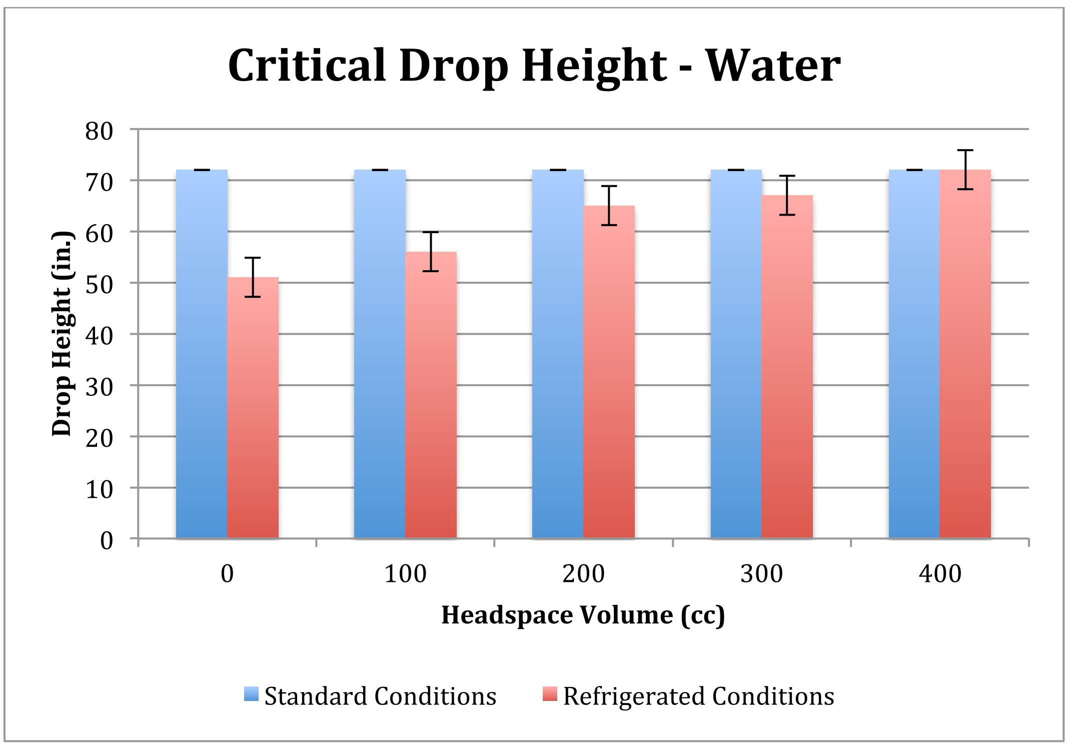

Figure 3.7. Comparison of critical drop height – water filled pouches

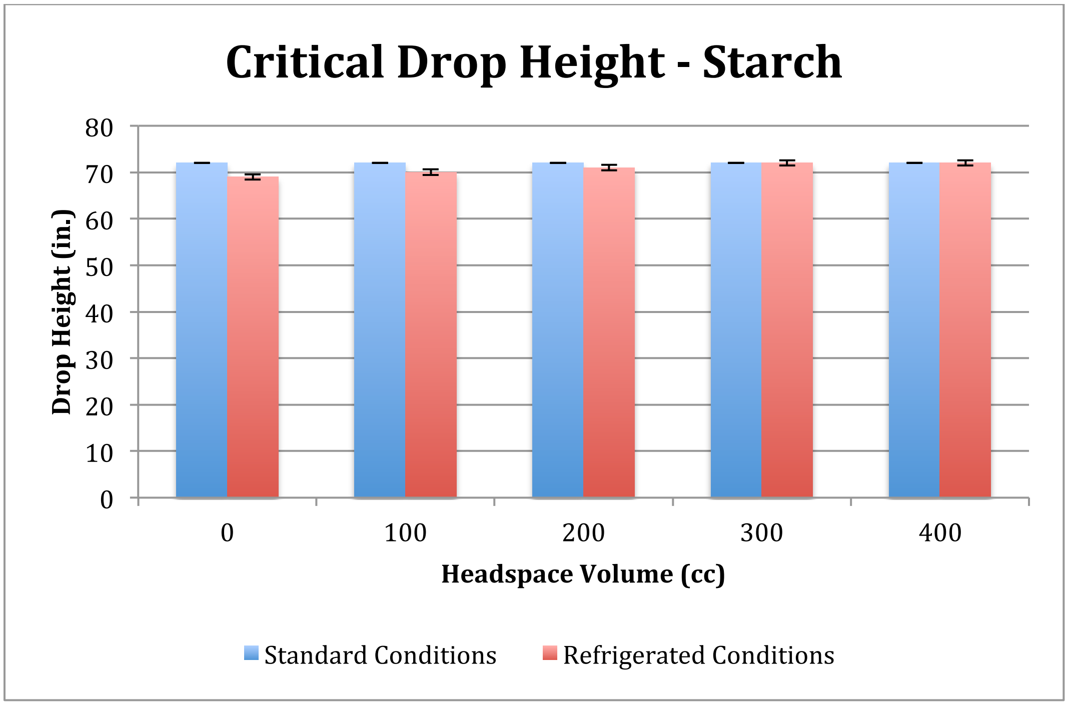

Figure 3.8. Comparison of critical drop height – 5% starch solution filled pouches

CONCLUSIONS

This research study examined the effects of individual laboratory simulated transport hazards on retort pouches filled with different food simulants while varying the headspace volume. These pouches were then processed and stored in either standard or refrigerated conditions prior to being evaluated by laboratory simulated engineering tests. It was determined sinusoidal vibration was not a critical distribution hazard to the retort pouch. Both simulants along with the varied storage conditions recorded no critical failures, and no statistical difference was determined for the headspace volume.

Compression of the individual pouches showed statistical differences between the storage conditions, but no statistical differences between the headspace volume ($P<0.05$). This was a result of the CPP becoming brittle at the refrigerated conditions. The pouches

with greater headspace volume could be deflected more before the pouch seal ruptured due to the headspace volume being compressed.

Shock testing showed that headspace volume did have an effect on pouch performance, especially at the refrigerated storage conditions. For pouches stored at refrigerated conditions the greater the headspace volume the higher drop height the pouch could withstand. Analysis shows there was not a statistical difference between the drop height and headspace volume for the 5% starch solution pouches, but there was for the water filled pouches ($P<0.05$). The shock test concluded headspace volume has a greater affect on pouch performance for lower viscosity food products than for more highly viscous products.

This research explored headspace volume, product viscosity, and storage temperature and how each affects a retort pouch during laboratory simulated hazards. Although retort pouches are shelf stable and do not require refrigeration, pouches could be exposed to extreme temperatures during transportation throughout the supply chain from manufacturer to consumer. The research showed that increasing the headspace volume inside a retort pouch could increase the pouches ability to arrive safely to the consumer, especially in cold temperatures.

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CHAPTER FOUR

EFFECTS OF TRANSPORTATION HAZARDS ON BARRIER PROPERTIES OF GAS FLUSHED RETORT POUCHES

Abstract:

Clear high-barrier retort pouches were filled with water and gas flushed with nitrogen. Two headspace volumes were utilized for this study: 200cc and 400cc. Each retort pouch was equipped with an OxyDot® which non-invasively measured the percent oxygen. The retort pouches were packaged inside regular slotted containers (RSC) and were subjected to laboratory simulated transportation performance tests for small parcel and unit load delivery systems. For the transportation tests, control samples of each variable were used for comparison. Oxygen ingress was measured for the various retort pouches for 63 days. Results indicated there were significant differences in all variables when comparing the transported samples to the control samples. When comparing the different headspace volumes, the 400cc pouches yielded less oxygen ingress into the pouch as compared with the 200cc pouches for the small parcel simulation ($P<0.05$). There were not significant differences for the two headspace volumes for the unit load simulation. Results from this study indicate that for individual packaged products being distributed through small parcel supply chains, which are handled more vigorously than those being shipped palletized, the use of increased headspace volume may aid in protection of certain distribution channels.

Introduction:

An alternative package format for traditional industrial canning methods is the flexible retort pouch. The institutional retort pouch is gaining popularity as a replacement for metal cans, especially in foodservice operations where companies are restricting the use of metal containers in food preparation areas [1]. Additionally, clear retort pouches, which were employed for this study, are becoming more widely used in applications where the ability to microwave, visibility of the product, and metal detection capabilities are of importance [2]. While these features provide advantages to the product manufacturer and consumer, retort pouches do have disadvantages to the metal can such as slower filling speeds and lack of physical durability.

Traditional glass or metal packaging materials provide ultimate barrier protection, but flexible packages, like retort pouches, made from polymers are permeable at different degrees to small molecules like gases, water vapor, and organic vapor and to other low molecular weight compounds like aromas, flavor, and additives present into food [3]. The rate of transfer of these molecules ranges dependent on factors like solubility and temperature, and can be dependent on the type of barrier material used and its properties. The knowledge of the diffusion and permeation behaviors of these molecules through the polymer film has become more and more important in recent years, especially for polymers used in the field of food packaging where contamination from external environment has to be avoided and the shelf life of the food is largely based on these assumptions [3]. Many factors influence the performance of flexible packages and all must be taken into consideration to design the correct package solution.

Clear high-barrier packaging films, such as silicon oxide (SiOx), ethylene-vinyl alcohol (EVOH), and aluminum oxide (AlOx), have become increasingly used for food packages and applications for food package systems. Product requirements and candidate materials are then matched to either determine the shelf life obtainable in a specific material, or to determine which material will supply a specified shelf life [4]. Often times the material data sheet of the film is used to determine what the expected shelf life of the product will be. The material data sheets can be useful in determining the performance of the film, but there are some drawbacks. For example some materials, such as EVOH, can experience an adverse effect as a result of thermal processing, which is commonly referred to as retort shock [5]. This phenomenon has an adverse effect on the performance and shelf life of products packaged in this material. When ceramics are used to improve the gas barrier properties, there are other factors that greatly influence and dominate the gas permeability parameter. These include the presence and the amount of defects in the coating oxide, pinholes, grain boundaries, and microcracks [3]. One thing missing from the literature is the effect of transportation on these high barrier films. The abuse from packages being distributed can result in additional abrasion and flexing, which can have an increased effect on the physical properties of the material that affect the barrier properties. These can affect shelf life if the flexing and cracking that can occur during transport of the material is enough to allow more oxygen ingress than was originally calculated for.

One of the critical factors in the selection of packaging materials is obtaining the gas transmission properties of the material. Oxygen transmission rate (OTR) is one of the

most important of the gas transmission properties because if the amount of oxygen ingress is excessive, it can result in unwanted reactions within the product inside the package [3]. Gas transmission properties are evaluated under steady-state conditions, using a relatively small surface area of the material to determine things such as OTR, which are available and published by manufacturers of flexible materials. These measurements and material data sheets are extremely accurate in their characterization of the materials in steady state conditions. This research evaluates the percent oxygen inside packaged products to determine if adverse effects occurring during the distribution of goods result in an increased amount of percent oxygen inside the package.

A relatively new non-destructive technique analyzes packages using fluorescent decay, which is referenced in ASTM F2714 [6]. Through real time, oxygen ingress that is non-invasive and passive, can be measured with an OxySense Gen III 300 system (OxySense, Inc., Dallas, TX). This system consists of two parts, an oxygen sensor, and a master box that evaluates and interprets the findings. The oxygen within an enclosed system is measured without destroying or altering the internal environment [7]. This is an added benefit as the same package system can be sampled indefinitely. Oxygen concentration measurements are possible based on fluorescence quenching. The OxyDot® is comprised of an oxygen sensing dye immobilized in polymer that can withstand high temperature and pressure processes yet is permeable to gas. The OxyDot® absorbs blue light emitting diode (LED) light and fluoresces light in the red region. Figure 4.1 represents the fluorescence decay over time. When oxygen is absent the OxyDot® will emit an intense red light for 5µs whereas when oxygen is available the

light intensity and emission is decreased to ~1μs. The decrease in intensity and emission can be calculated to accurately provide the amount of oxygen available. The OxyDot® does not consume oxygen in the process and the test can be repeated quickly (5 seconds) and indefinitely [8].

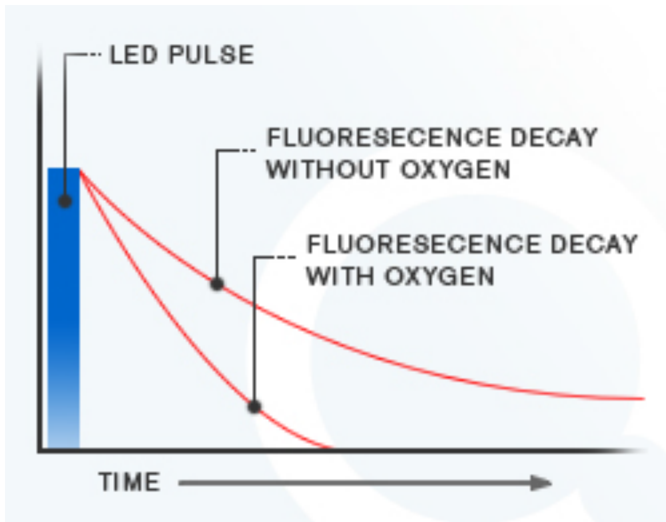

Figure 4.1. Graphical representation of Fluorescence decay with and without oxygen, within 5% accuracy of the reading. [8]

Throughout various distribution channels, packaged products are subjected to three major categories of dynamic hazards: shock, vibration, and compression [9]. Environmental conditions at which the package is transported and stored is another hazard having an adverse effect on package performance. Determining how a package will perform against these hazards is vital to establishing the success of a package. The performance of a packaged product can be evaluated in a laboratory setting using industry accepted test procedures to simulate the transport channel or packaged products can be field-tested. Laboratory testing provides the user with immediate feedback, and a more controlled and repeatable test environment. Field-testing provides more accurate

representation of handling and transport, but with very little control and repeatability of certain hazards. The International Safe Transit Association (ISTA) is an organization that publishes test procedures used to evaluate packages and unit loads passing through various distribution channels. Two of the more common test procedures published by ISTA is one simulating the small parcel environment (individual packaged products) and one simulating the unitized environment for palletized loads. The small parcel general simulation test procedure is ISTA 3A and the general simulation for unitized loads is ISTA 3E [10].

Previous research has shown the ability of institutional retort pouches to withstand harsher distribution hazards by increasing headspace volume (gas) inside of the retort pouch [11]. While this is an additional cost to the overall processing and packaging of goods, it has been shown that it can to lead to reduction of critical defects and product loss for products with low viscosities.

Once food comes in contact with air, it can suffer from physical, enzymatic, microbiological, and biochemical deterioration. Modified atmosphere packaging (MAP) is a process for packaging food together with a gas or a gas mixture. MAP gases are used to replace the air inside the packaging and eliminate or reduce any product deterioration. Common gases used for MAP applications are nitrogen (N_2), carbon dioxide (CO_2), and oxygen (O_2). The research for this project involved gas flushing with nitrogen, which is a type of mechanical replacement of oxygen. Traditionally, MAP is designed to extend the shelf life of the product by controlling and/or modifying the internal atmosphere inside the package. This research assessed whether the use of gas flushing could also act as an

air cushion, protecting the package through common hazards occurring during the transportation of packages.

The goal of this study was to determine if increasing the headspace volume reduces the amount of oxygen ingress over time into packages containing retort pouches traveling through two common distribution channels – small parcel delivery and unit load shipments. To accomplish this, retort pouches were gas flushed with nitrogen and the percent oxygen content was monitored. Because intermittent monitoring of the pouches was required for this study, a non-destructive oxygen analysis of packages using fluorescent decay was used.

Materials and Methods:

The institutional pouch chosen for this experiment was a 1.5 kg four sided seal retort pouch constructed of aluminum oxide (AlOx) coated polyethylene terephthalate (PET)/biaxially oriented nylon (BON)/cast polypropylene (CPP) (Cryovac® Sealed Air, Duncan, SC). Each retort pouch was affixed with an OxySense® Dot used to monitor the oxygen levels inside of the pouches. The Dot was adhered using Momentive RTV 118 adhesive to the middle of the package. The retort pouches were filled with water representing products having a low viscosity. The average filled pouch weight of the water pouches was 1.37 ± 0.12 kg.

The retort pouches were gas flushed with ultra high purity (UHP) nitrogen (N_2) using a Koch Ultravac® 250. The headspace volumes employed for this experiment were 200 cubic centimeters (cc) and 400cc. In order to calculate headspace volume, the sealed pouch was submerged in water with a graduated cylinder. The pouch was opened and the

pouch was slowly compressed into the opening of the graduated cylinder. The displaced volume inside the graduated cylinder was recorded. Five samples from each headspace volume were randomly selected and the average overall gas content was determined for that variable.

Prior to filling the retort pouches a seal analysis was performed on the pouch to determine the optimal seal temperature for the pouches using the Koch Ultravac® 250. The seals were evaluated using a SATEC Universal Tester and following ASTM F88 [7]. Since heat seal temperature is not an adjustable function of the equipment, dwell time was varied to determine optimum seal time producing the strongest seal. Based on the results of the seal curved developed for the pouch, the optimum dwell time was 1.6 seconds for the retort pouches.

Upon filling, the pouches were immediately placed into the retort for processing. A pilot scale rotary retort was employed for this experiment. The filled pouches were processed for 30 minutes at 121°C and 30 psi using a Surdry Model APR-95 Rotary Pilot Retort (Stock America, Cary, North Carolina). After being processed the pouches were stored at 23 ± 1°C and 50 ± 2% RH (standard conditions) per ASTM D4332-13 [7].

For the simulated laboratory distribution testing, five retort pouches were placed inside a corrugated regular slotted container (RSC) having an edge crush test (ECT) value of 44 lbs/in. The pouches were stacked on top of each other as shown in Figure 4.2. The corrugated boxes were designed with zero headspace inside the case.

Side View of RSC →

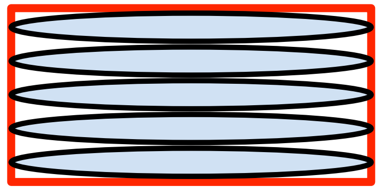

Figure 4.2. Schematic of pouch orientation inside RSC

The retort pouches were measured for oxygen content throughout the testing of both the small parcel and unit load simulations. An initial oxygen content measurement was recorded after the retort pouches were sealed. The pouches were again analyzed and oxygen content was recorded after thermal processing. Prior and following the transport simulation experiments the pouches were evaluated for oxygen content. After transport simulation experiments were completed, the pouches were analyzed and oxygen content was recorded every 7 days for two months. Aside from the transportation simulation and percent oxygen measurement the pouches were stored at standard conditions.

In addition to the instrumented test pouches used for this experiment, a set of control pouches equipped with OxySense® Dots, which were only thermally processed, were used as a baseline for comparing the two test phases. The control pouches were stored at standard conditions and percent oxygen measurements were taken every 7 days over the life of the study.

Small Parcel Simulation

RSC's being used for evaluation for small parcel evaluation were filled with five institutional retort pouches and were closed using pressure sensitive adhesive tape. Five packaged products per headspace volume were evaluated using laboratory simulation referencing the following test protocol: ISTA 3A – Packaged Products for Parcel

Delivery System Shipment 70 kg or Less. For both headspace volumes, the experiment was replicated. Table 4.1 outlines the overview of the testing phase.

Table 4.1. Small Parcel (ISTA 3A) and Unit Load (ISTA 3E) Simulation Overview

ISTA 3A and 3E Simulation Overview				
Headspace Vol. (cc)	**ISTA 3A** Sample Size (No. of RSC)	**ISTA 3E** Sample Size (No. of RSC)	Testing Type	Experiment Number
200 ± 9	1	1	Control	1
	5	7	Simulation	1
	1	1	Control	2
	5	7	Simulation	2
400 ± 12	1	1	Control	1
	5	7	Simulation	1
	1	1	Control	2
	5	7	Simulation	2

Changes in oxygen content from initial measurements for each pouch were computed for analysis. Observed initial differences were noted driving the decision to perform statistical analysis on the data set. A mixed model analysis of variance (ANOVA) was used to evaluate the impacts of pouch position, headspace volume, and simulation type on observed oxygen changes. Fixed effects included pouch position, headspace volume, and simulation type, as well as their interactions. Random effects included run and individual box. Interaction and/or main effects were evaluated as appropriate using least squares means. Separate models were run for each data collection day (i.e. Day 0, 1, 2, …63). All analyses were conducting using the Mixed Procedure in SAS®, with a significance level of 0.05 used for hypothesis test conducted.

Unit Load Simulation

Packaged products evaluated by unitized load simulation were palletized onto a standard Grocery Manufacturer's Association (GMA) wood pallet and stretch wrapped in a 3 x 2 x 3 wrapping pattern with 50% overlap using 80 gauge stretch film. Seven RSC's containing instrumented retort pouches were column stacked in one corner on the wood pallet. The remaining pallet was made up of RSC's containing dummy filled pouches to simulate a complete palletized unit load. One pallet was tested using laboratory simulation referencing the following test protocol: ISTA 3E – Unitized Loads of Same Product. The experiment was replicated for both headspace volumes. Table 1 outlines the overview of the testing phase.

Changes in oxygen content from initial measurements for each pouch were computed for statistical analysis. A mixed model analysis of variance (ANOVA) was used to evaluate the impact of box position within stack, pouch position within a box, headspace volume, and simulation type on observed oxygen changes. Fixed effects included box position, pouch position, headspace volume, and simulation type. Random effects included run, individual stack of boxes, and individual box. Separate models were run for each data collection day (i.e. Day 0, 1, 2, …63). No pouch position effects were observed for any of the collection days evaluated. Contrasts were used to test for box position effects within each headspace treatment. Additional contrasts investigated the difference between control and treatment boxes separately for each headspace volume, as well as differences between the two headspace volumes for treated boxes. All analyses

were conducting using the Mixed Procedure in SAS®, with a significance level of 0.05 used for hypothesis test conducted.

Results:

Small Parcel Simulation

The overall percent oxygen inside the retort pouches increased as a result of the simulated testing conducted. Based on the results shown in Figure 4.3, the overall percent oxygen increased inside the retort pouch as a result of the simulated transportation test. For both headspaces evaluated, the percent oxygen inside the retort pouch was significantly different ($P<0.05$) than the control pouches, which were only thermally processed. Starting at Day 28 and continuing to Day 63, there is a statistical difference ($P<0.05$) between the two simulated headspace volumes. As is illustrated by Figure 4.3, the test pouches flushed with 400cc on average provide better protection against the simulated hazards resulting in a lower percent oxygen content than the pouches with 200cc of nitrogen.

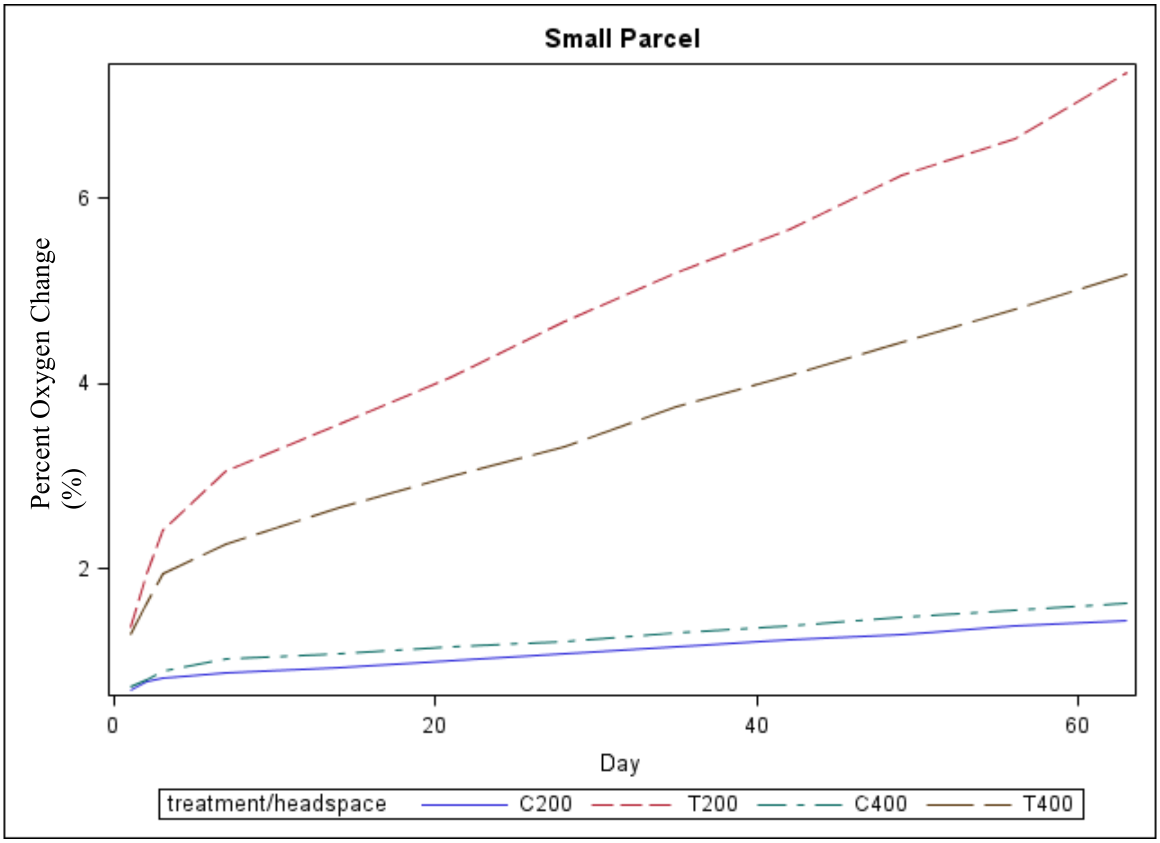

Figure 4.3. Percent oxygen inside retort pouches used during small parcel simulation

Unit Load Simulation

The overall percent oxygen inside the retort pouches increased as a result of the simulated testing conducted. Based on the results shown in Figure 4.4, the overall percent oxygen increased inside the retort pouch as a result of the simulated transportation test. For both headspaces evaluated, the percent oxygen inside the retort pouch was significantly different ($P<0.05$) than the control pouches, which were only thermally processed. No statistical differences were observed between the different headspace volumes evaluated during this experiment.

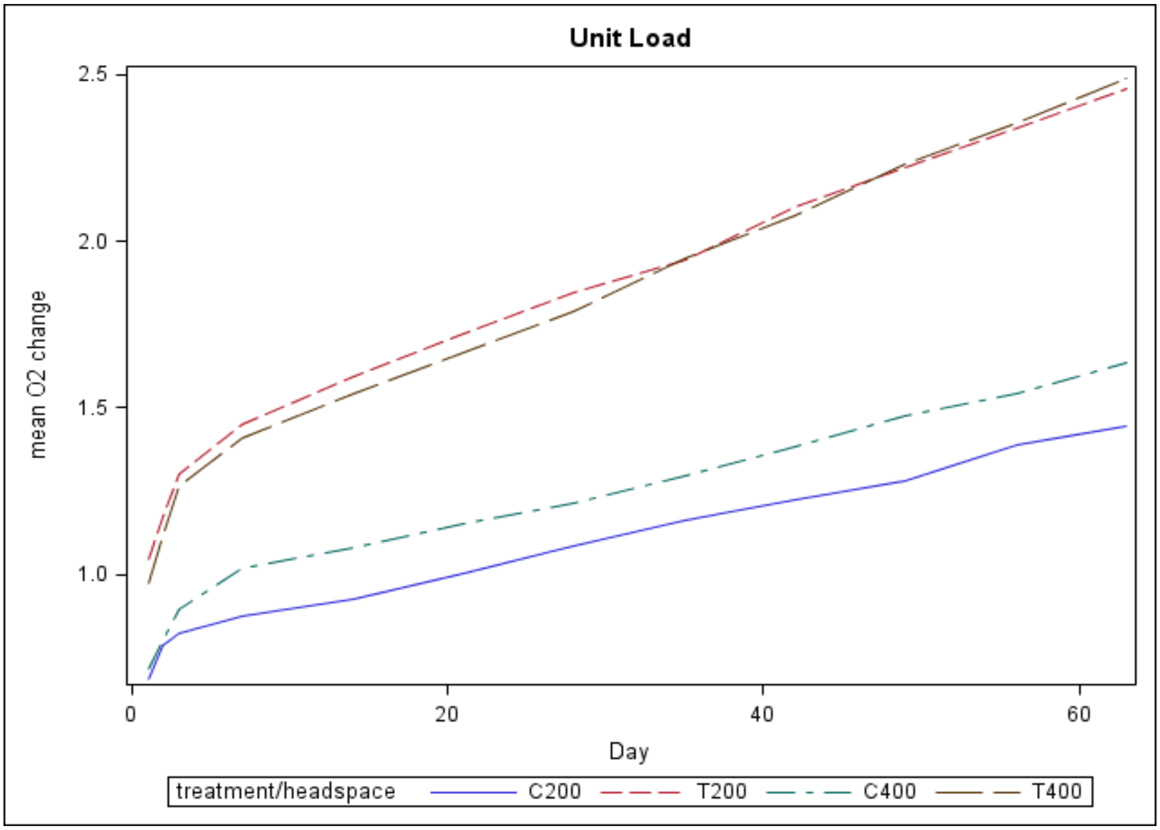

Figure 4.4. Percent oxygen inside retort pouches used during unit load simulation

Discussion:

Gaseous atmosphere inside a package can change as a result of diffusion through the package material and loss of seal integrity. Diffusion can be defined as the movement of molecules from a region of high concentration to as area of low concentration as a result of intermingling of molecules due to random thermal agitation [12]. The diffusion of molecules into polymers is a function of both the polymer and the diffusant. Factors which influence diffusion include: (1) the molecular size and physical state of the diffusant; (2) the morphology of the polymer; (3) the compatibility or solubility limit of the solute within the polymer matrix; (4) the volatility of the solute; (5) and the surface or interfacial energies of the monolayer films [13]. Gas transport through materials can take

place by two mechanisms: diffusive flow via solubility-diffusion or flow through defects in the material such as pinholes, porosities, microchannels, and microcracks. Oxygen permeation through high barrier coatings is dominated by flow through defects in the coating [14]. These defects are only enhanced as a result of transportation. Figure 4.5 provides an illustration of the type of defects occurring in the areas of the stress cracks to the high barrier coatings that were observed during this research. It should be noted the stress cracks and pinholes were abundantly more present in the pouches examined during the small parcel simulation and were the pouches having the lower of the two headspace volumes.

Figure 4.5. Defects noted to the barrier layer of the pouch (10x magnification)

Packages, especially thermally processed packages, must be able to withstand and protect against various hazards during storage and distribution, while maintaining the sterility of the package and integrity of the product. Forces exerted on the package or that result from movement of the contents within the package may inflict damage without affecting the hermetic seal [15]. The magnitudes of such forces incurred by or generated within a food package during distribution and the significance of such forces to the maintenance of the sterility of the package are not known [16-18].

The integrity of packages can be lost during the storage and distribution due to the presence of defects in the seal area or package body as a result of abusive handling [15]. Heat seals in packages are usually formed by applying thermal energy to the contacting surface of the layers of materials to be sealed, either directly by heat conduction or indirectly by high-frequency electromagnetic induction [19]. All of these types of seals are subject to the formation of defects from a variety of sources. Wrinkles in the materials, inclusion of foreign materials, and post sealing mechanical damage are all sources of defects which may compromise the seal integrity of the package [19].

During small parcel transport, packaged products can be handled and stowed in multiple orientations [20]. As a result the pouches inside can shift causing the pouches to fold onto themselves. Vibration caused from traveling over the road can cause the pouches to abrade against each other thousands of times during a shipment which could result in flex and microcracks to the barrier coating. The presence of flex cracks has been reported to show increased oxygen ingress for flexible packages containing high barrier films such as AlOx and SiOx [21,22]. The results of this study show the increased headspace volume fills the void inside the pouch reducing the likelihood of flex and microcracking on the barrier coating. The increased headspace volume of the 400cc pouches statistically shows an improvement in the protection against hazards occurring during small parcel modes of transportation. Lower percent oxygen contents were reported for the increased headspace volume at the end of the study, which could lead to an increase in the shelf life for an oxygen sensitive product.

Unit loads are handled much differently during transportation as compared to packages traveling via small parcel delivery systems [23]. During unit load shipments, packaged products are placed onto a pallet in proper shipping orientation and stretch wrapped or banded together. These packages are then handled as one unit creating less opportunity for packages to be positioned in multiple orientations during transport. For most shipments, unit loads are not subjected to the severity of shocks and movements occurring during small parcel delivery [24]. Because of this, pouches are unlikely to shift dramatically inside the container resulting in less opportunity for flex and microcracks to occur. The pouches evaluated during this experiment behaved similarly. Both of the headspace volumes evaluated produced very similar results in the amount of percent oxygen observed during the study. Based on the results from this study, headspace volume does not have an effect on the oxygen barrier performance of a barrier coated retort pouch traveling through a simulated unit load distribution environment.

Conclusions:

Examined, were the effects of varying headspace volume inside a retort pouch to determine if the increased headspace volume provided any additional physical or barrier protection to the product during laboratory simulated transport performance tests designed to replicate small parcel and unit load shipments. The retort pouches were gas flushed with different volumes of nitrogen and thermally processed prior to the transport simulations. A non-destructive analysis technique using florescent decay was employed to measure oxygen ingress into the pouches over 63 day period.

Previous research studies have not provided a correlation between headspace volume and the potential to cushion and protect the contents of a package. This research project utilized water as a food simulant due to its low viscosity. Using a low viscosity fluid like water it provided an extreme scenario for products typically thermally processed in retort pouches. The dynamic behavior of the fluid provided stress to the seal areas and because it was a flowable product also allowed for the pouches to fold onto themselves promoting flex and microcracks in the pouches.

Results from this study showed there was, on average, less percent oxygen present in the retort pouches containing 400cc of nitrogen than the pouches containing 200cc as a result of the small parcel simulation ($P<0.05$). This was as a result of the extra headspace volume protecting and cushioning the pouches during the simulation. The analysis performed on the pouches utilized for the unit load testing showed there was not a statistical difference in the percent oxygen present for the two different headspace volumes ($P<0.05$). Individual packaged products being distributed through small parcel supply chains are handled more vigorously than those being shipped palletized indicating the use of increased headspace volume may aid in the protection of the oxygen barrier coating for barrier coated packages passing through certain distribution channels.

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CHAPTER FIVE

THE EFFECTS OF TRANSPORTATION HAZARDS ON SHELF LIFE OF PACKAGED POTATO CHIPS

Abstract:

Kettle cooked potato chips packaged inside metallized oriented polypropylene bags were used to evaluate the effects of simulated transportation on shelf life. While this process provides accurate results for shelf dating, the research project assessed whether or not subjecting packaged products to known transportation hazards can increase or accelerate the deteriorative factors that affect the shelf life of a produce. By stressing the packages through laboratory simulated transportation hazards, it can create failures to the packages that would not have normally shown up during a traditional test protocol, such as accelerated shelf life testing (ASLT). This study showed that the transportation of food packages has a great affect on the performance of the package, ultimately affecting the quality of the food product. Outcomes from this study showed the rate at which some properties of the food and package can increase as a result of the simulated transportation, versus standard ASLT with only increased temperature and humidity. Differences of significance were observed between the simulated transportation and the standard ASLT samples when comparing headspace composition, moisture content, and TBA testing ($P < 0.05$).

1. Introduction:

Potato chips have become America's favorite snack, with U.S. retail sales over $6 billion a year [1]. Product development has led to the introduction of various styles and flavors of the potato chip, each with their own unique twist on an age old classic. Kettle cooked potato chips are promoted as a healthier alternative to the fried potato chip. The cooking process of kettle chips is done at low temperatures, and the natural flavor of potatoes are maintained due to minimum usage of artificial or excess ingredients in the preparation process [2]. Though kettle cooked chips give the impression they are hand made, industrial scale manufacturing is managed with high capacity continuous automated plants.

Regardless of potato chip type it is imperative to understand the intrinsic and extrinsic properties of the food in order to provide the consumer with the highest quality product. Foods that are moisture sensitive, like potato chips, are vulnerable to change in the environment and if left unpackaged will first become stale. Additionally, lipid oxidation can increase depending on the water activity [3]. Lipid oxidation is typically at it lowest near a water activity of 0.4, but increases with both decreasing and increasing water activity levels. These factors are often affected by the packaging material and process. The package serves as a barrier to provide resistance to the diffusion of gases, water vapor, and off-aromas. Furthermore, package atmosphere can be modified to retard deteriorative reactions and increase the expected shelf life of a product.

Modified atmosphere packaging (MAP) is defined as the packaging of a perishable product in an atmosphere having been modified so that its composition is other

than that of air [4]. The three main gases used in modified atmosphere packaging are oxygen (O_2), carbon dioxide (CO_2) and nitrogen (N_2). The choice of gas is very dependent upon the food product being packaged. Gases can be used individually or in combination. Inert gas such as nitrogen has low solubility in both water and lipids, and is useful in applications such as potato chip bags. The intended atmosphere for MAP can be achieved in two fundamental ways. These are the mechanical replacement of air with a gas or gas mixture or by generating the atmosphere within the package either passively, as in the case of fruit and vegetables, or actively by using suitable atmosphere modifiers such as oxygen absorbents [5]. MAP is designed to prolong the intended shelf life of a product through delaying product degradation, which can be onset by exposure to oxygen. In high-fat products, such as potato chips, MAP delays rancidity and preserves the smell, taste, texture and appearance [6].

While the intention of MAP is to extend the shelf life of a product, few studies have shown how or if the atmosphere inside the package changes as a result of the distribution environment. The key issue at present appears to be retention of the food's initial qualities during distribution [7]. Margareetta et al. [8] examined oxygen and carbon dioxide levels of cold cut meat products packaged in MAP at retail levels. The results of this study showed the atmospheres had been altered from the time the package was sealed to when the packages arrived for retail. This study revealed residual oxygen can be higher than acceptable ranges due to various reasons: minor leaks in structure, leaks formed during sealing, or improper or poor flushing of the protective gas. Oxygen

content can also increase if the packages are handled too roughly during distribution or shelving, and at the point of sale [8].

Shelf life can be defined as the period of time from the production and packaging of a product to the point at which the product first becomes unacceptable under defined environmental conditions [9]. The shelf life depends on series of variable parameters, first the product characteristics such as physical, chemical and biological characteristic, second, processing conditions, third, package characteristics and effectiveness, and finally, the environment to which the product is exposed during distribution and storage [3].

Despite the complexity of food systems, the systematic study of their deteriorative mechanisms can lead to sound methods of determining shelf life [10]. The prediction of shelf life can be performed and carried out in two general ways. The most common way of determining the shelf life involves exposing the food product to a single abuse condition for an extended duration, evaluating quality, generally by sensory methods, and then extrapolating the result to normal storage conditions [10]. The alternative method is to utilize a more elaborate design based on principles of chemical kinetics and to determine the actual temperature dependency of various quality attributes [10]. The initial method is commonly referred to as accelerated shelf life testing (ASLT) and is the method employed in this research.

ASLT is a method for testing food products under accelerated conditions to decrease the testing period or allowing more evaluations to be conducted in a timely manner. The basic assumption underlying ASLT is that the principles of chemical

kinetics can be applied to quantify the effects that extrinsic factors such as temperature, humidity, gas atmosphere, and light have on the rate of deteriorative reactions [11]. Although the use of ASLT is widely used, there are, however, potential problems and errors that can arise during the study. It should be noted during the analysis using ASLT is to only estimate the actual shelf life of a packaged product, except in the case of very simple chemical reactions [11]. One way to ensure the ASLT is correct is by conducting actual shelf life studies using the normal handling and storage conditions. Once this is established for a particular product, then ASLT can be used for that product when evaluating different processes and packaging variables [11].

Factors influencing the shelf life of a product are the initial quality and inherent nature of the product, processing methods, barrier properties of the packaging, and the transportation and storage conditions (temperature and relative humidity) [12]. Although the conditions of transportation and warehousing are included, missing from these factors are the other hazards occurring throughout the supply chain. Throughout the supply chain packaged products are subjected to additional hazards; shock, vibration, and compression, which all can lead to adverse effects on the packages integrity [13-14]. Packaged products traveling through the supply chain will be handled, dropped, vibrated, and compressed multiple times until they reach the consumer. Figure 5.1 displays the food deterioration as a function of product distribution. As a result, packages can breakdown and lose their integrity, which can increase deterioration factors of the food and ultimately adversely affect the product’s predicted shelf life. Ways in which the

integrity of a package can be lost during the storage and distribution are due to the presence of defects in the seal area or package body as a result of abusive handling [15].

Heat seals in packages are usually formed by applying thermal energy to the contacting surface of the layers of materials to be sealed, either directly by heat conduction or indirectly by high-frequency electromagnetic induction [16]. All types of seals are subject to the formation of defects from a variety of sources. Wrinkles in the materials, inclusion of foreign materials, and post sealing mechanical damage are all sources of defects that may compromise the seal integrity of the package [16]. The body of the package can lose its integrity as a result of foreign objects damaging the package or by excessive vibration causing flex cracking and leading to defects in the barrier layer [17].

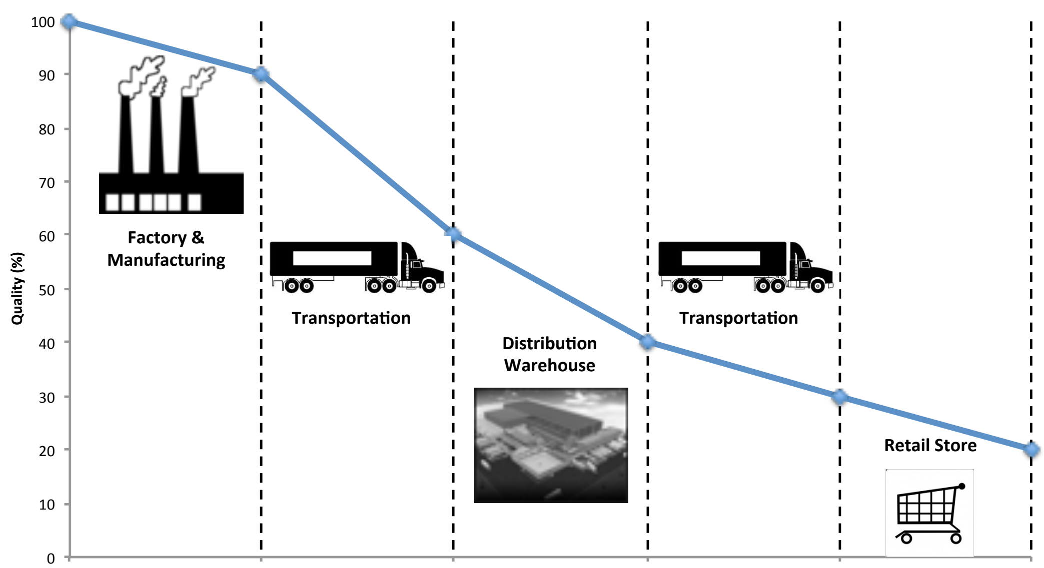

Figure 5.1. Notional food deterioration during the process of distribution [18]

ASLT typically involves the use of increased temperature to accelerate the deteriorative reactions, and doesn't subject the package or the food product to any other distribution hazards, such as shock, vibration, or compression, which packages will encounter during the movement of goods. Therefore part of this research will be evaluating whether the deteriorative rates of certain aspects of the food will be increased as a result of the mechanical and manual abuse the packages will be put through during distribution to the consumer.

The goals of this research are as follows: (1) determine what effects simulated distribution hazards have on package and product integrity and (2) determine if there is a difference in product performance for packaged products only accelerated by temperature as compared to packaged products undergoing simulated distribution hazards as part of the ASLT.

2. Materials and Methods:

2.1. Packaged Product

The packaged product selected for this research was 1.5 oz. bags of kettle cooked potato chips. Packaged products were obtained from three separate production runs. The ingredients used for the product were potatoes, canola oil, and salt. The package was constructed of the following materials: matte oriented polypropylene (OPP)/ink/white opaque low-density polyethylene (LDPE)-ethylene vinyl alcohol copolymer (EVOH)-white opaque low-density polyethylene (LDPE)/metalized oriented polypropylene (Met-OPP). The packages were gas flushed with nitrogen prior to sealing.

2.2. Headspace Analysis

Headspace composition for each sample was determined using a MOCON® PAC CHECK® Model 650 dual headspace gas analyzer for modified atmosphere packages (MOCON Inc., Minneapolis, MN). The needle of the analyzer was inserted through the neoprene plastic pad into the package through which a sample of gas was extracted and analyzed. Headspace gas was sampled directly after packaging and then every 7 days thereafter. The analysis was performed in triplicate for each variable.

2.3. Water Activity

Water activity for each sample was measured using an AquaLab® Series 3TE water activity meter (Decagon Devices Inc., Pullman, WA). 1.0 grams of product was selected from the packaged product and placed into sampling dish. Water activity was sampled directly after packaging and every 7 days thereafter until the end of the study. The analysis was performed in triplicate for each variable.

2.4. Moisture Content

Moisture content of each sample was measured using a Mettler-Toledo HR73 Halogen Moisture Analyzer (Mettler-Toledo GmbH, Laboratory and Weighing Technologies, Greifensee, Switzerland). 2.0 grams of product was selected from the packaged product and placed into sampling dish. Moisture content was sampled directly after packaging and every 7 days thereafter until the end of the study. The analysis was performed in triplicate for each variable.

2.5. Texture Analysis

Texture analysis of the potato chip sample was measured using a TA.XT*Plus* Texture Analyzer (Texture Technologies Corporation, Scarsdale, NY). A fixed flat platen was used to perform the analysis compressing the chips at a rate of 0.5 in/min until breakage of the chip occurred. Peak force in grams (g) was calculated for each chip. Twelve chips for each variable were analyzed per sampling. Texture analysis was sampled directly after packaging and every 14 days thereafter until the end of the study. The analysis was performed in triplicate for each variable.

2.6. Pressure Decay Leak Test

Pressure decay leak testing of the package was measured using a MOCON® Lippke Package Test System 4500 (MOCON Inc., Minneapolis, MN). Fifty percent of the burst pressure for the bag was used for the leak detection. Packages were probed and inflated with 0.60 psi and the decay was monitored for 60 seconds [19]. Three bags from each test variable were sampled directly after packaging and every 7 days thereafter.

2.7. Thiobarbituric Acid (TBA) Test

1.0 g of test product was added to 20 ml of 10% Trichloroacetic Acid (TCA) in a beaker and stirred for 20 minutes. Samples were split equally into two 15 ml falcon tubes and centrifuged for 20 minutes at 2500 rpm. 2 ml of filtrate from each falcon tube was removed and mixed with 2 ml of 0.3% Thiobarbituric Acid (TBA) in falcon tubes. Falcon tubes were placed in a boiled water bath and held for 20 minutes. Tubes were removed and allowed to cool. Tubes were centrifuged for 20 minutes at 2500 rpm. The absorbance

was measured at 531nm using a Genesys 10S UV-Vis Spectrophotometer (Thermo Fisher Scientific, Madison, WI). The value was expressed in terms of optical density (OD).

2.8. Control and Treatment Samples

Control Samples

Control samples did not undergo any accelerated testing or transportation abuse. The potato chips were processed and packaged, as they would normally for production and distribution. The control samples were packaged inside the corrugated shipping container and stored at a controlled atmosphere of 23° ± 2°C and 50% ± 5% RH during the duration of this study.

Treatment Samples

There were two treatment samples for this study and are referred to as Accelerated Shelf Life Testing (ASLT) samples and Transportation and Accelerated Shelf Life Testing (T-ASLT) samples.

ASLT samples were processed and packaged as they would normally for production and distribution. The ASLT samples were stored at a controlled atmosphere of 43° ± 2°C and 80% ± 5% RH during the duration of this study.

T-ASLT samples were processed and packaged, as they would be for production and distribution. Prior to the accelerated shelf life testing, the corrugated shipping containers containing the potato chip bags were subjected to a simulated transportation test. All packaged products were subjected to the International Safe Transit Association (ISTA) 2A test protocol for packaged-products 150 lbs. or less [20]. After the completion

of the simulated transport test, all samples were stored at a controlled atmosphere of 43° ± 2°C and 80% ± 5% RH during the duration of this study.

Packaged products were obtained from three separate production runs and placed inside 32 edge crush test (ECT) regular slotted containers (RSC). Each production run yielded one set of samples for evaluation: Control, ASLT, and T-ALST. Three production runs were sampled allowing for all analyzes to be performed in triplicate.

2.9. Statistical Analysis

Changes in the results from each analysis technique from initial measurements were computed for analysis. A mixed model analysis of variance (ANOVA) was used to evaluate the changes. Fixed effects included analysis technique and treatment type, as well as their interactions. Random effects included production run and treatment type. Interaction and/or main effects were evaluated as appropriate using least squares means. Separate models were run for each data collection day. All analyses were conducting using the Mixed Procedure in SAS®, with a significance level of 0.05 used for hypothesis test conducted (SAS Institute Inc., Cary, NC).

3. Results and Discussion

3.1. Headspace Analysis

The results presented in Figure 5.2 show the changes in headspace oxygen concentration of the three variables evaluated during this study. For each of the variables, the oxygen concentration increased throughout the storage time. The control variables maintained the lowest oxygen concentration over the storage time, while the T-ASLT samples saw the greatest increase in oxygen concentration. Initial through day 7

measurements yielded no significant differences between any of the three variables. Significant differences were noted between the control and the ASLT and T-ASLT beginning on day 14 ($P < 0.05$). Beginning on day 21 and continuing through to the end of the examination period, the T-ASLT and the ASLT samples were significantly different ($P < 0.05$).

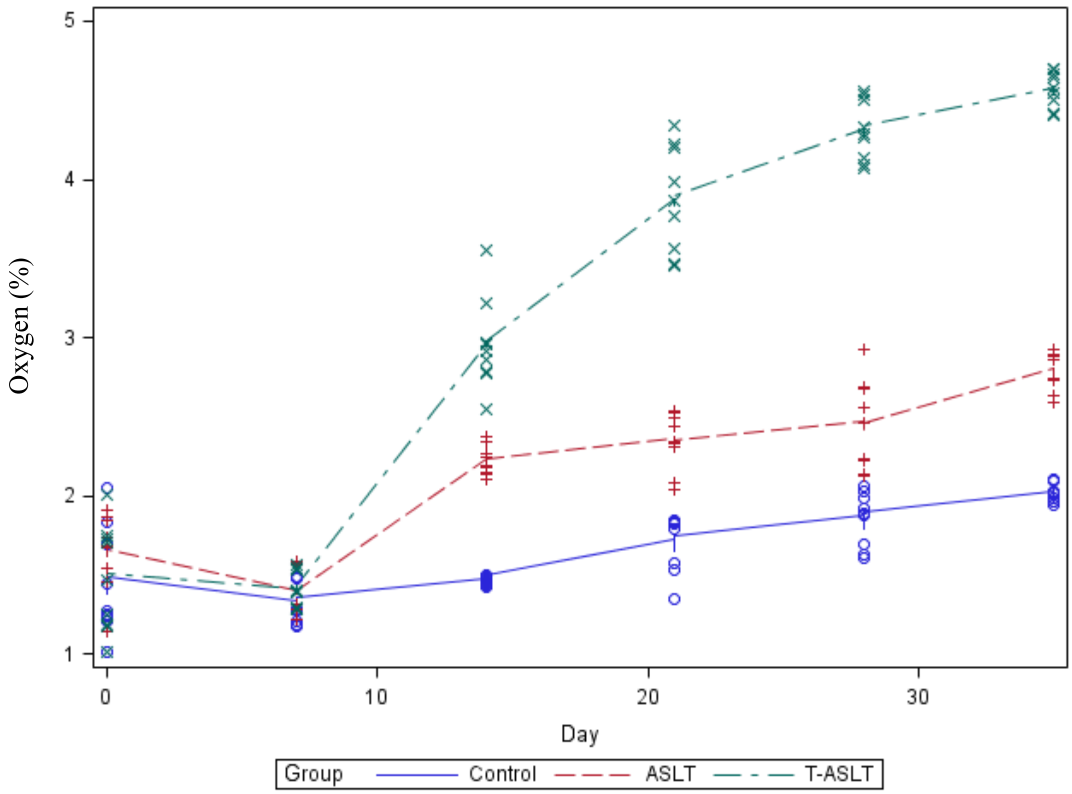

Figure 5.2. Changes in oxygen concentration during storage time

3.2. Water Activity

Figure 5.3 presents the change in water activity of the three variables over the duration of the evaluation period. No statistical differences were noted between the water activity of the control samples and either of the ASLT and T-ASLT samples on the initial measurement at day 0 ($P > 0.05$). Differences between the control and the ASLT occurred

on day 7, but not again until day 35 ($P < 0.05$). Differences between the control and the T-ASLT samples began on day 14 and continued through the end of the study ($P < 0.05$). When comparing the water activity of the ASLT and the T-ASLT, the variables were significantly different from day 14 and throughout the remainder of the study.

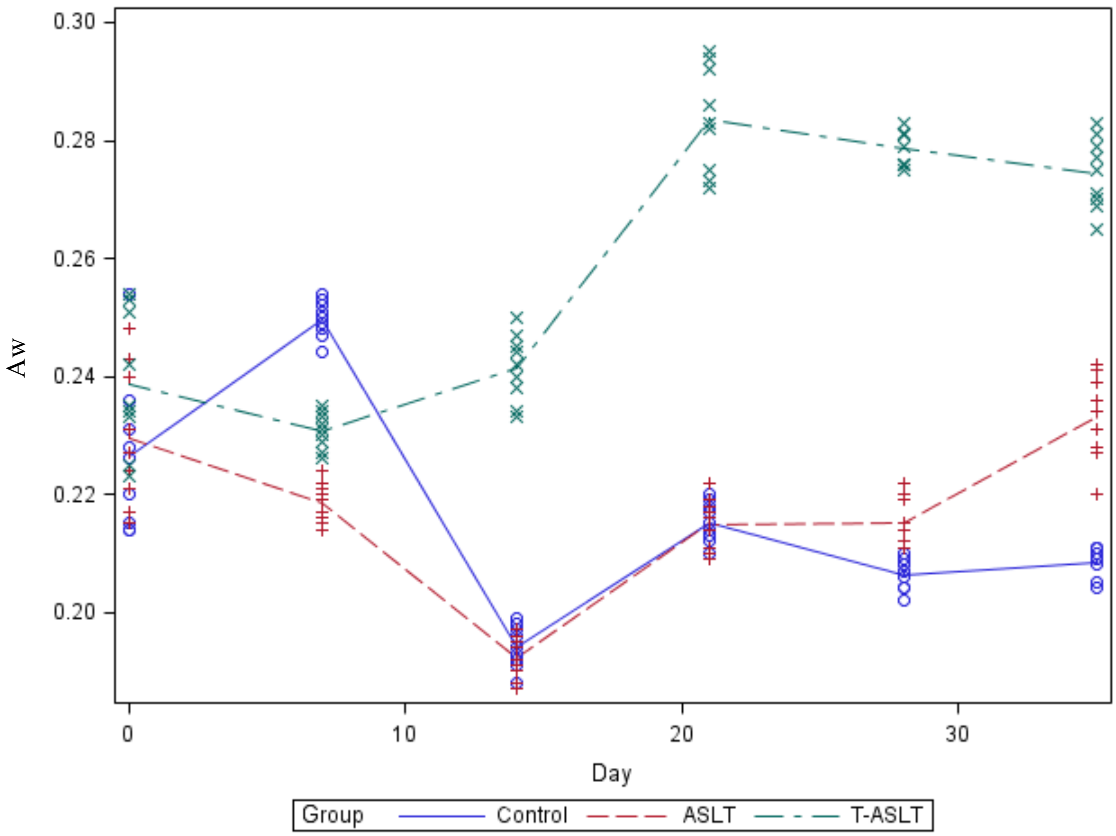

Figure 5.3. Changes in water activity during storage time

3.3. Moisture Content

Results presented in Figure 5.4 show the change in moisture content of the three variables. No statistical significance was determined for the control and the ASLT samples throughout the entire duration of the examination period ($P > 0.05$). Significant differences were noted between the control and the T-ASLT variables beginning on day 14 of the study and they continued to be significantly different throughout the remainder

of the study (P < 0.05). When comparing the ASLT and T-ASLT variables, statistical significance was observed beginning on day 14 and for each measurement throughout the remainder of the evaluation period (P < 0.05). Beginning from day 7, the control and the ASLT variables saw little change over time, whereas there was a steady increase in the observed moisture content of the T-ASLT variables that were sampled.

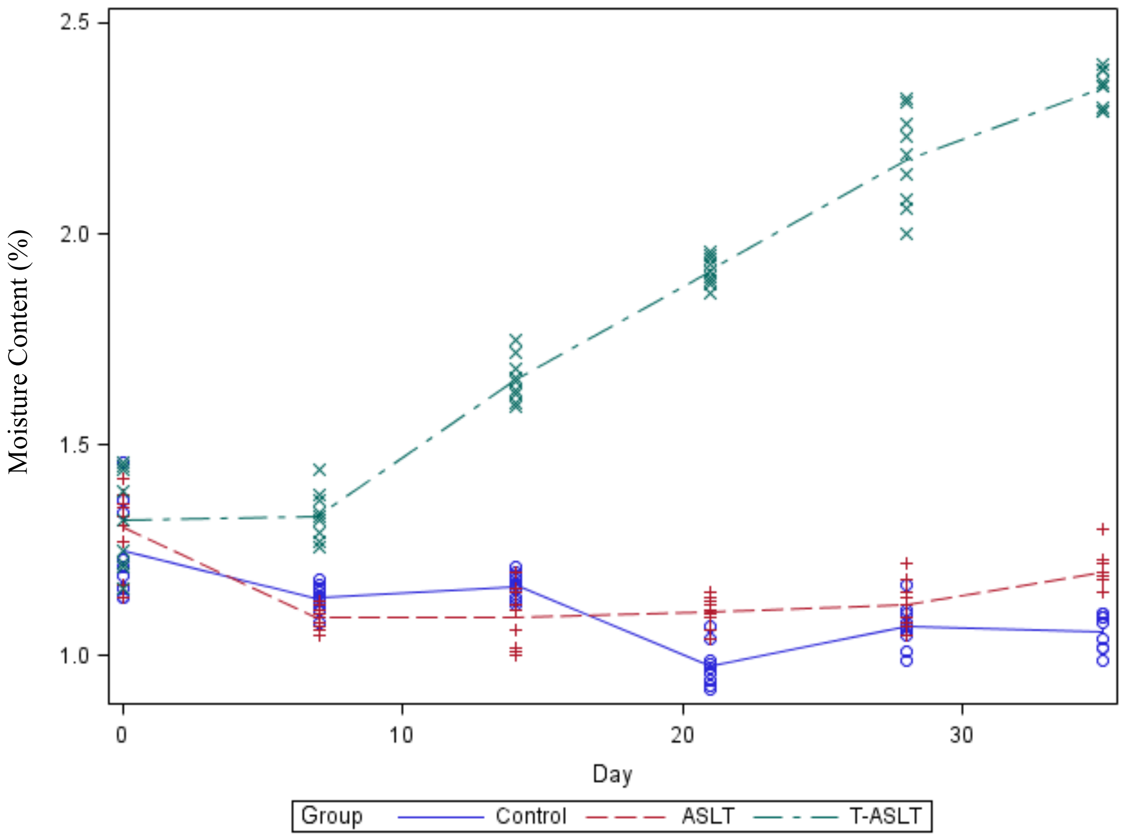

Figure 5.4. Changes in moisture content during storage

3.4. Texture Analysis

Texture analysis was performed on the potato chip samples for each of the three variables being evaluated. Twelve chips per variable and production were evaluated during each examination period. The peak force (g) was recorded for each test and the average of the twelve chips was calculated and used for comparison. The results of the

texture analysis are displayed in Figure 5.5. Comparing the control to both the ASLT and T-ASLT variables, significant differences were noted starting on day 28 ($P < 0.05$). When the ASLT and T-ASLT variables were compared to each other, although on average the T-ASLT required less force to fracture, no statistical differences were observed regarding the required force to create a failed potato chip ($P > 0.05$).

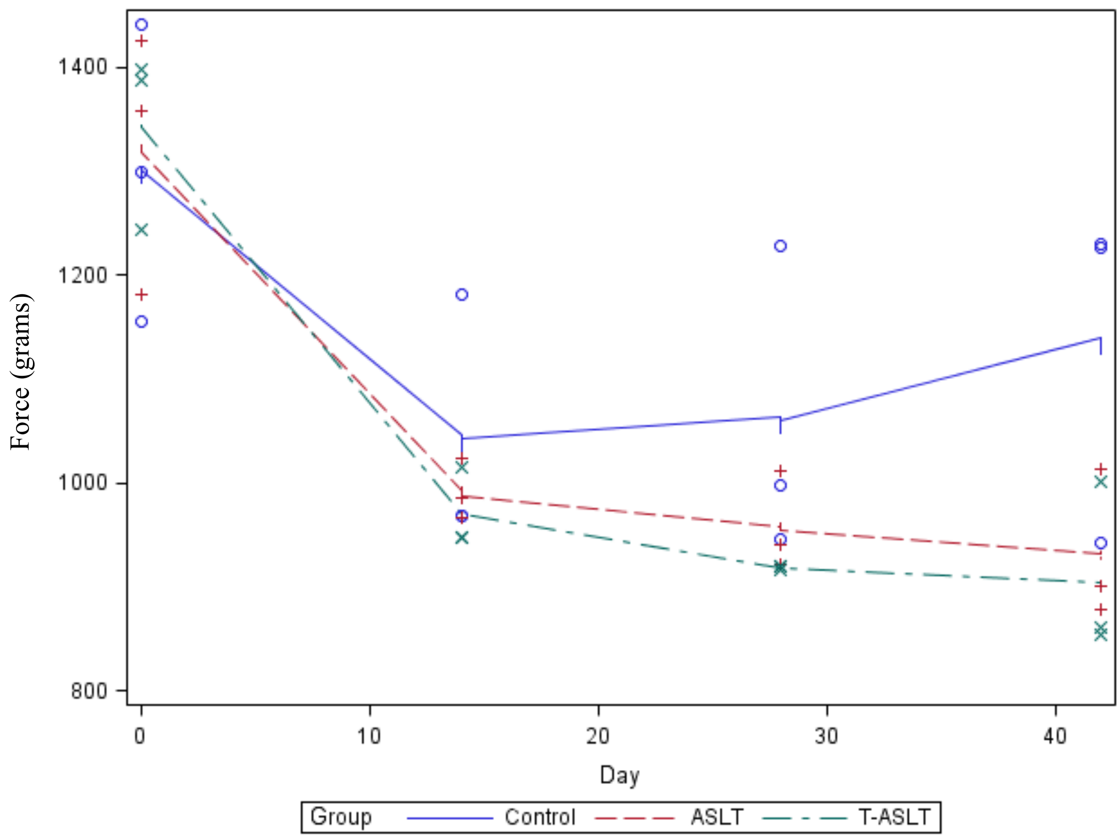

Figure 5.5. Texture analysis results for each variable

3.5. Pressure Decay Leak Test

Pressure decay leak testing was performed on potato chips bags. Pressure drop, recorded in pounds per square inch (psi), was collected throughout the study for the three variables in this study. Results of the pressure decay leak test are represented in Figure 5.6. Significant differences were noted between the control and ASLT and T-ASLT

samples starting on day 14 and day 7 respectively (P < 0.05). No significant differences were observed between the mean pressure drops for the ASLT and T-ASLT samples (P > 0.05).

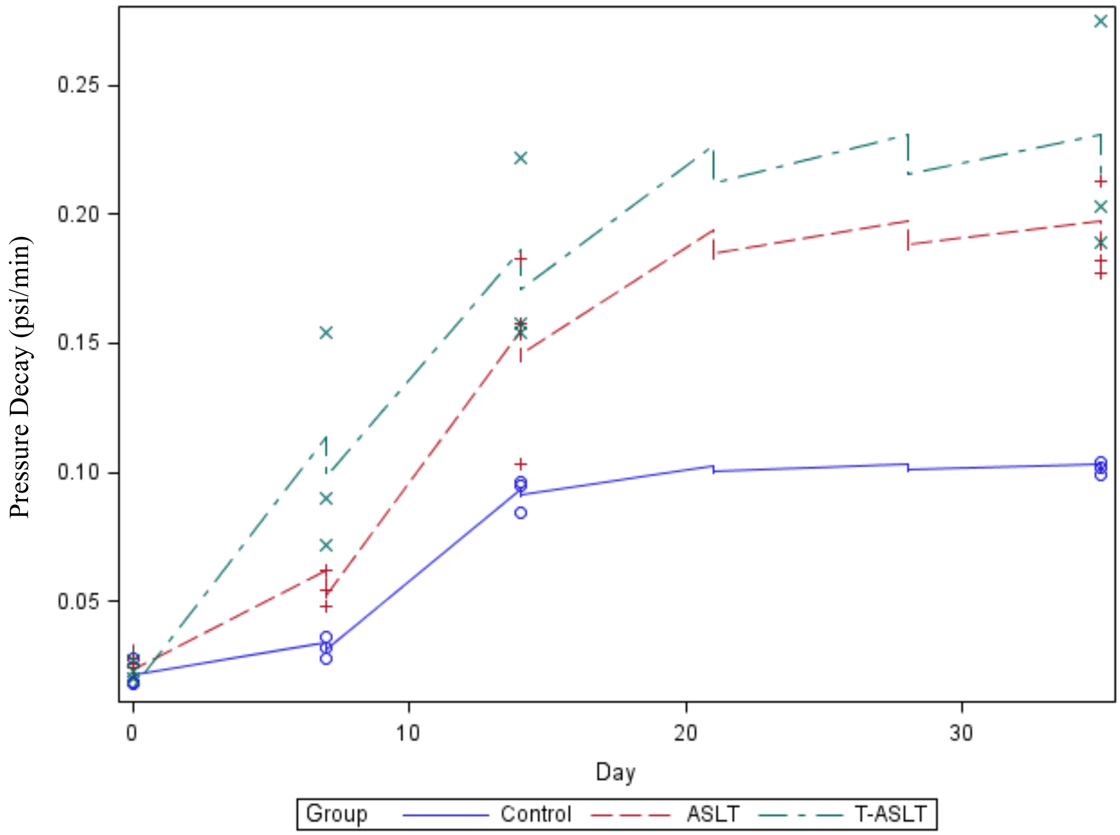

Figure 5.6. Pressure decay leak test results for each variable

3.6. TBA Test

The TBA test was used to detect oxidative deterioration of the potato chip samples. Figure 5.7 displays the results from the TBA analysis performed during this study. Differences of significance were observed between the control and both the ASLT and T-ASLT samples beginning on day 7 and continuing on until the end of the study (P < 0.05). Comparing the ASLT and T-ASLT variables, significant differences were

detected starting on day 14 and for each of the following analysis periods until the end of the study was reached (P < 0.05).

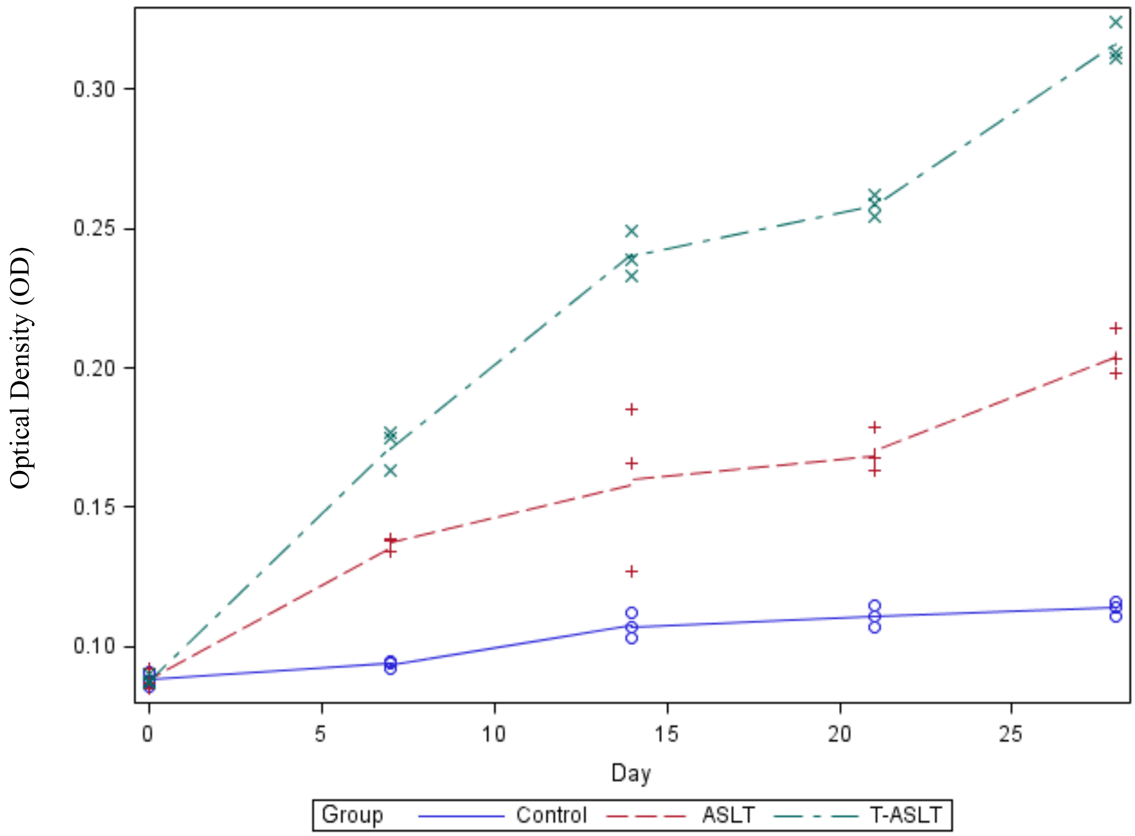

Figure 5.7. TBA analysis for the three different variables

The two major modes of deterioration of fried snack foods are the development of fat rancidity and loss of crispness [11]. Fats deteriorate byway of two mechanisms, oxidative and hydrolytic rancidity, leading to the formation of off flavors and odors. The more important of the two mechanisms is oxidative rancidity, which results in food spoilage. The oxidation of oils is mainly responsible for volatile compound changes in potato chips during storage [11]. The onset of rancidity can be reduced, but the product must be protected from oxygen and light both of which catalyze fat oxidation, or antioxidants, such as butylated hydroxyanisole (BHA) or butlyated hydroxytolune

(BHT), can be added into the product formula or packaging [9 and 21]. A by-product of moisture gain of the product is the loss of crispness. Water affects the texture of the snack foods by plasticizing and softening the starch/protein matrix, which alters the mechanical strength of the product [11]. The loss of crispness can be combated through the use of good moisture barrier films.

The TBA test provides means for evaluating lipid oxidation in foods, especially on a comparative basis [22]. Figures 2 and 7 both show the T-ASLT samples had a greater amount of oxygen in the headspace atmosphere as well as had higher TBA values, respectively, as compared to the control and ASLT variables. These two variables were statistically evaluated and the coefficient of determination (R^2) was computed for the TBA and the percent oxygen measurements. Computed results showed there was a strong correlation between the TBA and percent oxygen measurements of the T-ASLT samples (R^2=0.92), and there was only moderate correlation between the TBA and percent oxygen measurements of the Control (R^2=0.61) and ASLT (R^2=0.55) samples. This shows that transportation simulation of the packages resulted in a breakdown of the barrier layer and/or the stresses occurring to the packages as a result of the simulation increased the existing defects which occur during the forming and sealing of the package. However, no statistical differences in pressure decay were observed between the ASLT and T-ASLT samples. The increased oxygen levels as well as light emitting through the package can increase the oxidative rancidity [23]. Figure 5.8 displays 2” x 6” representative samples of the control, ASLT, and T-ASLT samples using a light table to compare level of transmittance through the package. Light, which can increase oxidation, is more apparent

in the T-ASLT samples when compared to the control and ASLT samples. Also, deterioration of the barrier layer of the bag structure was more prevalent in the T-ASLT samples than in the Control and ASLT samples evaluated during the study.

Figure 5.8. Light transmittance of bag samples (from L to R: Control, ASLT, T-ASLT)

Based on the results from this study, there were significant differences between the control and ASLT and T-ASLT variables, but no differences were detected between the ASLT and T-ASLT variable when comparing texture. Texture can be influenced by the style of potato chip. Kettle cooked potato chips differ from traditional style potato chips as the process used to manufacture the kettle cooked chips gives each chip a random and unique shape, unlike traditional chips [24]. Even though the samples were selected at random, Figure 5 showed the effects of moisture gain were not enough to provide statistical difference between the ASLT and T-ASLT variables ($P > 0.05$). Research has shown that texture analysis of potato chip products has a high amount of variability and often produces no useful qualitative information due to the irregular shapes, sizes and curvatures of the product [25 and 26].

Although no significant differences were observed between the two accelerated test variables when evaluating the texture, differences of significance were reported as a result of the moisture content analysis of these two variables. Increased moisture content was observed in the T-ASLT as compared to the control and ASLT variable, which were significant beginning on day 14 of the study ($P < 0.05$). Studies have shown that potato

chips reach unacceptable levels for texture once the moisture content reaches a level of 2.5% or greater [25 and 27]. Moisture contents recorded during this study period did not reach these levels, but it is clearly evident based on Figure 4 that the T-ASLT variable had a much greater moisture content and would lead to unacceptable levels at a more rapid rate than that of the control and ASLT variables.

4. Conclusion

This study showed that the transportation of food packages has a great affect on the performance of the package, which can ultimately affect the quality of the food product. This is of particular interest to personnel selecting materials and processes designed to extend or prolong the shelf life of a product. Because results showed differences of significance for the transportation variable, it should be reported that a transportation simulation be included or at least the hazards be understood prior to subjecting products to ASLT. This study has shown the rate at which some properties of the food and package can increase as a result of the simulated transportation. While each supply chain is different, the hazards that occur to the packaged products exist and do affect the performance of not only the package, but also the product.

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CHAPTER SIX

RESEARCH CONCLUSIONS AND RECOMMENDATIONS

Research Conclusions

Research Objective 1: Effect of Headspace Volume on Pouch Performance During Laboratory Simulated Hazards

This research study examined the effects of individual laboratory simulated transport hazards on retort pouches filled with different food simulants while varying the headspace volume. These pouches were then processed and stored in either standard or refrigerated conditions prior to being evaluated by laboratory simulated engineering tests.

It was determined sinusoidal vibration was not a critical distribution hazard to the retort pouch. Both simulants along with the varied storage conditions recorded no critical failures, and no statistical difference was determined for the headspace volume. Compression of the individual pouches yielded results showing statistical differences between the storage conditions, but no statistical differences between the headspace volume ($P<0.05$). This was a result of the CPP becoming brittle at the refrigerated conditions. The pouches with greater headspace volume could be deflected more before the pouch seal ruptured, but this was due to the headspace volume being able to be compressed. Shock testing produced results showing that headspace volume did have an effect on pouch performance, especially at the refrigerated storage conditions. For pouches stored at refrigerated conditions the greater the headspace volume the higher drop height the pouch could withstand. Analysis shows there was not a statistical

difference between the drop height and headspace volume for the 5% starch solution pouches, but there was for the water filled pouches ($P<0.05$). The shock test concluded headspace volume has a greater affect on pouch performance for lower viscosity food products than for more highly viscous products.

This research explored headspace volume, product viscosity, and storage temperature and how each affects a retort pouch during laboratory simulated hazards. Although retort pouches are shelf stable and do not require refrigeration, pouches could be exposed to extreme temperatures during transportation throughout the supply chain from manufacturer to consumer. The research shows increasing the headspace volume inside a retort pouch could increase the pouches ability to arrive safely to the consumer, especially in cold temperatures.

Research Objective 2: Analysis of Oxygen Levels in Gas Flushed Retort Pouches During Distribution and Storage

Examined were the effects of varying headspace volume inside a retort pouch to determine if the increased headspace volume provided any additional protection to the product during laboratory simulated transport performance tests designed to replicate small parcel and unit load shipments. The retort pouches were gas flushed with different volumes of nitrogen and thermally processed prior to the transport simulations. A non-destructive analysis technique using florescent decay was employed to measure oxygen ingress into the pouches over 63 day period.

Previous research studies have not provided a correlation between headspace volume and the potential to cushion and protect the contents of a package. This research

project utilized water as a food simulant due to its low viscosity. Using a low viscosity fluid like water it provided an extreme scenario for products typically thermally processed in retort pouches. The dynamic behavior of the fluid provided stress to the seal areas and because it was a flowable product allow for the pouches to fold onto themselves promoting flex and microcracks in the pouches.

Results from this study showed there was, on average, less percent oxygen present in the retort pouches containing 400cc of nitrogen than the pouches containing 200cc as a result of the small parcel simulation ($P<0.05$). This was as a result of the extra headspace volume protecting and cushioning the pouches during the simulation. The analysis performed on the pouches utilized for the unit load testing showed there was not a statistical difference in the percent oxygen present for the two different headspace volumes ($P<0.05$). Individual packaged products being distributed through small parcel supply chains are handled more vigorously than those being shipped palletized indicating the use of increased headspace volume may aid in protection of certain distribution channels.

Research Objective 3: Experimental Evaluation of the Effects of Transportation on Accelerated Shelf Life Testing of Packages Containing Potato Chips

This study showed that the transportation of food packages has a great affect on the performance of the package which can ultimately effect the quality of the food product. This is of particular interest to personnel selecting materials and processes designed to extend or prolong the shelf life of a product. Because results showed differences of significance for the transportation variable, it should be reported that a

transportation simulation be included or at least the hazards be understood prior to subjecting products to ASLT. This study has shown the rate at which some properties of the food and package can increase as a result of the simulated transportation. While each supply chain is different, the hazards that occur to the packaged products exist and do affect the performance of not only the package, but also the product.

Recommendations for Future Research

The following recommendations are made for further research involving the relationship between packaged products and the physical hazards occurring during transportation:

1. Continued research with various packaged products and determine how much headspace volume effects the performance of retort pouches containing flowable products like soups.
2. Increased awareness of transport hazards effecting packaged food products. Further data collection and analysis of the hazards (shock, compression, vibration, and environmental) to ensure the proper test levels are evaluated when performing and subjecting packages to these hazards.
3. Further research on specific packaged food products to determine what the effects of transportation hazards have on the predicted shelf life of certain goods.
4. Correlation of material property tests (i.e. gelbo flex testing, tensile testing, etc.) on transportation hazards. Is it possible to relate the effects of the material testing to hazards occurring during transportation of packaged products?