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THE EFFECT OF HOT FILL AND HOLD PROCESSING ON THE PERFORMANCE OF MULTILAYER PACKAGING FILMS

A Thesis Presented to the Graduate School of Clemson University

In Partial Fulfillment of the Requirements for the Degree Master of Science Food, Nutrition, and Culinary Sciences

> by Meredith Leigh Johnson May 2018

Accepted by: Dr. William S. Whiteside, Committee Chair Dr. Kyle D. Dunno Dr. Ronald L. Thomas

ABSTRACT

Heat transfer in thermal processing is crucial to ensure all parts of a product are sufficiently treated to achieve commercial sterility without unacceptable loss of quality. Optimizing pasteurization methods is recommended to preserve quality attributes such as color, texture, and flavor while maintaining food safety integrity. This research evaluated the temperature variability in pouches during a hot fill and hold process and the effect of those identified differences on color quality of a tomato based food simulant. The performance of multilayer films for pasteurized products in accelerated storage conditions were also studied.

The research project was separated into two phases. The objective of the first phase was to understand the profiles of heating and cooling in pouches processed in a simulated hot fill and hold process. The corners of the pouch were found to be the fastest cooling spot within the pouch (p<0.05). The center of the pouch was found to have the highest mean temperature during the hold step of the process and had the slowest cooling a low viscosity food simulant. This study compared the time and temperature profiles for a static hot fill process versus a process that incorporated rotating the pouch 180° every 10 seconds. For the static hot fill and hold process, mean temperatures of the center and corners of a pouch showed non-uniform heat transfer during the holding period and cooling process. More uniform heating and cooling within pouches was achieved by implementing 180° rotation during processing.

In the second phase, the time and temperature combinations representative of the hold step in a hot fill process were determined from the results obtained from the first phase of the research project. Processing treatments were selected for the corners, the fastest cooling location and the center, the slowest cooling location for both static and process conditions. Three different types of multilayer films were evaluated using a tomato based food simulant. Color was measured as a quality parameter to predict the effects of time-temperature combinations and the performance of the barrier properties in different films. Results showed that barrier properties had a significant effect (p<0.05) on color retention. In the case of low barrier films, the process with the highest temperature and longest retention time had the greatest loss of color (p<0.05).

This research evaluated the heating trends within a pouch during hot filling and lack of uniformity during processing. Food manufacturers producing acidic or acidified products could use this information to improve quality and safety in hot filled products. These findings also highlight the importance of selecting suitable packaging films for pasteurization processes.

DEDICATION

I dedicate this thesis to my loving family. My parents, Lori and Keith Johnson, have provided continuous love and support throughout this journey and I could not have completed this task without them.

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TABLE OF CONTENTS

		-
TITLE PA	AGE	i
ABSTRA	СТ	ii
DEDICAT	ΓΙΟΝ	iv
ACKNOV	VLEDGMENTS	v
LIST OF	TABLES	viii
LIST OF	FIGURES	x
CHAPTE	R	
I.	INTRODUCTION	1
	References	4
II.	LITERATURE REVIEW	5
	Shelf Stable Foods Thermal Processing Sterilization Pasteurization Food Packaging Shelf Life Evaluation Tomato Products and Color Literature Cited	5 6 10 11 16 21 23 26
III.	IDENTIFYING TEMPERATURE VARIABILITY IN POUCHE SIMULATED HOT FILL AND HOLD PROCESSING Abstract Introduction	2S DURING

IV.	THE EFFECTS OF THERMAL PASTEURIZATION AND MUL	TILAYER
	PACKAGING FILMS ON THE COLOR OF A FOOD SIMUL	LANT53
	Abstract	53
	Introduction	54
	Materials and Methods	
	Results and Discussion	62
	Conclusion	95
	References	96
V.	RESEARCH CONCLUSIONS	
APPEN	IDICES	100
A:	Temperature Profiles	
B:	Raw Data from Research Experiment	105
	-	

LIST OF TABLES

Table	Page
3.1	Study 1, SAS output for mean temperature (°C) recorded at time zero (seconds) organized from highest to lowest temperature
3.2	Study 1, SAS output for mean temperature (°C) at time 2 min. organized from highest to lowest temperature
3.3	Study 1, SAS output for k (cooling rate) from fastest to slowest rate46
3.4	Study 2, mean temperatures (°C) for Process 149
3.5	Study 2, mean temperatures (°C) for Process 249
3.6	Mean temperature comparison from static and rotational processes
4.1	Time and temperature combinations for each process
4.2	L* values over 8 weeks exposure at 35±2°C and 50±2% RH67
4.3	a* values over 8 weeks exposure at 35±2°C and 50±2% RH70
4.4	b* values over 8 weeks exposure at 35±2°C and 50±2% RH72
4.5	Tomato Paste Scores over 8 weeks exposure at $35\pm2^{\circ}C$ and $50\pm2\%$ RH 75
4.6	Tomato a/b Ratio over 8 weeks exposure at 35±2°C and 50±2% RH78
4.7	Rates of change for food simulant over 8 weeks exposure at 35±2°C and 50±2% RH
4.8	L* values over 8 weeks exposure at $35\pm2^{\circ}C$ and $50\pm2\%$ RH
4.9	a* values over 8 weeks exposure at 35±2°C and 50±2% RH87
4.10	b* values over 8 weeks exposure at 35±2°C and 50±2% RH
4.11	Tomato Paste Scores over 8 weeks exposure at $35\pm2^{\circ}C$ and $50\pm2\%$ RH 91
4.12	Tomato a/b Ratio over 8 weeks exposure at 35±2°C and 50±2% RH93

List of Tables (Continued)

Table		Page
4.13	Rates of change for food simulant over 8 weeks exposure at 35±2°C and 50+2% RH	94

LIST OF FIGURES

Figure	Page
3.1	Locations of thermocouples placed into pouch structure for Study 1
3.2	Thermocouple location 5 position prior to filling
3.3	Thermocouple locations for Study 2
3.4	Time temperature data for Study 142
3.5	Temperature profile of pouch at time 0
3.6	Temperature profile of pouch at 2 minutes during the holding period44
3.7	Time-temperature data for tomato food simulant process 1, Study 248
3.8	Time-temperature data for tomato food simulant process 2, Study 2
4.1	Packaged food simulant
4.2	In-container pasteurization method using a water bath60
4.3	Thermocouple inserted into the geometric center of a pouch
4.4	Changes in L* values for all films for P-164
4.5	Changes in L* values for all films for P-265
4.6	Changes in L* values for all films for P-365
4.7	Changes in L* values for all films for P-466
4.8	Changes in a* values for all films for P-168
4.9	Changes in a* values for all films for P-268
4.10	Changes in a* values for all films for P-369
4.11	Changes in a* values for all films for P-469
4.12	Changes in b* values for all films for P-170

List of Figures (Continued)

Figure	Page
4.13	Changes in b* values for all films for P-271
4.14	Changes in b* values for all films for P-371
4.15	Changes in b* values for all films for P-472
4.16	Changes in TPS for all films for P-172
4.17	Changes in TPS for all films for P-273
4.18	Changes in TPS for all films for P-374
4.19	Changes in TPS for all films for P-474
4.20	Changes in tomato a/b for all films for P-176
4.21	Changes in tomato a/b for all films for P-276
4.22	Changes in tomato a/b for all films for P-377
4.23	Changes in tomato a/b for all films for P-477
4.24	Changes in L*values for all process conditions for Film A83
4.25	Changes in L*values for all process conditions for Film B
4.26	Changes in L*values for all process conditions for Film C
4.27	Changes in a*values for all process conditions for Film A
4.28	Changes in a*values for all process conditions for Film B
4.29	Changes in a*values for all process conditions for Film C
4.30	Changes in b*values for all process conditions for Film A
4.31	Changes in b*values for all process conditions for Film B
4.32	Changes in b*values for all process conditions for Film C

List of Figures (Continued)

Figure		Page
4.33	Changes in TPS for all process conditions for Film A	89
4.34	Changes in TPS for all process conditions for Film B	90
4.35	Changes in TPS for all process conditions for Film C	90
4.36	Changes in tomato a/b ratio for all process conditions for Film A	91
4.37	Changes in tomato a/b ratio for all process conditions for Film B	92
4.38	Changes in tomato a/b ratio for all process conditions for Film C	92

CHAPTER ONE

INTRODUCTION

In the past few years, the demand for high quality processed products with fresh like characteristics has increased. Consumer preferences have shifted towards fresh and healthy foods with their natural nutritive values and sensory attributes such as color, flavor, odor, texture, and taste. Developing products to support this increase in demand can be a difficult task, while also maintaining the food safety and desired shelf life (Siddiqui & Rahman, 2014). Thermal processing is a widely used technique for preserving and extending the shelf life of food products. Thermal processing can range from moderate to more severe applications, the intensity of the thermal process affects food safety and quality, respectively. This processing method can be categorized in two main groups based on their intensity: pasteurization and sterilization (Fellows, 2009). Pasteurization involves a mild heat treatment in which foods are heated to below 100°C (212°F). For acidic foods having a pH equal or lower than 4.6, pasteurization is commonly used to destroy spoilage microorganisms and inactivate enzymes to extend shelf life of a product with an upward of several weeks (Smith, 2003). Thermally pasteurized foods retain higher sensory and nutritional quality compared to sterilized foods.

For every product, it is necessary to apply a suitable process of a given treatment time at a specified temperature, to ensure that products do not pose a public health problem. Therefore, the goal of thermal processing of foods is to determine the suitable time-temperature combinations and cooling methods that ensure those specifications

(Richardson, 2004). There is a growing interest in food processing and preservation methods that do not use heat or reduce the heat input of conventional technologies by reducing the treatment time and/or temperature (Verlent, Hendrickx, Rovere, Moldenaers, & Van Loey, 2006). A primary focus in processing of liquid and semisolid foodstuffs has been meeting the consumer demand for high-quality, shelf-stable, and safe food products (Motarjemi, Moy, & Todd, 2014).

In addition to process optimization, designing a suitable packaging material is essential for any product to achieve a desired shelf life. In the U.S., the flexible packaging industry reached \$30.2 billion of sales in 2016 (Flexible Packaging Association, 2017). Flexible materials can pack many a myriad of products. The choice of materials used are determined by the intrinsic needs and characteristics needed to guarantee the preservation of the food product. Food is complex in nature and can deteriorate when exposed to water vapor and gases, especially oxygen. Because monolayer materials do not possess all the properties needed to provide a sufficient barrier for these interactions, it is necessary to combine different materials to achieve that goal (Ramalingam, VA, George, & SN, 2015). Barrier films are usually multilayer flexible packaging material used for food application and have been designed to be impervious to gas migration. These multilayer materials can be produced by thermal lamination, coating, or coextrusion technologies. With that being said, packaging materials can be quite complex by incorporating one or more layers of the same or different types of polymers to provide several desirable properties such as optimal transparency, mechanical performance (tensile and impact strength), optical properties,

and thermal and dimensional stability. The main challenge in the food packaging industry is to design multilayer films to accommodate the desired properties without a significant cost increase (Ebnesajjad, 2013).

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CHAPTER TWO LITERATURE REVIEW

Shelf Stable Foods

The deterioration of foods cannot completely be prevented but the rate of degradation can be lowered by using appropriate techniques of processing, formulations, packaging, and storage conditions. Any deliberate change in a food before it is available to consumers that alters it from its natural state can be considered a processed food (Poti, Mendez, Ng, & Popkin, 2015). Processed foods can be categorized by the extent of applied processing to foods: frozen, refrigerated, and shelf stable.

In Title 21 Code of Federal Regulations (CFR) Part 108, Federal Regulations require commercial processors of shelf stable foods to register each establishment and file scheduled processes with the Food and Drug Administration (FDA) for each product, product style, container size and type, and processing methods (FDA, 2017). Shelf stable foods are further classified by water activity (a_w) and pH. These factors are used to determine the appropriate process for a shelf stable food. Water activity, also known as the relative vapor pressure (RVP), is the availability of water for microbial, enzymatic, or chemical activity. Water activity is an important intrinsic factor in food processing because most foods consisting of a water activity of 0.95 or higher support the growth of microorganisms, molds, and yeasts. Food can be made safe by lowering the water activity to a point that will not allow the growth of dangerous pathogens. The use of humectants to chemically bind product moisture is a common method used to lower water activity. Heat can also be applied to reduce the water activity enough to where microorganisms are unable to grow (Clark, Jung, Lamsal, & Amponsah Annor, 2014). If water activity is controlled to 0.85 or less in the finished product, it is not subject to the regulations of 21 CFR Parts 108, 113, and 114. Finished shelf stable products with a water activity higher than 0.85 are further categorized by their pH levels.

The amount of acid present in food is measured using the pH scale ranging from zero to 14. It is uncommon for foods to be in the alkaline range (pH>7). Acidic foods (pH<7) are subcategorized into low acid and high acid. This factor determines the most heat resistant enzyme, pathogen, or spoilage microorganism (Tewari & Juneja, 2007; 2008). The FDA defines any food with a finished equilibrium pH greater than 4.6 and a water activity greater than 0.85 as a low-acid canned food (LACF), excluding tomatoes and tomato products having a finished equilibrium pH less than 4.7. This category of shelf stable foods is also referred to as Low Acid Foods (LAF) due the growth of flexible packaging materials. These foods must achieve commercial sterility through thermal processing. Acidified or naturally acidic products are those with a pH less than 4.6. High acidity foods (pH<4.6) are significant in food processing since *Clostridium botulinum* is unable grow in this environment. In acidic foods, (pH< 4.6) enzyme inactivation is the main goal for processing to preserve food products and prevent spoilage. Other microorganisms (yeasts and fungi) and heat resistant enzymes are the major causes of food spoilage and are used to establish processing conditions (Smith, 2003).

Thermal Processing

Thermal Processing of Food Products

Thermal inactivation is the most widely used process for food preservation. The application of heat treatment on food products, their package, and surrounding

environment is one of the most important methods used in food processing. This preservation method creates desirable effects on eating quality and a preservative effect on foods by eliminating enzymes, microorganisms, and parasites. Major advantages of thermal processing include: ease of controlled processing conditions, capacity to produce shelf stable products, ability to destroy anti-nutritional factors, and enhancement of the availability of some nutrients. Heat treatment can also produce undesirable effects to the overall quality of a product by changing flavor, color, taste or texture which may present a product of lower quality and values (Fellows, 2009).

Thermal processing can range from moderate to more severe applications. The intensity of the thermal process affects food safety and quality respectively. Blanching and pasteurization are examples of mild heat processing, and in the case of canning operations the thermal process is more severe. The effectiveness and validity of every thermal process depends on a food product's composition and formulation, heat resistance of the microorganisms present, the characteristics of the container, and the product's rate of heating. The physical properties of a food influence heat transfer mode and heat penetration speed (Lewis, 2000).

General Terms Related to Thermal Processing

The decimal reduction time (*D* value) is the time needed to destroy 90% of microorganisms at a specific temperature (reduce their numbers by a factor of 10 or by one log cycle). The destruction of microorganisms by means of thermal processing have been understood to decrease logarithmically at high temperatures with a function of time. This theory means that a completely sterile product cannot be produced regardless of how

long thermal processing is applied. The number of microorganisms present can never be zero because there is a small chance of survival. The reduction of microorganisms needs to be reduced to an acceptable level contingent on the targeted organism for the process. The concept of "commercial sterility" is defined as a treatment process that reduces cell numbers by 12 decimal reductions, a 12D process. It can also be defined as the chance of survival of 1 spore of *C. botulinum* in 10^{12} containers. For thermally processed foods, a twelve-log cycle is needed to establish a safety margin. In theory, heat processing inactivates vegetative spores and cells that would be capable of multiplying under storage conditions (Fellows, 2009).

The D value for each microbial species differ. A species with a greater heat resistance will have a higher D value. The z value is the increase in temperature required for the D value to bring a ten-fold change or one log cycle. The D value and z value are used to determine the heat resistance and temperature dependence of a microorganism. The time and temperature combination is product specific to establish an adequate process for each batch.

The thermal death time (TDT) or F-value is important in calculating process times. It is the time needed to establish a specified reduction in microbial numbers or assure that there are no survivors at a given temperature. A TDT curve is constructed by combining D values at different temperatures. The temperature is plotted on the x-axis and the heating time on the y-axis. The z-value is defined by the slope of the TDT curve and the number of degrees Celsius needed to bring a ten-fold change in decimal reduction time (Clark et al., 2014).

The most dangerous pathogen likely to be present in low acid foods (pH > 4.6) is the heat-resistant, spore forming microorganism, *C. botulinum*. This pathogen can grow in anaerobic conditions inside a sealed container and produce a deadly neurotoxin which is 65% fatal to humans (Fellows, 2009). The minimum requirement in sterilized foods is to target and destroy *C. botulinum*. Spoilage bacteria and heat resistant enzymes may be present in foods and are used to establish processing conditions receiving more than the minimum treatment. However, in acidic foods (pH 4.5-3.7), the germination of *C. botulinum* is inhibited and the target for processing is dependent on spoilage organisms and heat resistant enzymes (Holdsworth & Simpson, 2008).

Heat Penetration Tests

The rate of heat penetration and cooling of a food product is used to calculate a safe thermal process for a specific product. These rates can be influenced by the shape of a given container, the headspace of a container, and product viscosity. A low rate of heat penetration to the thermal center can be an issue with processing of solid or viscous foods (Erdogdu & Tutar, 2012). The validation of a thermal processes uses a temperature recording system consisting of two parts: a temperature sensor and data logging component. A temperature sensor or thermocouple is inserted inside a container at the point of slowest heating to record temperatures during processing (Richardson, 2004). It is assumed that all other locations within a container receive more heating than the slowest heating point. The temperature measurement of this critical point determines if the thermal process has adequately heated all food particles to the proper temperature. Thermocouples are linked to a data logger that records temperature and time readings at

various intervals. Thermal processing software can evaluate data collected from thermal processes and be used to create processing parameters for a specific food product (Fellows, 2009).

Sterilization

Sterilization is a heat treatment in which foods are heated at a combination of adequately high temperatures and time used to primarily inactivate and destroy vegetative microbial cells, spores, and enzymes. This heat treatment is used to reduce public health hazards of pathogenic microorganisms and extend shelf life in low acid foods, consisting of a pH greater than 4.6 (Richardson, 2001). The application of sterilization produces products with a shelf life of six months and beyond. These foods require little heating before consumption due to pre-cooking that is acquired from sterilization which attributes to their characteristic of being a convenient product. Sterilization can be administered by two methods: (1) in-container processing, the food is sealed into a container and thermally processed; (2) within a sterile environment, food is sterilized and filled into pre-sterilized packaging and sealed (Nelson, Chambers, & Rodriguez, 1987). These methods of heat sterilization are also referred to as retorting and ultra-high temperature (UHT) aseptic processing.

In-container sterilization uses severe heat treatment that can alter the sensory and nutritional qualities of the product. The damage to these characteristics of food can be minimized by reducing processing time or processing foods before packaging. There are many ways processing times can be reduced or altered including changing the geometry

and shape of the container, utilizing flexible packaging materials such as pouches or trays, and acidification of products (Fellows, 2009).

Aseptic processing is another technique used to process low-acid foods. This process is defined by the filling of a sterilized product into a pre-sterilized container in an aseptic environment followed by the application of a hermetic seal to prevent recontamination. Hermetic sealing prevents any form of mass transport between the packaged product and the environment such as microorganisms, water vapor, and other gases. Unlike in-container sterilization, the product and package are sterilized independently (David, Graves, & Szemplenski, 2013).

Pasteurization

Pasteurization is a food preservation technique based on partial thermal degradation of microorganisms and denaturation of enzymes. Pasteurization is applied to a wide range of foods such as dairy products, fruit, alcoholic beverages, sauces, etc. to prolong shelf life from days to months. The thermal process of pasteurization is defined as a mild heat treatment in which foods are heated to below 85°C (185°F) or higher (Smith, 2003). Generally, pasteurization operations for liquids are carried out in continuous heat exchangers (shell and tube or plate and frame). In the first heat exchanger, the product temperature is quickly raised to appropriate levels, held for a required length of time in the holding tubes, and quickly cooled in a second heat exchanger (Ramaswamy & Marcotte, 2006). In comparison with other heat treatments, pasteurization results in minimal affects to the sensory characteristics and nutritional value of a food.

Unlike sterilization, pasteurization does not aim to destroy all microorganisms present in food but reduce the number of viable pathogens so they are unlikely to cause disease. For acidic foods having a pH equal or lower than 4.6, pasteurization is commonly used to destroy harmful organisms such as bacteria, enzymes, molds, and yeast to create a shelf stable product that can be stored at ambient temperatures for several months (Castberg, Osmundsen, & Solberg, 1995). The primary goal of pasteurization is to increase the shelf life of product, providing necessary preservation while minimizing nutritional loss by preventing thermal degradation. There are various techniques to extend the shelf life of fruit juice and puree products such as aseptic technology, electrical conductivity, heat, and pressure. These conditions are determined on the nature of the product, pH, and the resistance of the test microorganism or enzyme, and type of heat application (Plazl, Lakner, & Koloini, 2006). There are many advantages and disadvantages for different methods used for preserving foods. Common thermal techniques to achieve pasteurization include in-container methods and hot-fill and hold processes (Gonzalez-Mulet, 2008).

In-container

The concept of in-container pasteurization involves the application of the thermal process after the food product is sealed in a container. In-container pasteurization can employ equipment like steam retorts, pressure steam tunnels, hot water baths, and hot water sprays. The thermal process can be applied continuously where a procession of food products in containers are moved along conveyors through pasteurization tunnels with sections of heating, holding and cooling (Bown, 2010).

Hot-fill and hold

In a hot-fill process, the food is pasteurized prior to filling and it is assumed that the temperature given to the food will also pasteurize the container. The fluid food is heated in either a batch or continuously in a heat exchanger before filling into containers and immediately sealed or capped before the product is cooled. A hot fill process will require the short hold time at high temperature so the surfaces of the inside container are pasteurized (Rahman, 2011). The temperatures required for this process are 85°C (185°F) or higher and a hot-holding period may be required before the container is cooled. The temperatures of a hot fill process must be high enough to reduce the microbial concentration to safe levels and inactivate enzymes that could cause browning and fermentation. This process is sufficient to heat treat viable vegetative forms of microorganisms but not heat-resistant spores. Hot filling is suitable for non-carbonated beverages and juices (Ashurst, 2005).

The material used to package hot filled products must ensure integrity when exposed to high temperatures and contain a high barrier to protect against gas penetration and sorption. The packaging material must also provide a good barrier to prevent post processing contamination. The application of this process is limited to materials that are heat sensitive. Some advantages of hot-filling pasteurization are its simplicity as the product and container are pasteurized together and aseptic filling is not necessary, equipment is relatively inexpensive, and there are reduced quality control issues (Adegoke, 2004).

Hot Filling Processing Procedure

The food product is cooked thoroughly and then filled into a container during hot filling processes. Products are heated to temperatures above 85°C (185°F) and filled into a given container to ensure a safe product. Once the container is filled and sealed it can be inverted and held in air for three minutes before being cooled by water. Inverting of the package is done to pasteurize the headspace and lid, this method has been used in industry for filling into glass jars, plastic trays, cartons, and pouches. The product is cooled after the holding period to preserve the quality and avoid excessive heat exposure to the product. If spoilage occurs within the product, it is likely due to insufficient heating of the package surfaces or package failure that can cause post process contamination (Bown, 2010).

Pasteurization and hot filling processes can be optimized in a way that achieves the goal of food safety and minimizes the quality changes. If a thermal process is applied longer than necessary to pasteurize or sterilize a product it can contribute to losses in product quality. A study by Silva et. al. (2003) described the design and optimization of hot filling pasteurization conditions. To optimize a thermal process the thermal kinetics of microbial target, relevant quality factors, and process heat transfer into the product must be modeled. Process optimization can also take non-quality factors into consideration such as productivity and energy consumption. This idea of minimizing qualities losses during thermal processing of foods is not new, and there are several studies available in literature for quality optimization in the field of sterilization of prepackaged food. There are few studies found in literature regarding pasteurization quality optimization studies for fruit processing (Silva, Martins, & Silva, 2003).

However, there are studies that have evaluated and developed mathematical models to predict time and temperature relationships for hot filling. An article by Sandoval et. al. (1994) developed a mathematical model to predict the heat transfer and integrated sterilization values of double concentrated tomato paste in glass jars. Various jar sizes were used in a simulated hot fill hold and air cooling process. Three hot filling temperatures of 85, 90, and 95°C were applied and the temperature was stabilized in a water bath. The jars were removed after the retention time and allowed to cool in stagnant air. As the glass jar size increased, the hot filling temperature and required retention time to achieve the integrated sterilization value decreased. A lower hot filling temperature required a longer retention time. A time and temperature combination of low temperature, long time can induce undesirable changes in the quality attributes and nutritive value of the product. It is recommended to determine an adequate thermal process that achieves food safety and provides the best quality of the product (Sandoval, Barreiro, & Mendoza, 1994).

In a study conducted by Skinner et. al. (2005), the hot fill process of juices in bottles was examined for its ability to a potential adulterant, *Clostridium botulinum* neurotoxin (BoNT). The temperature profiles of commercial sports drink bottles were established to find the location the received the lowest heat treatment after the hot fill and the fastest cooling spot. Two bottles (12 and 20 oz.) sizes with two orientations of inverted and upright were examined during a hot fill process of orange and apple juice evaluating filling temperatures of 80 and 85°C. Sixteen type T thermocouples were positioned within the bottle to monitor temperature and 20 minutes was allowed for data

acquisition. The fastest cooling point in upright bottle orientation was the bottom outer rim of the bottle. In the inverted bottles, the region in the cap of the bottle was the location of the fastest cooling point. These findings were in align with the understanding that the cap region had a smaller diameter, thus, a smaller amount of fluid that cools more easily than the fluid in larger diameters of the bottle. The upright bottle had a faster cooling rate than the inverted bottle. Overall the results demonstrated that filling temperature is a critical factor and that 85°C should be used in a filling operation to ensure adequate inactivation (Skinner et al., 2015).

Food Packaging

The primary function of a packaging material is the quality preservation of the packed product. Among those products, food is especially important due to their principal chemical instability. Packaging serves an important role in protecting food from physical damage, physiochemical deterioration, microbial spoilage, and product tampering. Interactions between a packaging material and the food contained within the packaging are undesirable and can cause toxicological effects on the consumer or it may reduce the shelf life or sensory qualities of the food (Piringer & Baner, 2000; Fellows, 2009). Packaging prevents the migrations of compounds from the material to the products and prevents absorption of flavor compounds, preserving sensory properties.

The protection of a food product is an essential part of the preservation process. The main purpose of a package is to protect its contents from the outside environment and the effects of water, water vapor, gases odors, microorganisms, dust, shocks, vibrations, and compressive forces. A primary package is in direct contact with the

contained product and is usually the initial protective barrier. In retail stores, consumers generally are making product purchases with only the primary packaging material. Examples include metal cans, glass bottles, paperboard cartons, and plastic pouches. It is important that the packaging in direct contact with the food product consists of a material that provides a suitable barrier to ensure that microorganisms cannot contaminate the product making it unsafe. Once the integrity of a package is breached, the product can no longer be preserved or be fit for consumption (Robertson, 2009). The methods of how a product is packaged and packaging material play an important role in contributing to a product's shelf life and can greatly influence the environment surrounding a food. Shelf studies are conducted to select an adequate packaging material for food products.

Metal Cans

Metal cans are the oldest packaging used for food preservation and a representation of the modern Industrial Age. The metal can is a hermetically sealed container, it is designed to be secure against the entry of microorganisms and to maintain commercial sterility of the product after processing (Featherstone, 2015). Metal cans withstand high temperature processing and are impermeable to light, moisture, odors, and microorganisms. Tinplate, electrolytic chromium coated steel (ECCS) also referred to as tin-free steel, and aluminum are metals used for canning (Fellows, 2009). The performance of the metals used for canning have been continuously been improved by implementing better lacquers and improvements in materials science.

Glass

Glass is one of the oldest materials used for containers. Glass is an inorganic product of fusion that has been cooled to a rigid condition without crystallization. Glass provides a barrier to moisture, gasses, and odor without reacting or migrating into food products. The primary types of glass containers used for food packaging are bottles jars (Robertson, 2009). Glass is preheated prior to thermal processing due to its lower resistance to fracturing and thermal shock than other materials. Glass weighs more than other packaging materials and is prone to breakage which has a potential hazard of glass fragments in food products (Featherstone, 2015).

Paper and Paperboard

Most paper is converted by further treatment after being manufactured including embossing, coating, laminating, which can be formed into different shapes and sizes. Paper that has been laminated or coated with plastic polymers provides a good barrier to gases and water vapor. Paper packaging also protects product from exposure to light. This material is used to form rigid boxes, folding cartons, beverage cartons, and similar products (Kilcast & Subramaniam, 2011).

Plastics

Plastics used for food packaging are categorized by rigid, semi-rigid, or flexible plastic packaging. The U.S. Food and Drug Administration recognizes plastics as "indirect food additives" and must meet the requirements in Title 21 of the Code of Federal regulations (CFR) for application to food packaging. The U.S. flexible packaging industry is the second largest packaging segment behind corrugated paper and had an estimated \$30.2 billion in sales in 2016. This industry has steadily grown over the past 10

years with a compound annual growth rate (CAGR) of 1.9 percent (Flexible Packaging Association, 2017). Flexible packaging has been leading the way in packaging innovation. This area of packaging is increasing in popularity and trends of product protection, design, performance, sustainability, and consumer convenience.

Flexible packaging includes any package made of paper, plastic, film, foil, metalized, or coated papers and film. Multiple layer films have been developed by using one or more layers of the same polymer or different polymers blended or coextruded together. The layers of different material provide the film properties such as high barrier, mechanical strength, antimicrobial, or heat sealing ability. Multilayer packaging films are developed to provide barrier to gases such as oxygen, nitrogen, carbon dioxide, and water vapor (Gherardi, Becerril, Nerin, & Bosetti, 2016). A disadvantage is that multilayer materials are not recyclable, however, flexible materials can reduce the volume and weight of waste compared to rigid packaging. To develop successful flexible packaging a good understanding of its target application is critical to ensure the protection and preservation of a food product. The conditions the package will undergo during filling, processing, distribution, and storage is used to determine the permeation needs of the multilayer material. An inadequate understanding of the product barrier requirements can pose issues for predicting the shelf life of a product. Extensive shelf life testing of individual food products is needed to evaluate barrier film performance (Ebnesajjad, 2013).

Barrier Properties

The mechanism of permeation occurs in polymers films allowing interaction with the environment. If water vapor and atmospheric gases permeate in or out of a package the taste, color, and nutritional content of the product can be altered. Films are used to provide barrier to oxygen, nitrogen, carbon dioxide, and water vapor. Most films used for food packaging are composed of a multilayer structure with barrier materials of ethylenevinyl alcohol (EVOH), high density polyethylene (HDPE), nylon, or oriented polypropylene. High performance films are commonly used in food packaging for fresh produce, meat, dairy, liquids, dry goods, and frozen foods. These films can be produced in various ways such as blow and cast film, and coextrusion (McKeen, 2012). *Polyethylene*

Polyethylene (PE) is the most widely used mass produced plastic. PE is a thermoplastic material that softens at room temperature (80-130°C) and has good chemical stability. It is easily heat sealed and has high elasticity. There are many different types of PE and they are classified by their density. Low density polyethylene (LDPE), and linear low-density polyethylene (LLDPE), and high-density polyethylene (HDPE). LDPE is a tough, flexible, and slightly translucent material that provides a good barrier to water vapor but poor barrier to gases. It is typically used in food packaging and is easily heat sealed to itself. HDPE has more of a linear structure than LDPE making it more hard and stiff. It is used in both film form and rigid packs (Piringer & Baner, 2000).

Nylon

High-molecular weight polyamides or more commonly known as nylon is often used in multilayer structures to prove strength and toughness in food packaging systems.

Generally, nylon 6 and nylon 66 are used for packaging materials. Both polymers are known for hardness and are semi crystalline. These polymers have good puncture resistance, impact strength, temperature resistance, and gas barrier properties. Nylon films can be thermoformed and biaxially stretched (Han, 2014).

EVOH

Ethylene-vinyl alcohol (EVOH) is a copolymer of ethylene and vinyl alcohol. The material is highly crystalline and produced with different contents of ethylene. This barrier resin is used in multilayer food packages for its effective barrier properties against oxygen, odors, and gases. Due to its sensitivity to wet conditions it is always used in multilayered structure systems (Han, 2014). The desirable qualities of EVOH film include: antistatic properties, printability, resistance to oil and organic solvents, luster and transparency, and permeability (Ebnesajjad, 2013).

Shelf Life Evaluation

The shelf life of a food is the period during which a food remains safe and acceptable for consumption. The goal of a shelf-life study is to find out the point in time at which a product has become unsafe or unacceptable under specified storage conditions to the target consumer. The characteristics of food will inevitably change during storage. These changes in food are categorized by microbiological or non-microbiological (biochemical, chemical, physical, temperature-related) deterioration (Kilcast & Subramaniam, 2011). The shelf life of packaged food is controlled by the structure and composition of the product, the environment outside of the package, and the interaction of the product and packaging leading to chemical and physical reactions. Color and flavor

changes can occur from these reactions and could deem a product unsafe, undesirable, or unacceptable for consumption. The issue of food spoilage contributes to the large amount of food that is wasted and the associated financial losses (Lianou, Panagou, & Nychas, 2016).

A common and direct method to determine shelf life is to carry out experimental storage trials under the conditions that simulate those the product is likely to be exposed to during storage, distribution, retail display, and consumer use. The application of accelerated storage trials is one method of storage that can be used to shorten the time to predict shelf life for products that would otherwise take a long time to determine under normal conditions. Accelerated shelf life testing applies the principles of chemical kinetics to determine the rate of deterioration by subjecting products to extrinsic factors such as temperature, humidity, gas atmosphere, and light. The rates of deterioration are accelerated resulting in a shorter time to product failure when one or more extrinsic factors factors are maintained at a higher than normal level (Man, 2016; Robertson, 2009).

There are many parameters that can be assessed to determine the changes within a food during storage. Instrumental techniques are widely used to examine the changes in quality attributes of foods during extended shelf life. The overall appearance of a food gives consumers their first impression of a product which can influence their purchasing decision. Color is an important attribute associated with the freshness of a product and is frequently used as a quality control measure of a food during processing and storage. The food color typically degrades during storage because of enzymatic and non-enzymatic

reactions, oxidation, and other various physical and chemical reactions (Kilcast & Subramaniam, 2011).

Tomato Products and Color

Tomatoes are one of the most important horticultural crops worldwide. Tomatoes are characterized by their taste, color, and flavor. They provide a rich source of dietary antioxidants including carotenoids, vitamins, and phenolic compounds. Tomatoes are used as fresh fruits or in processed forms such as diced products, paste, whole peeled tomatoes, and various forms of juices and soups (Grandillo, Zamir, & Tanksley, 1999). Two conventional thermal methods, "cold break" and "hot break", are applied in industrial production of tomato based products to extract juice from tomatoes. High processing temperature, prolonged processing time, and light or oxygen exposure may degrade tomato pigment and decrease nutritional value (Verlent, Hendrickx, Rovere, Moldenaers, & Van Loey, 2006).

Color is used as an indicator of quality and freshness for tomato products with the perception of "the redder the better". The carotenoid, lycopene is responsible for the vibrant red color of tomatoes. Lycopene comprises 90% of the pigment responsible for the red color in mature red tomatoes. Beta-carotene also contributes to the color profile in immature and orange pigmented tomatoes. Tomatoes consisting of a deep red color, compared to those that have a lighter color red or pink, are typically more mature having a more desirable flavor and higher content of lycopene. The USDA Processed Products Standards and Quality Certification program has developed color standards representing
minimum color grades "A" and "C" in tomato catsup, juice, paste, and puree. Canned tomato color is judged with a grade "C" or better (Barrett & Anthon, 2008).

Prior to advanced technology, the color acceptability of tomatoes was measured subjectively by sensory evaluation. The development of instruments for color measurement has allowed for the quantification and classification of tomato color. Many instrumental methods exist for determining tomato color, such as employing filters or light-emitting devices or more complex tri-stimulus colorimeters and spectrophotometers. One of the simpler techniques is the L*a*b* color system. This system defines color as lightness (luminance) ranging from black to white (L* axis), greenness to redness (a* axis), and blue to yellowness (b* axis) (Zambre, Venkatesh, & Shah, 2010).

A study by the University of California at Davis and the USDA was conducted to correlate visual scoring or tomato product quality to instrumental color measurements. The color measuring capabilities of five different color measuring devices (ColorFlex, LabScan XE and D25 from HunterLab; and Color Guide and Color View from BYK Gardner) were compared. The study determined the equations associated with each instrument for the calculation of color for products covered by the U.S. Standards for Grades of Canned Tomato Sauce, Tomato Juice, Tomato Paste, and Tomato Catsup (Barrett & Anthon, 2008; USDA Fruit and Vegetable Program Specialty Crops Inspection (SCI) Division,).

The color of tomato fruits is evaluated prior to processing to ensure quality in finished products and can also be used as an indicator of quality in shelf life studies (Aguilo-Aguayo, Charles, Renard, Page, & Carlin, 2013). The main factors affecting the

shelf life of fruits include: temperature, maturity stage, atmosphere, genetic background, and length of storage (Baltazar, Aranda, & González-Aguilar, 2008).

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CHAPTER THREE

IDENTIFYING TEMPERATURE VARIABLILTY IN POUCHES DURING SIMULATED HOT FILL AND HOLD PROCESSING

ABSTRACT

Two studies were performed to understand the profiles of heating and cooling in pouches processed in a simulated hot fill operation. The first study focused on identifying the fastest cooling spot within a pouch. Eight thermocouples were placed into various areas of a pouch to indicate the location of the highest and lowest average temperature during hot fill, hold, and cooling steps. There was a significant difference (p<0.05) found between mean temperatures of the center and corner locations using a low viscosity food simulant. The center of the pouch was found to have the highest mean temperature (p<0.05) during holding and thus the slowest rate of cooling. The corners of the pouches had the lowest mean temperature during the holding step (p<0.05) and thus the fastest rate of cooling. The second study used the findings from the first study to evaluate the trends of heating and cooling using a low viscosity food simulant. This study compared the time and temperature profiles for a static hot fill process versus a process that incorporated rotating the pouch 180° every 10 seconds. For the static hot fill and hold process, mean temperatures of the center and corners of a pouch showed non-uniform heat transfer during the holding period and cooling process. More uniform heat transfer and cooling within pouches was achieved by implementing 180° rotation during processing.

Practical Application:

Uniform heat transfer in thermal processing is crucial to ensure all parts of a product are sufficiently treated to achieve commercial sterility without unacceptable loss of quality. Optimizing pasteurization methods is recommended to preserve quality attributes such as color, texture, and flavor while maintaining food safety integrity. This research evaluated the temperature variability within a pouch during hot filling and lack of uniformity during processing. Food manufacturers producing acidic or acidified products could use this information to improve quality and safety in hot filled products. **Keywords**: Pasteurization, hot-fill and hold, uniform heating, acidic foods

INTRODUCTION

The application of heat treatment on food products, their package, and surrounding environment is one of the most important methods used in food processing. The purpose of thermal processing is to preserve food by destroying or inactivating microorganisms. The severity of heat treatment and resulting shelf life are determined mostly by the pH of the food product. Naturally acidic and acidified foods having a pH less than 4.6 can undergo a less severe heat treatment such as pasteurization or hot-filling (Fellows, 2009). Hot filling, also called "hot fill and hold", involves filling a sterilized acid or acidified product that is hot enough to sterilize the container into unsterilized containers (Adegoke, 2004). The temperatures required for hot filling pasteurization are typically 85°C or higher and a hot holding period (predetermined) may be required prior to the package being cooled (Ashurst, 2005). An adequate hot filling process can yield

shelf stable foods for acidic or acidified foods packaged in hermetically sealed containers (Kumar & Sandeep, 2014). Pasteurized products require a mild heat treatment resulting in minimal affects to sensory and nutritional characteristics.

The development of a proper thermal process relies on an understanding of how a product heats and cools under processing conditions. Thermal processes are time-temperature dependent: the higher the temperature, the shorter the time needed to destroy target microorganisms. The purpose of optimizing thermal processes is to improve the overall quality while assuring that the food is adequately heated throughout the container (Bornhorst, Liu, Tang, Sablani, & Barbosa-Canovas, 2017). The heat transfer within a container depends on the product type, size and shape of the container, processing method, and the heat transfer mechanism (Weddig, 2007). Thermal processing has a detrimental effect on some food quality attributes such as color, flavor, texture, and nutrients. It is important that food processors design a thermal process to balance the unavoidable needs required for food safety with the commercial desire to present a high-quality product (Bown, 2010).

Pasteurization processes can be optimized in a way that achieves the goal of food safety and minimizes the quality changes (Silva, Martins, & Silva, 2003). If a thermal process is applied longer than necessary to pasteurize or sterilize a product it can contribute to losses in product quality. More recent research has focused on process design and optimization for hot-filling in bottles (Claudia, Spinelli, Sant'Ana, Pacheco-Sanchez, & Massaguer, 2009; Skinner et al., 2015), and glass (Sandoval, Barreiro, & Mendoza, 1994). This research targets the hot filling of industrial sized pouches by

providing heat penetration and quality optimization studies, which is an area lacking in the literature available.

Due to this fact, this study examined the hot fill and hold process of tomato paste food simulant to determine the temperature variability within an industrial sized pouch. The objectives of this study were to (1) identify temperature profiles and rate of cooling within industrial sized pouches during a hot fill and hold process and (2) evaluate temperature differences throughout the process using a tomato based food simulant.

MATERIALS AND METHODS

The materials and methods used are described by two separate experiments referred to as Study 1 and Study 2. Study 1 evaluated the temperature trends occurring inside an industrial size pouch using water as the product. The results from Study 1 were used to identify the locations of importance for Study 2. Two locations were evaluated for Study 2 using a tomato food simulant as the product for the hot filling procedure. Study 2 compares the trends of temperature during hot-filling observed when the pouch was static versus agitated by rotation.

Pouch Preparation

A total of 75 clear institutional sized retort pouches measuring 29.21 x 38.1 cm (W x L) (Sealed Air Corporation, Charlotte, NC) were used in this experiment. The laminate structure of the pouches consisted of 12 micron polyethylene terephthalate (PET) with an oxide barrier coating, adhesive, 15 micron biaxially oriented nylon (BON), adhesive, and 100 micron retortable cast polypropylene (RCPP) (outside to inside).

Pouches were fitted with 26-gauge solid conductor copper/constantan wire, type T thermocouples with a hot junction (Ecklund-Harrison Technologies, INC., Fort Myers, FL). Thermocouple leads were 0.91 m in length and connected by subminiature male connectors (Cole-Parmer, Vernon Hills, IL) to a 12-channel hand held data logger (Model TM500 Extech Instruments, FLIR Commercial Systems Inc., Nahua, NH). All thermocouples were calibrated using boiling water as a reference to ensure precision within ±1°C. Thermocouples were connected to the pouch by puncturing a hole 4 cm from the desired location and inserted into position. After the thermocouple wire was inserted it was stabilized using red RTV gasket maker (Permatex, Hartford, CT) to seal the area around the wire to prevent leaks. The gasket maker was set and cured for at least 24 hours before filling.

Study 1. Identifying heating patterns and cold spots within industrial sized pouches during hot filling

Thermocouple Locations

Eight thermocouples were fitted to a pouch (Figure 3.1). Thermocouples 1 and 8 were positioned at the top of the pouch close to the where the pouch was sealed after filling. Location 5 was positioned in the geometric center of the pouch. Location 4 was placed directly below the geometric center at the bottom of the pouch, measured in the middle of the pouch horizontally. Locations 2 and 7 were fitted to the sides of the pouch measured in the middle by the length (vertically). Locations 3 and 6 were placed into the bottom corners of the pouch.

Process Methods

A steam jacketed kettle (Model KDPT-20, Crown Food Service Equipt. Ltd., Toronto, ON, Canada) was used to heat water to 100°C. Thermocouples fitted to the pouch were plugged into the datalogger. The data logging instrument was set to record at 10 second intervals and activated before filling. The thermocouple located in the center of the pouch (location 5) was adjusted into place prior to filling so the temperature recording end of the thermocouple was in the product of the vertically positioned pouch (Figure 3.2). After the water came to a boil, the pouch was filled with 3.80 L of water and immediately sealed using an impulse heat sealer (Model 9MS #1091, Toyo Jidoki CO., LTD, Tokyo, Japan) at 135°C sealing temperature with a heating time of one second and cooling time of one second. Pouches were then placed level on a conveyor and held for 4 minutes with an external air temperature of 21°C.

Cooling was simulated using a water spray constructed with PVC piping and six spray nozzles placed 45 cm apart allowing overlap in water spray range. The average flow rate of each nozzle was 1.60 L/min. The pouch was then allowed to cool for 16 minutes under the water spray reaching temperatures below 38°C. Twenty-five replicates were performed and a new premade pouch fitted with thermocouples was used to perform each experimental set.



Figure 3.1. Locations of thermocouples placed into pouch structure for Study 1.



Figure 3.2 Thermocouple location 5 position prior to filling.

Study 2. Evaluating temperature differences throughout hot filling process using tomato based food simulant

Tomato Based Simulant Preparation

The food simulant used for this study was composed of tomato paste (31 brix, The Morning Star Packing Company, Jacksonville, FL) and water. An immersion blender (Waring WSB50 Big Stick Heavy-Duty, Conair Corporation, East Windsor Township, NJ) was used to blend the tomato paste and water for at least 5 minutes until uniform mixing was achieved. The viscosity of the food simulant was measured using a Brookfield Viscometer (Model LVT, Brookfield AMETEK, Middleboro, MA). The sample was placed under the spindle attached to the viscometer and inserted into the beaker until the marking on the spindle was immersed into the sample. The samples were analyzed at 21±2°C for 5 minutes using spindle #1 at 30 rotations per minute (RPM). The ratio of the mixture of tomato paste and water by weight was 1:7 with a viscosity of 180±10 cP. Viscosity was measured before and after processing to ensure viscosity did not change (n=10).

Thermocouple Locations

Each experimental run used one pouch containing two thermocouples, one inserted in the geometric center and one inserted at the bottom corner of each pouch (Figure 3.3). These locations were chosen based on the statistical analysis from Study 1. Process Methods

A volume of 3.8 L of the tomato based food simulant was placed into a metal pot and heated using an induction cooker (Model IND-A120V, Admiral Craft Equipment

Corp., Westbury, NY) until boiling was reached. Thermocouples fitted to the pouch were plugged into the datalogger. The data logging instrument was set to record at 10 second intervals and was activated before filling. The thermocouple located in the center of the pouch (location 1) was adjusted into place prior to filling so the temperature recording end of the thermocouple was in the center of the vertical positioned pouch in the product. The tomato based simulant (3.8 L) was filled into the pouch after the water came to a boil and immediately sealed using an impulse heat sealer (Model 9MS #1091, Toyo Jidoki CO., LTD, Tokyo, Japan). Pouches were then placed level on a conveyor and held for 4 minutes and cooled for 16 minutes using the same methods as Study 1. The experiment consisted of 50 total simulated hot fill processes: 25 replicates of a 'static' process and 25 replicates of a 'rotational' process. The preparation and processing methods were repeated for 25 pouches with an additional step to the processing design. In this portion of the experiment, after the pouch was filled and sealed, the pouch was rotated 180° every 10 seconds during holding and cooling. The pouch was rotated by holding the end of the pouch by the seal area (the last seal made after filling) and flipped over to lay onto the flat surface. These two separate processes will be referred to as Process 1 and Process

2.



Figure 3.3. Thermocouple locations for Study 2.

Statistical Analysis

Analysis of variance (ANOVA) was used to interpret thermocouple location for each process. Differences among mean values were processed by Least Significant Difference (LSD). All analyses were conducted using SAS® (SAS Institute, Cary, NC) software, α =0.05. Temperature profiles were creating using the mean temperatures of each location with Tecplot 360 (Tecplot, Inc., Bellevue, WA).

RESULTS AND DISCUSSION

Study 1. Identifying heating patterns and cold spots within industrial sized pouches during hot filling

Temperature versus time was plotted for eight thermocouple locations (Figure 3.4). After pouches were filled, sealed, and placed onto the flat surface, location 5 at time zero had the highest average temperature and was significantly different than the average temperatures for locations 3, 4, 6, 7, and 8. Location 6 had the lowest average temperature and lower than locations 1, 2, 3, 4, 5, and 7 (p<0.05) (Table 3.1). The linear slopes during the four-minute period of holding were not significantly different (p>0.05). The average temperature was analyzed at 2 minutes during holding period (Table 3.2). Location 5, the geometric center of the pouch, had the highest average temperature and was significantly different than the rest of the locations in the pouch (p<0.05). The temperature profile of the pouch at time zero and two minutes (Figure 3.5 and 3.6) show the areas with the lower temperatures shown by a blue color (corners of the pouch) and the higher temperatures indicated by red (the center). Locations 3 and 6 were lower in average temperature than locations 1, 5, and 7 (p<0.05).

The rate of cooling was determined for the processing portion from minute/time interval 4:10 to 20:00, the duration which the water spray was applied to cool the pouch. Newton's Law of cooling states the rate of change of the temperature of an object is proportional to the difference between its own temperature and the ambient temperature. There was a significant difference found between the mean rates of cooling for location 5 and 6 (Table 3.3). Location 5 represents the area in the geometric center of the pouch and

location 6 represents the bottom corner of the pouch. Location 5, the geometric center of the pouch cooled at the slowest rate and had lower rates than location 1,3, 4, 6, 7, 8 (p<0.05). Location 6 had a higher rate of cooling than locations 1, 2, 3, 4, 5, and 7 (p<0.05). The rates of cooling for locations 1, 2, 3, 4, 7, and 8 were not significantly different (p>0.05).

It was expected the geometric center would have the slowest rate of cooling. The center region of the pouch has a larger amount of fluid that cools slower than the fluid in the corners and sides of the pouch that have a smaller area of fluid. Different rates of heat transfer have been demonstrated during a hot fill process within inverted bottles. The differences in lowest and highest temperatures within a cooling bottle were identified (Skinner et al., 2015). In thermal processing, it is important to adequately cool food products to ambient or chilled conditions to avoid product damage after heat treatment (Motarjemi, Moy, & Todd, 2014). The area of the pouch with a slower rate of cooling may receive additional heat treatment contributing to losses of sensory and quality attributes, overall producing a lower quality product. It is crucial to have uniform and rapid cooling to preserve a product's nutritive value, color, texture, and external appearance.

The center of the pouch was initially the highest temperature during hot filling (Table 3.1 and 3.2) and had the slowest rate of cooling. Location 6, the bottom left corner of the pouch was found to be initially the lowest temperature during holding and had the slowest rate of cooling. These two locations were chosen to test in the research

experiment representing the areas receiving over processing and under processing within a pouch during hot filling.



Figure 3.4. Time temperature data for Study 1.



Figure 3.5. Temperature profile of pouch at time 0.



Figure 3.6. Temperature profile of pouch at 2 minutes during the holding period.

Location	Mean		t Grouping
5	95.241		А
			А
1	94.184	В	А
		В	А
2	93.997	В	А
		В	
3	92.728	В	С
		В	С
4	92.640	В	С
			С
7	92.269		С
8	92.300		D
			D
6	89.787		D

Table 3.1. Study 1, SAS output for mean temperature (°C) recorded at time 0 (seconds) organized from highest to lowest temperature.

Significance (p<0.05) represented by different letters.

Location	Mean		t Grouping
5	93.946		А
1	93.239		В
			В
7	92.923		В
			В
2	92.789	С	В
		С	В
8	92.729	С	В
		С	В
4	92.672	С	В
		С	
6	92.270	С	
		C	
3	92.198	Ċ	

Table 3.2. Study 1, SAS output for mean temperature (°C) at time 2 min. organized from highest to lowest temperature.

Significance (p<0.05) represented by different letters.

Location	Mean	t Grouping		
6	0.132291		А	
			А	
8	0.124489	В	А	
		В		
7	0.121197	В	С	
		В	С	
4	0.121043	В	С	
		В	С	
3	0.117087	В	С	D
		В	С	D
1	0.116541	В	С	D
			С	D
2	0.114137		С	D
				D
5	0.112228			D

Table 3.3. Study 1, SAS output for k (cooling rate) from fastest to slowest rate.

Significant difference at 0.05 level is indicated by different letters.

Study 2. Evaluating temperature differences throughout hot filling process using tomato based food simulant

For the static process, locations 1 and 2 showed no difference between average temperature (p<0.05) at time '0', once the pouch was sealed and placed onto the flat surface (Table 3.4). Location 1 had a higher average than location 2 for the rotational process at the initial temperature measurement (0 seconds, Table 3.5) (p<0.05). At 60, 120, and 240 seconds of processing time, the average temperature for location 1 and 2 were significantly different (p<0.05) for both processes.

The mean temperatures for both processes were compared for each location throughout the holding period, 0 to 240 seconds (Table 3.6). For location 1, the average temperatures evaluated at 0, 60, 120, and 240 seconds were significantly different between static and rotational process methods (p<0.05). At 0, 120, and 240 seconds the

average temperatures for location 2 were different between the processes (p<0.05). At 60 seconds, location 2 was not different between the static and rotational processes (p>0.05).

In Figure 3.7, the trends of the time-temperature data are shown for the static process. The rate of cooling for the center and corner locations of the pouch vary greatly. The center of the pouch did not show a trend of cooling with the average temperature remaining above pasteurization temperatures (86.4°C) at the end of the cooling time of 20 minutes. The corner of the pouch had a much faster rate of cooling and fell below 85°C after 60 seconds, with a final temperature of 21.8°C at the end of the cooling process. The time-temperature data for the rotational process with tomato simulant is shown by Figure 3.8. The trends of heating for both locations had a similar trend and demonstrated that the product was being cooled. The average temperature of the pouch corner fell below 85°C at 130 seconds after filling. The center fell below pasteurization temperatures after 90 seconds in the cooling step. The average temperatures at the end of the cooling process for location 1 and 2 were 33.2°C and 27.5°C, respectively.

Uniform heating and cooling was achieved by implementing 180° rotation during processing. Without implementing rotation, the center of the pouch (location 1) retained more heat than the corner of the pouch (location 2). After applying 180° rotation during the process, a longer retention of heat was observed for the corner of the pouch during the holding step and the center of the pouch had a faster rate of cooling. Uniform and rapid heat transfer is needed to produce higher quality products. If the thermal process is applied longer than necessary to pasteurize or sterilize a product it can contribute to losses in product quality. It is important to determine optimal thermal processes methods

capable of achieving sterilization and the highest quality attributes, especially color for tomato products (Sandoval et al., 1994).



Figure 3.7. Time-temperature data for tomato food simulant process 1, Study 2.



Figure 3.8. Time-temperature data for tomato food simulant process 2, Study 2.

Time	Location	Mean	Letter Group
0	1	95.917	А
0	2	94.785	А
60	1	95.964	А
60	2	85.584	В
120	1	95.800	А
120	2	77.957	В
240	1	95.497	Α
240	2	72.025	В

Table 3.4. Study 2, average temperatures (°C) for Process 1.

Table 3.5. Study 2, average temperatures (°C) for Process 2.

			Letter
Time	Location	Mean	Group
0	1	94.772	А
0	2	94.456	В
60	1	93.700	А
60	2	89.356	В
120	1	92.544	А
120	2	83.719	В
240	1	89.186	А
240	2	83.167	В

Table 3.6. Mean temperature comparison from static and rotational processes. Significant difference p<0.05.

Time	Location	F Value	Pr>F
0	1	17.47	0.0005*
0	2	1.27	0.2703
60	1	46.78	<.0001*
60	2	34.09	<.0001*
120	1	72.01	<.0001*
120	2	23.30	<.0001*
240	1	51.99	<.0001*
240	2	88.53	<.0001*

CONCLUSION

This study concluded during a simulated hot fill process, the product in the center of a pouch receives more heat treatment than compared to the corner. During the hot fill and hold steps the product at the center of the pouch had a higher mean temperature than product located in the corner of the pouch. The corner of the pouch had a faster cooling rate than the center. There was a significant difference (p<0.05) found between mean temperatures of the center and corner locations using a low viscosity food simulant. The application of 180° rotation to the package throughout the process helped create a more uniform heat transfer and rate of cooling of the product.

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CHAPTER FOUR

THE EFFECTS OF THERMAL PASTEURIZATION AND MULTILAYER PACKAGING FILMS ON THE COLOR OF A FOOD SIMULANT

ABSTRACT

An accelerated shelf life study was conducted to investigate the effects of time and temperature combinations during the retention period for a hot fill and hold process of pouches and the influence of multilayer film on color quality of a tomato based food simulant. A low viscosity food simulant consisting of tomato paste and water mixture was filled into pouches constructed of three different multilayer films. The pouches were pasteurized in container using a hot water bath and held using four different time and temperature combinations. After processing, the pouches were cooled and stored in an environmental chamber for eight weeks $(35\pm2^{\circ}C \text{ and } 50\pm2\% \text{ RH})$. The change in color of the food simulant was evaluated using a HunterLab ColorFlex EZ spectrophotometer model 45/0 LAV (Hunter Associates Laboratory, Inc., Reston, Virginia) measuring luminosity (L*), red-green component (a*), blue-yellow component (b*), tomato paste score (TPS), and a*/b* ratio. Food simulant packed in Film A and C pouches showed greater color retention during accelerated storage than Film B. The structures of these films provided a good barrier to oxygen resulting in a higher retention of color. Films A and C showed similar quality degradation with no significant difference in TPS across all processing conditions (p < 0.05). The different time and temperature combinations had little impact on the retention of TPS at the end of storage for Film A and C. The type of barrier had a greater impact on the color quality of tomato paste than the different process combinations of time and temperature.

INTRODUCTION

For acidic foods having a pH lower than 4.6, pasteurization is commonly used to destroy harmful organisms such as bacteria, enzymes, molds, and yeast to create a shelf stable product that can be stored at ambient temperatures for several months (Castberg, Osmundsen, & Solberg, 1995). The product high in acidity (pH<4.6) can be stored in ambient conditions after pasteurization processing due to the acidic environment of the food that is not conductive of growth for harmful organisms. The application of a hot fill and hold process is one technique used to pasteurize liquid products such as fruit and vegetable juices, fruit purees, and non-carbonated beverages are a few examples. During hot fill and hold processes, the commercial sterility of a product is achieved by pasteurizing the product and by inactivating spoilage organisms that may be in the container with the transfer of heat from the sterile product to the container. Current procedures for hot fill and hold process procedures for the filling temperature and holding time are based on conservative estimates that tend to over process product. There is a need to determine optimum fill temperature and retention times to assure commercial sterility and preserve the quality of the product. Sandoval et al. (1994) reported that a time and temperature combination of low temperature, long time can induce undesirable changes in the quality attributes and nutritive value of the product. It is recommended to determine an adequate thermal process that achieves food safety and provides the best quality of the product (Sandoval, Barreiro, & Mendoza, 1994).

Packaging plays a major role in preserving the safety and quality of processed foods during the entire period from its production until it reaches the end user. Food is

complex in nature and may deteriorate when it encounters water vapor and gases, especially oxygen. The presence of oxygen provides favorable conditions for the growth of aerobic microorganisms, favors enhanced respiration for fruits and vegetables including enzymatic reactions. Packaging type, oxygen and water vapor transmission rate, and amount of residual oxygen in the package are the main factors that determine how fast the degradation of a food can occur (Robertson, 2009). Today, there is a large variety of packaging materials available with a growing development in plastic films and materials. Packaging film is a very thin plastic, primarily composed of polymers and is instrumental in decreasing the amount of the food supply lost to spoilage. The development of multilayer films has provided better packaging materials for food products that undergo long storage periods. Multiple layers of polymers are combined to achieve the needed properties (barrier and others) and the required shelf life. Polymeric packaging materials are used to surround a product completely, providing a barrier to gasses, moisture, and biological effects of the outside environment. Most films used for food packaging are composed of a multilayer structure with barrier materials of ethylenevinyl alcohol (EVOH), high density polyethylene (HDPE), nylon, or oriented polypropylene. These films can be produced in various ways such as blown and cast film, and coextrusion (McKeen, 2012). Packaging materials must withstand the processing conditions, maintain its physical integrity, and visual appearance. Thermal processing can have a significant effect on the barrier properties of a packaging material thus, deteriorating food quality. Research has been carried out to study the effect of thermal

pasteurization on the properties of packaging materials (Ramalingam, VA, George, & SN, 2015).

While many authors have investigated the effect of thermal pasteurization on overall quality of a product or the effects of processing on barrier properties of polymeric packaging materials, separately, few researchers have published results investigating the application of polymeric films on adverse shelf life and color quality. The objectives of this study were (1) to compare the effect of time and temperature combinations of hot fill and hold process and (2) evaluate different film structures on the color quality of a tomato based food simulant during accelerated shelf life storage.

MATERIALS AND METHODS

Tomato Based Simulant Preparation

The food simulant used for this study was composed of tomato paste (31 brix, The Morning Star Packing Company, Jacksonville, FL) and water. An immersion blender (Waring WSB50 Big Stick Heavy-Duty, Conair Corporation, East Windsor Township, NJ) was used to blend the tomato paste and water for at least 5 minutes until uniform mixing was achieved. The viscosity of the food simulant was measured using a Brookfield Viscometer (Model LVT, Brookfield AMETEK, Middleboro, MA). The sample was placed under the spindle attached to the viscometer and inserted into the beaker until the marking on the spindle was immersed into the sample. The samples were analyzed at 21±2°C for 5 minutes using spindle #1 at 30 rotations per minute (RPM). The ratio of the mixture of tomato paste and water by weight was 1:7 with a viscosity of

 180 ± 10 cP. Viscosity was measured before and after processing to ensure viscosity did not change (n=10).

Film structures

The film structures used in this study were supplied by Sealed Air Corporation (Charlotte, NC) as follows:

<u>Film A:</u>

0.47 mils polyethylene terephthalate (PET) with an aluminum oxide (AlO_x) barrier coating/ Adhesive/ 0.60 mils biaxially oriented nylon (BON)/adhesive/ 3.9 mils retort-able cast polypropylene (RCPP)

<u>Film B:</u>

1.38 mils Polyethylene/ 0.55 Adhesive/ 0.55 nylon/0.55 ethylene-vinyl alcohol (EVOH)/ 0.55 nylon/0.55 Adhesive/ 1.38 Polyethylene

Film C:

1.46 mils Polyethylene/ 0.65 mils Adhesive/0.65 mils nylon/ 0.98 mils ethylenevinyl alcohol (EVOH)/ 0.65 mils nylon/ 0.65 mils Adhesive/ 1.46 Polyethylene

Pouches were created from rolled stock film. Films were cut into 5.75 x 7.5in rectangles. Films were fabricated into three-sided seal pouches using impulse heat sealer (Model 9MS #1091, Toyo Jidoki co., LTD, Tokyo, Japan) with the following parameters of 135°C sealing temperature with a heating time of one second and cooling time of one second. 200 mL of the tomato food simulant were added to each pouch to create a thin

profile and were manually sealed with minimal headspace (Figure 4.1). Thirty replicates were made for each film type.



Figure 4.1. Packaged food simulant.

Processing Conditions

Results from the previous study (described in Chapter Three) were used to select the processing time and temperature combinations representative of hot filling conditions of a pouch. The processing combinations of time and temperature were selected for the center and corner pouch of a static and rotation hot filling process procedure (Processing conditions: P-1: static middle process, P-2: static corner, P-3: rotation middle, and P-4: rotation corner process) (Table 4.1). Prefilled pouches containing food simulant were processed by in container pasteurization using a hot water bath (Model WB10, Cole-Parmer, Vernon Hills, IL). Test tube racks were placed horizontally in the water bath to hold pouches into place. Six pouches were placed into the water bath at a time (Figure 4.2), two of those pouches with one type T thermocouple with a hot junction (Ecklund-Harrison Technologies, INC., Fort Myers, FL) inserted into the center of the pouch (Figure 4.3). Thermocouple leads were 0.91 m in length and connected by subminiature male connectors (Cole-Parmer, Vernon Hills, IL) to a 12-channel hand held data logger (Model TM500 Extech Instruments, FLIR Commercial Systems Inc., Nahua, NH). All thermocouples were calibrated using boiling water as a reference to ensure precision within $\pm 1^{\circ}$ C. Thermocouples were connected to the pouch by puncturing a hole at the geometric center of the pouch and placed one centimeter into the desired location. After the thermocouple wire was positioned it was stabilized using red RTV gasket maker (Permatex, Hartford, CT) to seal the area around the wire to prevent leaks. The gasket maker was set and cured for at least 24 hours before filling. Once processing times were completed, pouches were removed and cooled using the same methods previously described (Ch. 3).

Process	Temperature (°F)	Time (minutes)
P-1	204	9
P-2	204	3
P-3	200	4
P-4	180	4

Table 4.1. Time and temperature combinations for each process.


Figure 4.2. In-container pasteurization method using a water bath.



Figure 4.3. Thermocouple inserted into the geometric center of a pouch.

Storage Conditions and Sampling

The processed pouches were stored in an environmental chamber (Model 6020, Caron Products and Services, Inc., Marietta, OH) at 35±2°C with 50±2% RH for 8 weeks. Samples were measured every 2 weeks. Triplicate samples from each film material and process condition were removed from the chamber on each sampling day for color analysis. Mean values were calculated from 6 measurements for each variant. <u>Color analysis</u>

The change in color during storage was measured using a HunterLab ColorFlex EZ spectrophotometer model 45/0 LAV (Hunter Associates Laboratory, Inc., Reston, Virginia). The samples were analyzed using EasyMatch QC version 4.84 (Hunter Associates Laboratory, Inc., Reston, Virginia). The instrument was calibrated prior to use for every test with the standard white and black tiles, in addition the instrument was calibrated using a reference tile, HunterLab Tomato Tile, 45/0 Reference Standard (Hunter Associates Laboratory, Inc., Reston, Virginia). A glass sample cup containing sample was placed above the light source and the provided black cup was placed over the sample. L*a*b*, TPS (Tomato Paste Score), a/b ratio were the indices used to measure samples. Color was expressed by Hunter Lab units: L* (white to black or light to dark), a* (red to green), and b* (yellow to blue). Where a* and b* readings are reported by the instrument, TPS:

 $TPS = -81.582 + 1.069 a + 15.390 b - 0.591 b^{2} (USDA Fruit and Vegetable$ Program Specialty Crops Inspection (SCI) Division)

Statistical Analysis

All color analysis readings were subjected to analysis of variance (ANOVA) to determine significant differences (p<0.05) between means with consideration of process, film type, and time using SAS® (SAS Institute, Cary, NC).

RESULTS AND DISCUSSION

Color is an important attribute of tomato paste and is used to determine the final quality of thermally processed tomato products (Ganje et al., 2016). The lycopene pigments are responsible for the degree of redness of a tomato product. The degradation of lycopene occurs mainly due to thermal isomerization and autoxidation. Accelerated shelf life testing is an effective way to study the changes in quality attributes and desirable performance of a product with a considerably long shelf life, such as tomato paste. HunterLab parameters (L*, a*, and b* values, and TPS, and a/b ratio) were measured in different processes, film types, over 8 weeks in accelerated storage conditions.

Film Performance

Process 1 (P-1)

The mean L*, a*, b*, TPS, and a/b ratio were all significantly different at the end of storage for P-1, Film B (p<0.05) (Table 4.2, 4.3, 4.4, 4.5). At the end of storage, the mean values for all measurements were also significantly different compared to Film A and B (Table 4.2, 4.3, 4.4, 4.5). The highest rates of change were also observed for Film B (Table 4.7). For Film A, the mean L* value was significantly different at the end of storage, where a*, b*, TPS, and a/b ratio were not significantly different. For Film C, the mean L* and a* values were significantly different at the end of storage. At 8 weeks of

storage, L*, a*, b*, TPS, and a/b ratio were not significantly different between Film A and C (p>0.05) (Table 4.1, 4.2, 4.3, 4.4). Overall, Film 2 had the greatest rate of change for all indices measured under storage conditions (Table 4.7).

Process 2 (P-2)

The mean L*, a*, b*, TPS, and a/b ratio were all significantly different at the end of storage for P-2, Film B (p<0.05) (Table 4.1, 4.2, 4.3, 4.4). At the end of storage, the mean values for all measurements were also significantly different compared to Film A and B (Table 4.1, 4.2, 4.3, 4.4). The greatest rates of change were also observed for Film B (Table 3.7). For Film A, the mean L* value was significantly different at the end of storage. For Film C, L*and a* values, TPS, and a/b ratio were significantly different at the end of storage. For P-2, at the end of storage, the mean values of all indices were not significantly different between Film A and B (p>0.05).

Process 3 (P-3)

The mean L*, a*, b*, TPS, and a/b ratio were all significantly different at the end of storage for P-3, Film B (p<0.05) (Table 4.1, 4.2, 4.3, 4.4). At the end of storage, the mean values for all measurements were also significantly different compared to Film A and B (Table 4.1, 4.2, 4.3, 4.4). L*, a*, and b* values, and TPS were significantly different at the end of storage (p<0.05). All indices for Film C at the end of storage were not significantly different (p>0.05).

Process 4 (P-4)

The mean L*, a*, b*, TPS, and a/b ratio were all significantly different at the end of storage for P-4, Film B (p<0.05). At the end of storage, the mean values for all

measurements were also significantly different compared to Film A and B (Table, 4.2, 4.3, 4.4, 4.5). All indices for Film A at the end of storage were significantly different (p<0.05). L*, and a* values, TPS and a/b ratio were significantly different at the end of storage (p<0.05). For P-1 and P-2 there were no significant differences in color retention for Films A and B at the end of storage. For P-3 and P-4, film C resulted in a higher mean L*value than film 1. Film C also had higher b*values at the end of storage with P-4. The performance of Film A and C were similar in preventing color degradation from occurring.



Figure 4.4. Changes in L* values for all films for P-1.



Figure 4.5. Changes in L* values for all films for P-2.



Figure 4.6. Changes in L* values for all films for P-3.



Figure 4.7. Changes in L* values for all films for P-4.

	Time					
L* value	(weeks)	0	2	4	6	8
P-1						
	Film A	23.247 ^{a1}	23.047 ab1	23.217 ^{a1}	22.897 ^{b1}	22.968 b1
	Film B	23.172 a1	22.195 ^{b2}	21.643 ^{c2}	21.210 d2	20.905 e2
	Film C	23.387 ^{a1}	22.872 ^{c1}	23.205 ab1	23.035 bc1	23.117 b1
<i>P-2</i>						
	Film A	23.348 a1	23.222 ab1	23.368 ^{a1}	23.005 b2	23.018 ^{b1}
	Film B	23.495 a1	22.408 ^{b2}	22.012 ^{c2}	21.465 d3	21.193 e ²
	Film C	23.527 ^{a1}	23.258 b1	23.393 ab1	23.357 ab1	23.012 ^{c1}
<i>P-3</i>						
	Film A	23.248 a12	22.975 b1	22.438 d2	22.583 ^{cd2}	22.697 ^{c2}
	Film B	23.383 ^{a1}	22.393 ^{b2}	21.822 ^{c3}	21.328 d3	21.078 e3
	Film C	23.122 ^{b2}	23.010 ^{b1}	23.473 ^{a1}	23.140 ^{b1}	22.968 b1
<i>P-4</i>						
	Film A	23.222 a1	23.292 a1	22.975 b1	22.380^{d2}	22.628 ^{c2}
	Film B	23.262 a1	22.427 ^{b2}	22.173 ^{c2}	21.528 d3	21.348 d3
	Film C	23.253 a1	23.0867 ab1	23.138 ab1	23.122 ab1	23.010 ^{b1}

Table 4.2. L* values over 8 weeks exposure at $35{\pm}2^{\circ}C$ and $50{\pm}2\%$ RH.



Figure 4.8. Changes in a* values for all films for P-1.



Figure 4.9. Changes in a* values for all films for P-2.



Figure 4.10. Changes in a* values for all films for P-3.



Figure 4.11. Changes in a* values for all films for P-4.

	Time					
a* value	(weeks)	0	2	4	6	8
P-1						
	Film A	20.477 ^{c2}	21.677 ^{a1}	21.153 b1	20.478 ^{c1}	20.5750 ^{c1}
	Film B	21.103 a1	19.652 ^{b3}	17.770 ^{c2}	16.822 ^{d2}	16.303 ^{e2}
	Film C	21.143 a1	$20.977 \ ^{ab2}$	20.867 ^{abc1}	20.417 ^{c1}	20.653 bc1
<i>P-2</i>						
	Film A	21.013 b2	21.923 a1	21.158 b1	20.730 ^{b2}	20.685 b1
	Film B	21.487 ^{a12}	20.102 ^{b2}	18.438 ^{c2}	17.377 ^{d3}	16.837 ^{e2}
	Film C	21.570 ^{b1}	22.087 ^{a1}	21.457 ^{b1}	21.470 ^{b1}	20.697 ^{c1}
P-3						
	Film A	$20.878 b^2$	21.502 ^{a1}	19.417 ^{d2}	19.818 cd2	20.128 ^{c1}
	Film B	21.430 a1	20.115 b2	18.133 ^{c3}	17.138 ^{d3}	16.563 ^{e2}
	Film C	20.692 ^{b2}	21.357 ^{a1}	21.440 ^{a1}	20.858 ^{b1}	20.553 b1
<i>P-4</i>						
	Film A	20.818 b1	22.135 ^{a1}	20.107 ^{c1}	19.717 ^{c2}	19.912 ^{c1}
	Film B	21.118 ^{a1}	$20.177 b^{3}$	18.498 ^{c2}	17.533 ^{d3}	17.162 ^{d2}
	Film C	21.148 ab1	21.497 ^{a2}	20.525 ^{c1}	20.808 bc1	20.348 ^{c1}

Table 4.3. a* values over 8 weeks exposure at 35±2°C and 50±2% RH.



Figure 4.12. Changes in b* values for all films for P-1.



Figure 4.13. Changes in b* values for all films for P-2.



Figure 4.14. Changes in b* values for all films for P-3.



Figure 4.15. Changes in b* values for all films for P-4.

	Time					
b* value	(weeks)	0	2	4	6	8
P-1						
	Film A	12.753 ab2	12.868 ^{a1}	12.827 ^{a1}	12.622 ^{b1}	12.762 ab1
	Film B	12.850 a12	12.147 ^{b3}	11.555 ^{c2}	11.207 ^{d2}	11.077 ^{d2}
	Film C	12.948 ^{a1}	12.653 ^{b2}	12.753 ^{b1}	12.630 ^{b1}	12.800 ab1
<i>P-2</i>						
	Film A	12.892 ab2	12.973 ^{a1}	12.913 ^{ab1}	12.662 ^{c2}	12.761 bc1
	Film B	13.068 a12	12.308 ^{b2}	11.807 ^{c2}	11.430 ^{d3}	11.313 ^{d2}
	Film C	13.075 ^{a1}	13.013 ^{a1}	12.973 ^{a1}	12.985 ^{a1}	12.770 ^{a1}
<i>P-3</i>						
	Film A	12.810 ^{a2}	12.750 ^{a1}	12.135 ^{c2}	12.345 b2	12.508 b1
	Film B	12.993 a1	12.310 ^{b2}	11.658 ^{c3}	11.337 ^{d3}	11.173 ^{d2}
	Film C	12.693 b1	12.743 ^{b1}	12.928 a1	12.742 ^{b1}	12.668 b1
<i>P-4</i>						
	Film A	12.750 ^{b1}	13.017 ^{a1}	12.525 ^{c1}	12.307 d2	12.433 cd2
	Film B	12.842 a1	12.277 ^{b3}	11.792 ^{c2}	11.468 ^{d3}	11.382 ^{d3}
	Film C	12.820 a1	12.772 ^{a2}	12.653 a1	12.725 a1	12.663 a1

Table 4.4. b* values over 8 weeks exposure at 35±2°C and 50±2% RH.



Figure 4.16. Changes in TPS for all films for P-1.



Figure 4.17. Changes in TPS for all films for P-2.



Figure 4.18. Changes in TPS for all films for P-3.



Figure 4.19. Changes in TPS for all films for P-4.

		Time					
TPS		(weeks)	0	2	4	6	8
	<i>P-1</i>						
		Film A	$40.457 b^2$	41.732 ^{a1}	41.192 ^{a1}	40.392 ^{b1}	40.532 ^{b1}
		Film B	41.125 ^{a1}	39.167 ^{b3}	36.335 ^{c2}	34.647 ^{d2}	33.800 ^{e2}
		Film C	41.198 ^{a1}	40.903 ab2	40.865 ab1	40.333 b1	40.655 ab1
	<i>P-2</i>						
		Film A	41.060 bc1	42.037 ^{a1}	41.313 ^{b1}	40.683 ^{c2}	40.383 ^{c1}
		Film B	41.598 ^{a1}	39.798 ^{b2}	37.445 ^{c2}	35.685 ^{d3}	34.885 ^{e2}
		Film C	41.677 ^{ab1}	42.213 a1	41.548 ^{b1}	41.555 ^{b1}	40.687 ^{c1}
	P-3						
		Film A	40.898 b12	41.540 ^{a1}	38.735 ^{d2}	39.517 ^{c2}	39.957 ^{c1}
		Film B	41.507 ^{a1}	39.812 ^{b2}	36.898 ^{c3}	35.250 ^{d3}	34.293 ^{e2}
		Film C	40.673 ^{c2}	41.345 ab1	41.522 ^{a1}	40.848 bc1	40.448 ^{c1}
	<i>P-4</i>						
		Film A	40.818 ^{b1}	42.267 ^{a1}	39.908 cd1	39.322 ^{d2}	39.980 ^{c1}
		Film B	41.148 ^{a1}	39.853 ^{b3}	37.488 ^{c2}	35.930 ^{d3}	35.363 ^{d2}
		Film C	$41.187 \ ^{ab1}$	41.540 ^{a2}	40.467 ^{c1}	40.800 bc1	40.273 ^{c1}

Table 4.5. Tomato Paste Scores (TPS) over 8 weeks exposure at 35±2°C and 50±2% RH.



Figure 4.20. Changes in tomato a/b for all films for P-1.



Figure 4.21. Changes in tomato a/b for all films for P-2.





Figure 4.23. Changes in tomato a/b for all films for P-4.

	Time					
a/b Ratio	(weeks)	0	2	4	6	8
P-1						
	Film A	1.606 ^{c2}	1.761 ^{a1}	1.649 ^{b1}	1.622 ^{c1}	1.612 ^{c1}
	Film B	1.637 ^{a1}	1.618 ^{a3}	1.528 ^{b2}	1.501 ^{c2}	$1.472 d^2$
	Film C	1.628 ^{b12}	1.657 ^{a2}	1.636 ab1	1.617 ^{b1}	1.613 ^{b1}
<i>P-2</i>						
	Film A	1.630 ^{b1}	1.689 ^{a1}	1.644 ^{b1}	1.637 ^{b1}	1.621 ^{b1}
	Film B	1.648 ^{a1}	1.633 ^{a2}	1.562^{b2}	1.520 ^{c2}	1.489 ^{d2}
	Film C	1.651 ^{b1}	1.697 ^{a1}	1.654 ^{b1}	1.653 ^{b1}	1.621 ^{c1}
P-3						
	Film A	1.629 ^{b1}	1.686 ^{a1}	1.598 ^{c2}	$1.605 bc^2$	1.609 ^{bc1}
	Film B	1.647 ^{a1}	1.634 ^{a2}	1.555 ^{b3}	1.512 ^{c3}	$1.482 d^2$
	Film C	1.629 ^{c1}	1.675 ^{a1}	1.658^{ab1}	1.637 bc1	1.621 ^{c1}
<i>P-4</i>						
	Film A	1.633 ^{b1}	1.700 ^{a1}	1.605 ^{c1}	1.601 ^{c2}	1.604 ^{c1}
	Film B	1.645 ^{a1}	1.643 ^{a2}	1.569 ^{b2}	1.529 ^{c3}	1.509 ^{c2}
	Film C	1.648 ^{b1}	1.683 ^{a1}	1.622 ^{cd1}	1.638 bc1	1.607 ^{d1}

Table 4.6. Tomato a/b Ratio over 8 weeks exposure at 35±2°C and 50±2% RH.

Process	Film					
		L*	a*	b*	TPS	a/b
P-1						
	Film A	-0.035	0.012	0.001	0.009	0.001
	Film B	-0.283	-0.600	-0.222	-0.916	-0.021
	Film C	-0.034	-0.061	-0.018	-0.068	-0.002
<i>P-2</i>						
	Film A	-0.041	-0.041	-0.016	-0.085	-0.011
	Film B	-0.288	-0.581	-0.219	-0.839	-0.105
	Film C	-0.064	-0.109	-0.038	-0.124	-0.015
<i>P-3</i>						
	Film A	-0.069	-0.094	-0.038	-0.118	-0.003
	Film B	-0.288	-0.608	-0.227	-0.902	-0.020
	Film C	-0.019	-0.017	-0.003	-0.028	-0.001
<i>P-4</i>						
	Film A	-0.074	-0.113	-0.040	-0.105	-0.004
	Film B	-0.239	-0.495	-0.182	-0.723	-0.017
	Film C	-0.030	-0.100	-0.020	-0.114	-0.005

Table 4.7. Rates of change for food simulant over 8 weeks exposure at $35\pm2^{\circ}$ C and $50\pm2\%$ RH.

Processing Condition

Film A

Color was measured after processing and is represented as time zero (Figures 4.24, 4.27, 4.30, 4.34, 4.36). There were no significant differences between the mean TPS across all processes for Film A at time zero (p>0.05). P-2 had highest TPS value at the end of storage and was significantly different compared to P-3 and P-4. P-3 and P-4 significantly decreased at the end of storage (p<0.05) (Table 4.11). P-2 decreased in mean TPS by 0.942 and P-4 by 0.838. The greatest rate of change in TPS values was shown by P-3, followed by P-4, P-2, and P-1 (Table 4.13).

Film B

There was variation in color change between the four combinations of time and temperature processing conditions for Film B (Figures 4.25, 4.28, 4.31, 4.34, 4.37). Initially at time 0, there were no significant differences between processing conditions for TPS and a/b ratio means (p>0.05). Regardless of process type, the mean values for all indices of L*, a*, b* values, and TPS, and a/b ratio significantly decreased throughout storage. Regarding TPS, P-1 and P-3 were not significantly different at the end of storage. P-4 had the greatest mean TPS and was significantly different than the other three processes (p<0.05) (Table 4.11). Throughout storage, the TPS value decreased for P-1 by 7.32 units, P-2 by 6.71 units, P-3 by 7.21 units, P-4 by 5.78 units compared at time zero. The greatest rate of change in TPS values was shown by P-1, followed by P-3, P-2, and P-4 (Table 4.13).

Film C

The change in color is represented by means of L*, a*, and b* values, TPS, and tomato a/b ratio (Figures 3.26, 3.29, 3.32, 3.35, and 3.38). At time zero, P-2 had the highest TPS value and was significantly different than P-3 (p<0.05) (Table 4.11). The TPS means for P-2, P-3, and P-4 were not significantly different after the process was applied (p>0.05). P-2 and P-4 significantly decreased in TPS values at the end of storage (p<0.05). P-2 decreased in mean TPS by 0.989 and P-4 by 0.913. At the end of eight weeks of storage, there were no significant differences between TPS values for all processes (p>0.05).

Overall, Film B had the greatest rate of change under all processing conditions compared to Films A and C which had significantly higher L*, a*, b* values, TPS, and a/b ratio after 8 weeks of accelerated storage conditions. Regarding TPS, the rate of change for Film A across all four processes ranged from 97.7 to 6.90 times less than Film B. The rate of change for Film C across all processes ranged from 32.0 to 6.33 less than Film B. The slow rate of change observed in tomato paste color packaged and processed in Films A and C indicate that these materials have a good barrier to oxygen. The degradation of lycopene, which imparts red color to tomatoes, during storage is mainly caused by oxidation. The fate of lycopene in processed tomato products is influenced by storage conditions. The most important factor contributing to lycopene degradation during storage is the availability of oxygen. Exposure to oxygen can cause the naturally occurring all-trans lycopene to be isomerized and oxidized. This conversion to mono-cis and poly-cis forms of lycopene leads to a loss of red color (Shi & Maguer, 2000). The presence of oxygen could be one of the reasons for tremendous change in color observed in Film B than compared with other films. This demonstrates that barrier properties of a material prove to be a significant factor, but it is not the only factor contributing to color change.

The presence of sugar, acids, and amino acids also affects the color of processed tomato products by causing the formation of brown pigments (Gould, 1992). During the heating of fruits and vegetables various reactions can occur that affect their color, such as pigment destruction (carotenoids and chlorophylls) and non-enzymatic browning (Maillard) reactions (Ávila & Silva, 1999). Figure 4.34 shows the effect of each process

on the mean values of TPS for Film B. There was a difference between processes, with a decrease in TPS with an increase of the time and temperature combination. P-1 had the lowest mean value of TPS, which was also the highest temperature treatment with the longest retention time (Table 4.1). The shortest retention time and lowest temperature combination, P-4, had the highest TPS mean value (greatest retention of color). The loss of color in processed tomato products is accelerated by high temperatures and long treatment (Shi & Maguer, 2000). Longer retention times in hot fill and hold can produce undesirable changes in quality attributes of tomato paste (Sandoval et al., 1994).

The barrier properties of Film A and C resulted in a higher retention of color. Film A's barrier layers consisted of PET with an AlO_x barrier coating, BON, and RCPP. The method of surface modification is used in food packaging to improve barrier properties such as silicon oxide (SiO_x) and aluminum oxide (AlO_x) coating on polymers. The biaxial film stretching process improves and increases the mechanical and barrier properties of the film, specifically moisture vapor transmission rate (DeMeuse, 2011). Nylon films provide good puncture resistance, impact strength, temperature resistance, and gas barrier properties (Han, 2014). One difference between Film B and C is their thickness. Film B and C have a gauge of 5.5 and 6.5 mils, respectively. Films with a smaller gauge or thickness will be less effective at stopping the migration of oxygen and contaminants that can lead to spoilage and oxidation. Film C also had an active oxygen barrier. Active oxygen barrier films contain oxygen scavenging components that scavenge any oxygen migrating from the inside of outside of the pouch. The use of oxygen scavenging materials for food packaging have been driven by the wide range of

mechanisms by which oxygen can contribute to quality degradation (Ebnesajjad, 2013). The structures of Film A and C provided these films with a better barrier to oxygen than Film B, resulting in a greater retention of color.



Figure 4.24. Changes in L*values for all process conditions for Film A.



Figure 4.25. Changes in L*values for all process conditions for Film B.



Figure 4.26. Changes in L*values for all process conditions for Film C.

	Time					
L*value	(weeks)	0	2	4	6	8
Film A						
	T-1	23.247 ^{a1}	23.047 ab13	23.217 ^{a1}	22.897 ^{b1}	22.968 ^{b1}
	T-2	23.348 ^{a1}	23.222 ^{ab12}	23.368 ^{a1}	23.005 ^{b1}	23.018 ^{b1}
	T-3	23.248 ^{a1}	22.975 ^{b3}	22.438 d3	22.583 ^{cd2}	22.697 ^{c2}
	T-4	23.222 ^{a1}	23.292 ^{a1}	22.975 ^{b1}	22.380 ^{d2}	22.628 ^{c2}
Film B						
	T-1	23.172 ^{a2}	22.195 ^{b2}	21.643 ^{c3}	21.210 ^{d2}	20.905 ^{e3}
	T-2	23.495 ^{a1}	22.408 b12	22.012 ^{c12}	21.465 ^{d1}	21.193 e12
	T-3	23.383 a12	22.3933 ^{b12}	21.822 ^{c13}	21.328 d12	21.078 e23
	T-4	23.262 ^{a2}	22.427 ^{b1}	22.173 ^{c1}	21.528 d1	21.348 e1
Film C						
	T-1	23.387 ^{a12}	22.872 ^{c2}	23.205 ab13	23.035 bc2	23.117 ^{b1}
	T-2	23.527 ^{a1}	23.258 ^{b1}	23.393 ab12	23.357 ^{ab1}	23.012 ^{c1}
	T-3	23.122 b3	23.010 b2	23.473 ^{a1}	23.140 b12	22.968 b1
	T-4	23.253 ^{a23}	23.087 ab12	23.138 ab3	23.122 ab2	23.010 ^{b1}

Table 4.8. L* values over 8 weeks exposure at 35±2°C and 50±2% RH.



Figure 4.27. Changes in a*values for all process conditions for Film A.



Figure 4.28. Changes in a*values for all process conditions for Film B.



Figure 4.29. Changes in a*values for all process conditions for Film C.

	Time					
a*value	(weeks)	0	2	4	6	8
Film A						
	P-1	20.477 ^{c1}	21.677 ^{a12}	21.153 ^{b1}	20.478 ^{c1}	20.575 ^{c1}
	P-2	21.013 ^{b1}	21.923 ^{a1}	21.158 ^{b1}	20.730 ^{b1}	20.685 ^{b1}
	P-3	20.878 ^{b1}	21.502 ^{a2}	19.417 ^{d3}	19.818 ^{cd2}	20.128 ^{c2}
	P-4	20.818 ^{b1}	22.135 a1	20.107 ^{c2}	19.717 ^{c2}	19.912 ^{c2}
Film B						
	P-1	21.103 ^{a2}	19.652 ^{b1}	17.770 ^{c2}	$16.822 d^2$	16.303 e ³
	P-2	21.487 ^{a1}	20.102 b1	18.438 ^{c1}	17.377 ^{d2}	16.837 e12
	P-3	21.430 a12	20.115 b1	18.133 c12	17.138 d12	16.563 e13
	P-4	21.118 ^{a2}	20.177 ^{b1}	18.498 ^{c1}	17.533 ^{d1}	17.162 ^{d1}
Film C						
	P-1	21.143 a12	$20.977 \ ^{ab2}$	20.867 abc13	20.417 ^{c1}	20.653 bc1
	P-2	21.570 ^{b1}	22.087 ^{a1}	21.457 ^{b1}	21.470 ^{b1}	20.697 ^{c1}
	P-3	20.692 b3	21.357 ^{a2}	21.440 a12	20.858 ^{b2}	20.553 b1
	P-4	21.148 ab23	21.497 ^{a2}	20.525 ^{c3}	20.808 bc2	20.348 ^{c1}

Table 4.9. a* values over 8 weeks exposure at 35±2°C and 50±2% RH.



Figure 4.30. Changes in b*values for all process conditions for Film A.



Figure 4.31. Changes in b*values for all process conditions for Film B.



Figure 4.32. Changes in b*values for all process conditions for Film C.

	Time					
b*value	(weeks)	0	2	4	6	8
Film A						
	P-1	12.753 ab1	12.868 a12	12.827 ^{a1}	12.622 ^{b1}	12.762 ^{ab1}
	P-2	12.892 ^{ab1}	12.973 ^{a1}	12.913 ^{ab1}	12.662 ^{c1}	12.761 bc1
	P-3	12.810 ^{a1}	12.750 ^{a2}	12.135 ^{c3}	12.345 ^{b2}	12.508 ^{b2}
	P-4	12.750 ^{b1}	13.017 ^{a1}	12.525 ^{c2}	12.302 ^{d2}	12.433 ^{cd2}
Film B						
	P-1	12.850 ^{a2}	12.147 ^{b1}	11.555 ^{c2}	11.207 ^{d2}	11.077 ^{d3}
	P-2	13.068 a1	12.308 b1	11.807 ^{c1}	11.430 d1	11.313 d12
	P-3	12.993 a12	12.310 ^{b1}	11.658 ^{c12}	11.337 d12	11.173 ^{d23}
	P-4	12.842 ^{a2}	12.277 ^{b1}	11.792 ^{c1}	11.468 ^{d1}	11.382 ^{d1}
Film C						
	P-1	12.948 a12	12.653 ^{b2}	12.753 b13	12.630 ^{b2}	12.800 ^{ab1}
	P-2	13.075 ^{a1}	13.013 ^{a1}	12.973 ^{a1}	12.985 ^{a1}	12.770 ^{a1}
	P-3	12.693 b3	12.743 ^{b2}	12.928 a12	12.742 ^{b2}	12.668 ^{a1}
	P-4	12.820 ^{a23}	12.772 ^{a2}	12.653 ^{a3}	12.725 ^{a2}	12.663 ^{a1}

Table 4.10. b* values over 8 weeks exposure at 35±2°C and 50±2% RH.



Figure 4.33. Changes in TPS for all process conditions for Film A.



Figure 4.34. Changes in TPS for all process conditions for Film B.



Figure 4.35. Changes in TPS for all process conditions for Film C.

	Time					
TPS	(weeks)	0	2	4	6	8
Film A						
	P-1	40.457 ^{b1}	41.732 a12	41.192 ^{a1}	40.392 b1	40.532 b12
	P-2	41.060 bc1	42.037 a12	41.313 ^{b1}	40.683 ^{c1}	40.383 ^{c1}
	P-3	40.898 ^{b1}	41.540 ^{a2}	38.735 ^{d3}	39.517 ^{c2}	39.957 ^{c2}
	P-4	40.818 ^{b1}	42.267 ^{a1}	39.908 cd2	39.322 ^{d2}	39.980 ^{c2}
Film B						
	P-1	41.125 ^{a1}	39.167 ^{b2}	36.335 ^{c2}	34.647 d ³	33.800 ^{e3}
	P-2	41.598 ^{a1}	39.798 ^{b1}	37.445 ^{c1}	35.685 d12	34.885 e12
	P-3	41.507 ^{a1}	39.812 b1	36.898 c12	35.250 d23	34.293 e ²³
	P-4	41.1483 ^{a1}	39.853 ^{b1}	37.488 ^{c1}	35.930 ^{d1}	35.363 ^{d1}
Film C						
	P-1	41.1983 a12	40.903 ab3	40.865 ab2	40.333 ^{b2}	40.655 ab1
	P-2	41.6767 ^{ab1}	42.213 a1	41.548 ^{b1}	41.555 ^{b1}	40.687 ^{c1}
	P-3	40.6733 ^{c2}	41.345 ab23	41.522 a1	40.848 bc2	40.448 ^{c1}
	P-4	41.1867 ab12	41.540 ^{a2}	40.467 ^{c2}	40.800 bc2	40.273 ^{c1}

Table 4.11. Tomato Paste Score (TPS) over 8 weeks exposure at 35±2°C and 50±2% RH.



Figure 4.36. Changes in tomato a/b ratio for all process conditions for Film A.



Figure 4.37. Changes in tomato a/b ratio for all process conditions for Film B.



Figure 4.38. Changes in tomato a/b ratio for all process conditions for Film C.

	Time					
a/b Ratio	(weeks)	0	2	4	6	8
Film A						
	P-1	1.606 ^{c2}	1.761 ^{a1}	1.649 ^{b1}	1.622 ^{c12}	1.612 ^{c1}
	P-2	1.630 ^{b12}	1.689 ^{a2}	1.644 ^{b1}	1.637 ^{b1}	1.621 ^{b1}
	P-3	1.629 ^{b12}	1.686 ^{a2}	1.598 ^{c2}	1.605 bc2	1.609 bc1
	P-4	1.633 ^{b1}	1.700 ^{a2}	1.605 ^{c2}	1.601 ^{c2}	1.604 ^{c1}
Film B						
	P-1	1.637 ^{a1}	1.618 ^{a1}	1.528 ^{b2}	1.501 ^{c2}	1.472 ^{d2}
	P-2	1.648 ^{a1}	1.633 ^{a1}	1.562 ^{b1}	1.520 ^{c12}	1.488 ^{d12}
	P-3	1.647 ^{a1}	1.634 ^{a1}	1.555 ^{b1}	1.512 ^{c12}	$1.482 d^{12}$
	P-4	1.645 ^{a1}	1.643 ^{a1}	1.569 ^{b1}	1.529 ^{c1}	1.508 ^{d1}
Film C						
	P-1	1.628 ^{b1}	1.657 ^{a2}	1.636 ab12	1.617 ^{b2}	1.613 ^{b1}
	P-2	1.651 ^{b1}	1.697 ^{a1}	1.654 ^{b1}	1.653 ^{b1}	1.621 ^{c1}
	P-3	1.629 ^{c1}	$1.675 a^{12}$	1.658 ab1	1.637 bc12	1.621 ^{c1}
	P-4	1.648 ^{b1}	1.683 ^{a12}	1.622 ^{cd1}	1.638 bc12	$1.607 d^{1}$

Table 4.12. Tomato a/b Ratio over 8 weeks exposure at 35±2°C and 50±2% RH.

	Proce	SS				
		L*	a*	b*	TPS	a/b
Film 1						
	P-1	-0.035	0.012	0.001	0.009	0.001
	P-2	-0.041	-0.041	-0.016	-0.085	-0.011
	P-3	-0.069	-0.094	-0.038	-0.118	-0.003
	P-4	-0.074	-0.113	-0.040	-0.105	-0.004
Film 2						
	P-1	-0.283	-0.600	-0.222	-0.916	-0.021
	P-2	-0.288	-0.581	-0.219	-0.839	-0.105
	P-3	-0.288	-0.608	-0.227	-0.902	-0.020
	P-4	-0.239	-0.495	-0.182	-0.723	-0.017
Film 3						
	P-1	-0.034	-0.061	-0.018	-0.068	-0.002
	P-2	-0.064	-0.109	-0.038	-0.124	-0.015
	P-3	-0.019	-0.017	-0.003	-0.028	-0.001
	P-4	-0.030	-0.100	-0.020	-0.114	-0.005

Table 4.13. Rates of change for food simulant over 8 weeks exposure at $35\pm2^{\circ}$ C and $50\pm2\%$ RH.

CONCLUSION

The results from this study indicate film barrier properties can significantly affect the degradation of color. Food simulant packaged in Film A and C pouches showed greater color retention during accelerated storage than Film B. Film A and C showed similar quality degradation with no significant difference in TPS across all processing treatments (p<0.05). Films A and C provided a better barrier to oxygen than Film B. The color change observed in Film B was likely due to oxygen permeation into the film during storage, thus proving barrier to be a significant factor. After processing, there were no significant differences between processing treatments for TPS means for Films A and B (p>0.05). There were no significant differences in TPS means between treatment type for Film C at the end of storage (p>0.05). Based on this study it can be concluded that type of barrier plays a significant role in maintaining color quality for tomato based products.
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CHAPTER FIVE

RESEARCH CONCLUSIONS

The present research was focused on the thermal pasteurization process technique of hot fill and hold. The temperature variability of industrial sized pouches was identified and temperature differences throughout a hot fill and hold process were evaluated using a low viscosity food simulant. Initially, the process was applied using water to hot fill pouches. The corners of the pouch were found to be the fastest cooling spot within the pouch (p<0.05). The center of the pouch was found to have the highest mean temperature during the hold step of the process and had the slowest cooling rate in the pouch (p<0.05). When the process was applied to a low viscosity food simulant these temperature differences were greater and agreed with the findings that the corners of the pouch were the fastest cooling spot, while the center retained heat longer and was the slowest cooling spot. An additional processing step was applied to implement 180° rotation every 10 seconds during the hot fill and hold process with the food simulant. The time and temperature profiles for a static hot fill and hot process were compared to process with the additional rotation. For the static hot fill and hold process, mean temperatures of the center and corners of a pouch showed non-uniform heat transfer during the holding period and cooling process (p<0.05). More uniform heating and cooling within pouches was achieved by implementing 180° rotation during processing.

These findings were further investigated by evaluating the effect of hot fill and hold process time and temperature differences between the corners and the center of a pouch (for both static and rotation methods) on the color quality of the tomato based food

98

simulant. These pasteurization treatments were also applied using different multilayer packaging materials to study the effect of barrier properties on the color retention during accelerated storage. In high barrier films, there were no significant differences found after processing between different processing treatments in terms of TPS (p>0.05). The type of barrier had a greater impact on the color quality of tomato paste than the different process combinations of time and temperature.

APPENDICES

Appendix A

Temperature Profiles for Pouches



Figure A-1: Temperature profile at 4 minutes at the end of the holding step.



Figure A-2: Temperature profile at 10 minutes during the cooling step.



Figure A-3: Temperature profile at 15 minutes during the cooling step.



Figure A-3: Temperature profile at the end of processing (20 minutes).

Appendix B

Raw Data from Research Experiment

	Spindle			Viscosity	Temp
Run	#	RPM	Output	(cP)	(°C)
Before					
Process	1	30	90	180	21.1
Before					
Process	1	30	91	182	21.7
Before					
Process	1	30	90	180	21.1
Before					
Process	1	30	89	178	21.7
Before					
Process	1	30	90	180	20.8
1-Static	1	30	90	180	21.0
2-Static	1	30	89	178	22.0
3-Static	1	30	92	184	21.6
4-Static	1	30	94	188	21.9
5-Static	1	30	91	182	22.3
6-Static	1	30	92	184	22.5
7-Static	1	30	90	180	21.1
8-Static	1	30	93	186	20.8
9-Static	1	30	92	184	21.2
10-Static	1	30	92	184	20.5
1-Rotation	1	30	89	178	22.3
2-Rotation	1	30	90	180	21.8
3-Rotation	1	30	90	180	21.2
5-Rotation	1	30	91	182	22.6
6-Rotation	1	30	93	186	23.1
7-Rotation	1	30	90	180	21.9
8-Rotation	1	30	92	184	22.3
9-Rotation	1	30	94	188	22.0
10-Rotation	1	30	92	184	21.8

Table B-1: Raw Data for Viscosities of Tomato Paste and Water Mixtures.

Process		Run								
Туре	Run 1	2	3	4	5	6	7	8	9	10
Water	29	10	14	23	25	18	30	28	24	17
Tomato										
Paste-Static	27	21	33	19	22	18	15	30	28	24
Tomato										
Paste-										
Rotation	16	12	32	22	17	31	19	16	14	18

Table B-2: Raw Data for Residual Air Values (mL) for Pasteurized Tomato Paste and Water Mixtures.

Table B-3: Raw Data for Color Values for Undiluted Tomato Paste.

Sample	L*	a*	b *	TPS	a/b ratio
1	21.52	23.51	11.71	42.73	2.007
2	21.57	23.95	11.95	43.53	2.004
3	20.98	24.28	11.93	43.86	2.035

Table B-4: Raw Data for Color Values for Pre-Processed Tomato Paste and Water Mixtures.

Batch	L*	a*	b *	TPS	a/b ratio	
1	23.247	20.477	12.753	40.457	1.606	
1	23.193	21.100	12.873	41.153	1.639	
1	23.363	21.167	12.987	41.233	1.630	
2	23.348	21.013	12.892	41.060	1.630	
2	23.510	21.487	13.063	41.610	1.648	
2	23.533	21.577	13.077	41.673	1.650	
3	23.248	20.878	12.810	40.898	1.630	
3	23.387	21.403	12.997	41.487	1.647	
3	23.117	20.700	12.703	40.680	1.629	
4	23.222	20.818	12.750	40.818	1.633	
4	23.273	21.127	12.840	41.173	1.645	
4	23.257	21.153	12.830	41.203	1.649	