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The Effect of Retort Processing Factors on the Severity of Film Surface Impression

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THE EFFECT OF RETORT PROCESSING FACTORS ON THE SEVERITY OF FILM SURFACE IMPRESSION

A Thesis
Presented to
the Graduate School of
Clemson University

In Partial Fulfillment
of the Requirements for the Degree
Master of Science
Packaging Science

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by
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Accepted by:
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ABSTRACT

Retort processing conditions of temperature, overpressure, and sterilization time were used to determine the impression depth on the pouch surface from contact with the retort rack during retort processing, also referred to as a waffling defect. Retortable flexible pouches were filled with 1,000 mL of water and processed in a horizontal water spray retort. A confocal laser scanning microscope was used to measure severity of the waffling defects.

Data collected during this study showed that higher temperatures resulted in higher measured impression depth values ($p<0.05$) in all of the tested combinations. However, when the pouches were retorted at different temperatures, different effects of retort overpressure were observed. At a low temperature (111°C), the higher overpressure resulted in higher severity of the waffling defects ($p<0.05$). In contrast, at a high temperature (131°C), the higher overpressure resulted in lower severity of the waffling defects ($p<0.05$). When samples were retorted at different sterilization time settings, there was only one condition where a statistical difference in the impression depth values was observed ($p<0.05$). The longer sterilization time resulted in higher measured impression depth ($p<0.05$) only if the samples were retorted at a high temperature of 131°C with a low overpressure of 26 psig. There was no statistical difference ($p>0.05$) in measured impression depth when samples were retorted at different sterilization time settings in other tested combinations.

The difference in variance of the impression depth values between the samples retorted at different processing factor combinations was also studied. Processing factor combinations with a high temperature of 131°C and a long sterilization time of 60 minutes, at any overpressure setting, resulted in a higher variance of waffling defect severity ($p<0.05$) compared to other combinations.

The relationship between retort processing factors and waffling defect severity was explained using a prediction equation. The lack of fit of the proposed equation was not significant ($p>0.05$), while the hypothesis test (overall F-test) showed that the overall model was statistically significant ($p<0.05$). Therefore, the proposed equation is useful in predicting the average impression depth value when retort temperature, overpressure, and sterilization time are known.

This study showed how retort processing factors affect the severity of waffling defects and proposed a method to predict the waffling defect severity. This information could allow pouch manufacturers to develop retortable pouches and food processors to create new retort processes to reduce waffling.

DEDICATION

This thesis is dedicated to my family and the memory of my beloved father, Dr. Jirasak Panin, who always believed in my ability to be successful in the academic arena. He is gone but his belief in me has made this journey possible. A special feeling of gratitude to my loving mother, Khemaporn Panin, for her endless love, support, and encouragement. I would also like to give gratitude to my sister, Arinwan Panin, who never left my side. I could not have completed this task without them.

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CHAPTER ONE

INTRODUCTION

Changes in lifestyles and diets have resulted in the changing needs of consumers. Food processors must think beyond traditional rigid, glass, or aluminum packaging to drive growth and profitability of their products. This has led to the development of flexible pouches which have beneficial aspects over traditional packaging. For example, flexible pouches allow marketers to highlight product freshness through the use of clear film to allow consumers to view product color and texture. Also, due to its light weight, flexible pouches provide portability that traditional plastic, glass, and metal packages do not offer, creating an opportunity for expanded usage occasions of products. In addition, features such as easy opening, re-sealability, compatibility with microwave ovens, easy storage, and ability to be emptied completely and effortlessly serve the changing needs of consumers and escalate the demand for flexible packaging. For this reason, flexible packaging is beginning to dominate new food product releases, and its demand is continuously growing (Bemis, 2016).

The global flexible packaging market size was valued at USD 221.82 billion in 2016 and is expected to expand at a compound annual growth rate of nearly 4.7% during the period of 2017 to 2022. This growing market has been segmented into food and beverage, pharmaceutical, cosmetics, and others. Food and beverage was the largest application segment, accounting for more than 75% of the global volume in 2014. Therefore, the development direction of flexible pouches is largely based on the requirement of the food industry. Increasing consumer demand for microbiologically safer foods, greater convenience, smaller packages, and longer product shelf-life is forcing the industry to develop new food processing and packaging strategies. One of the challenges facing the packaging industry is to match packaging with food processing methods, for example, in-package sterilization (Grand View Research, 2018).

In-package sterilization requires packages to be capable of withstanding the time and temperature demands of thermal processing cycles such as those required for cans and retortable pouches. A retortable flexible pouch is a laminated food package that not only withstands the required processing conditions, but also provides a reduced thermal processing time compared to that of a can due to its shape and structure. The growing demand of flexible packages for retort processing has led researchers to develop new grades of retortable pouches in order to serve each specific need and provide better quality pouches. Higher quality retortable pouches generally refers to better barrier properties, abuse resistance, sealability, machinability, and consumer appeal (Dixon, 2011; Richardson, 2008).

With the development of new materials and equipment, there are several grades of retortable pouches available in the market today. It is essential to choose the proper grade for each application. Using a retortable pouch that does not match with the food processing technique may lead to product defects that result in a totally unusable, and therefore wasted, product. Retortable pouch defects are classified, according to the severity, as a serious or minor defect. A serious defect provides evidence that the hermetic seal of the container has been either lost or seriously compromised, there is microbial growth in the container’s contents, or the container is unsuitable for distribution and sale as stipulated in food and drug regulations. A minor defect results in an abnormal container characteristic but does not either lead to the potential loss of container integrity or represent a potential public health risk (Canadian Food Inspection Agency, 2002).

An example of a minor defect is waffling, which appears as heavy embossing of the retort tray rack pattern on the surface of the pouch from contact with the racks during retort processing (Canadian Food Inspection Agency, 2002). As an appearance problem, waffling defects may negatively affect consumers' confidence in food product quality and brand image. Therefore, understanding the factors affecting waffling defect severity will allow pouch manufacturers to reduce customer complaints and allow food processors to reduce production defects. In this study, the severity of waffling defects was determined by measuring impression depth on the pouch surface. Different retort process conditions were used to compare the results.

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CHAPTER TWO

REVIEW OF LITERATURE

Thermal Processing of Food

One common food preservation method used in the food industry is thermal processing, which uses heat for a specific length of time to kill microorganisms present in the food. To preserve and prevent recontamination of processed food, products are packed into a container and hermetically sealed prior to thermal processing. (Black & Barach, 2015). Generally, the goal of thermal processing is to reduce or destroy microbial and enzymatic activity as well as producing physical or chemical changes to food to achieve a certain quality standard (Safefood 360, 2014).

Thermally processed food that meets sterility requirements can be safely stored at room temperature for the remainder of its shelf life. Food products are never completely sterilized, they are only rendered a condition of commercial sterility (Kumar & Sandeep, 2014). "'Commercially sterility' or, as it is sometimes known as 'shelf stability', means rendering the product free of viable microorganisms of public health significance as well as those capable of reproducing in the food under normal non-refrigerated conditions of storage and distribution." (Black & Barach, 2015). Most commercially sterile canned and bottled food products have a shelf life of at least two years and lose shelf life quality after longer periods due to texture or flavor changes rather than microbial growth (Potter & Hotchkiss, 1995). To determine if a processed food meets commercially sterility requirements, the water activity (a_w), acidity (pH), and preservation method of the product are essential pieces of information.

Water activity (a_w) is a measure of the total amount of water available in a food. When substances such as sugar and salt are dissolved in water, they reduce the amount of unattached water molecules and thus reduce the amount of water available for microbial growth. Since most bacteria, yeasts, and molds can grow above a water activity of 0.95, using a water activity of 0.85 as a cut-off to determine whether a thermal processing method is required provides a large margin on safety. In cases where the water activity of a food product is higher than 0.85, the known acidity is necessary to select the proper thermal processing method (Black & Barach, 2015).

The degree of acidity or pH of a food influences the types of microorganisms able to grow in it. A main concern of food processors is to control the growth of *Clostridium botulinum*, which is a bacterium that can produce a deadly toxin and is able to grow in the absence of oxygen, also referred to as anaerobic conditions. These conditions typically occur in hermetically sealed packages. Scientific investigation has determined that the spores of *C. botulinum* will not germinate and grow in food below a pH of 4.8. Therefore, a pH of 4.6 has been selected as the dividing line between acid and low-acid foods. Even though spores of *C. botulinum* and other spoilage types can be found in both acid and low-acid foods, the growth and toxin formation will not occur in acid foods (Black & Barach, 2015). Figure 2.1 depicts the relationship between different food classifications based on their a_w and pH, and the required preservation method to produce a shelf stable product.

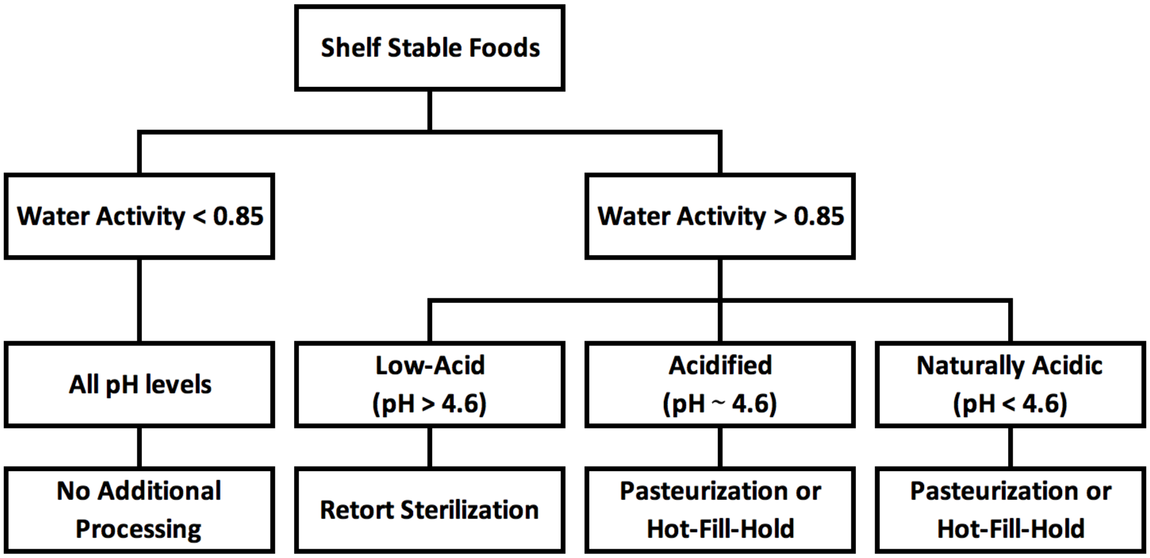

Figure 2.1. Relationship between a_w, pH, and preservation methods (Ward, 2012).

Thermal processing methods used in the food industry include pasteurization, hot-fill-hold, and sterilization (Kumar & Sandeep, 2014). Pasteurization is a relatively mild heat treatment, compared to sterilization, in which the food is heated to below 100°C to destroy enzymes and relatively heat sensitive microorganisms (Safefood 360, 2014). The heat treatment in pasteurization alone is insufficient to inactivate all spoilage causing vegetative microorganisms or heat-resistant spores. As a result, pasteurization is suitable to produce a shelf stable product when the food has a pH of 4.6 or below (acidified or naturally acidic foods). The shelf life of pasteurized low-acid foods, such as milk and some other dairy products, is approximately 2-3 weeks under refrigerated conditions (Kumar & Sandeep, 2014). Pasteurization of unpackaged, low viscosity liquids such as milk and fruit juices is usually carried out using continuous tubular or plate heat exchangers. These liquids are heated and then cooled before filling into containers followed by sealing to prevent recontamination (Robertson, 1992). Some liquid foods such as beer and fruit juices are pasteurized after filling into containers, which is known as in-package pasteurization that uses steam-air mixtures or hot water as a heating medium (Safefood 360, 2014).

Another relatively mild heat treatment used for acidified or naturally acidic foods is the hot-fill-hold process. This process requires the food product to be heated prior to filling, then filled hot, sealed in a container, and held for a given time period at a given temperature prior to cooling. The heat from the hot filled product should be enough to heat the container to destroy microorganisms (Black & Barach, 2015).

Unlike pasteurization and hot-fill-hold process, where the survival of heat resistant non-pathogenic microorganisms is acceptable, a more severe heat treatment called sterilization is used to inactivate most heat resistant microorganisms (Safefood 360, 2014). Sterilization temperatures above 100°C, usually ranging from 110°C to 121°C depending on the type of product, must be reached inside the product and then kept for a defined period of time (Safefood 360, 2014). Since sterilization is a severe heat treatment, it is a required process for low-acid foods in order to achieve commercially sterile conditions. Commercial sterility of low-acid foods can be achieved by in-flow sterilization or in-package sterilization. In-flow sterilization is generally referred to as an aseptic process, whereas in-package sterilization is referred to as a retort process (Kumar & Sandeep, 2014).

The concept of an aseptic process is to bring together a commercially sterile food product and a commercially sterile container, which are both sterilized separately and hermetically sealed in a sterile environment to produce a commercially sterile finished product (Black & Barach, 2015). A food product, such as fruit juice or soup, is heated and held for a specified period of time in a holding tube before it is cooled and then packaged in a sterile container. Since the food product is heated outside of the package, high temperatures for only a short period of time used in an aseptic process yield a high-quality product in terms of nutrients, flavor, color, or texture, compared to that obtained by conventional canning (Kumar & Sandeep, 2014). However, the limitations of an aseptic process generally cited are a large capital investment, applicability to a limited range of products, requirement of a relative homogenicity of the fluid, and a need for sophisticated instrumentation (Featherstone, 2015a).

Another process that is also capable of achieving commercial sterility, where food products are filled into containers prior to sterilization, is retort processing. Conventional retorting involves filling of the product into retortable containers and hermetically sealing them followed by heating, holding, and cooling them in a retort vessel (Kumar & Sandeep, 2014). In order to reach temperatures above 100°C, the heat treatment has to be performed under pressure within the retort vessel, also known as an autoclave or pressure cooker (Safefood 360, 2014). Since a retort process can be used with a variety of food products and containers, it requires a wide range of processing techniques, retort designs, and operating procedures (Rahman, 2007).

Retort Process

There are many different retort systems used to process commercially sterile foods that are prepackaged in hermetically sealed containers. Common characteristics of modern retort systems require pressurization of the system in order to reach higher temperatures than boiling water and the use of a heating medium such as saturated steam, steam-air mixtures, and water to transfer heat to the product (Black & Barach, 2015).

In addition to enabling steam to reach higher temperatures, pressurization of retort systems is typically used to prevent a container from bursting outward due to internal pressure buildup. This is also known as overpressure and allows for processing of a wide variety of containers, including glass, rigid plastics, and flexible pouches (Kumar & Sandeep, 2014). In fact, it is possible to process pouches in saturated steam without using overpressure during the heating cycle and holding cycle, also referred to as the sterilization step, as long as there are good controls of pressure variations within the retort and of container headspace to minimize air expansion within the containers. However, overpressure is generally required during the cooling cycle since the most critical container differential pressure occurs at the start of the cooling cycle (Featherstone, 2015b).

From a food safety point of view, it would be ideal to retort using an intensive heat treatment to eliminate the risk of any surviving microorganisms. However, most food products cannot be exposed to such intensive heat without suffering degradation of their sensory quality or loss of nutritional value. In order to comply with both aspects, an optimum process has to be established to keep the heat sterilization intensive enough for the products to meet a condition of commercial sterility and as moderate as possible for product quality reasons (Safefood 360, 2014).

In addition to heat, numerous publications have cited the superior product quality that can be obtained through decreased process time. Proposed practices are, however, to adopt higher temperatures and consequently a shorter process time to maximize organoleptic and nutrient retention within the food product (Rahman, 2007). A shorter process time followed by prompt and rapid cooling not only protects food product quality but also shortens total time required for each processing cycle and results in efficient retort use (Featherstone, 2015).

Retorts can be operated in either batch or continuous mode. A batch retort is considered as a versatile sterilization system due to its ability to handle different food products and package types. Specifically, a batch retort could be distinguished into still (or static) and agitating (rotary or oscillating) retort. Still retorts use a non-agitating pressure vessel for food processing, while rotary and oscillating retorts provide product agitation during processing which promotes faster heating. In addition, batch retorts can further be classified as shown in Figure 2.2 (Kumar & Sandeep, 2014; Stowe, 2015).

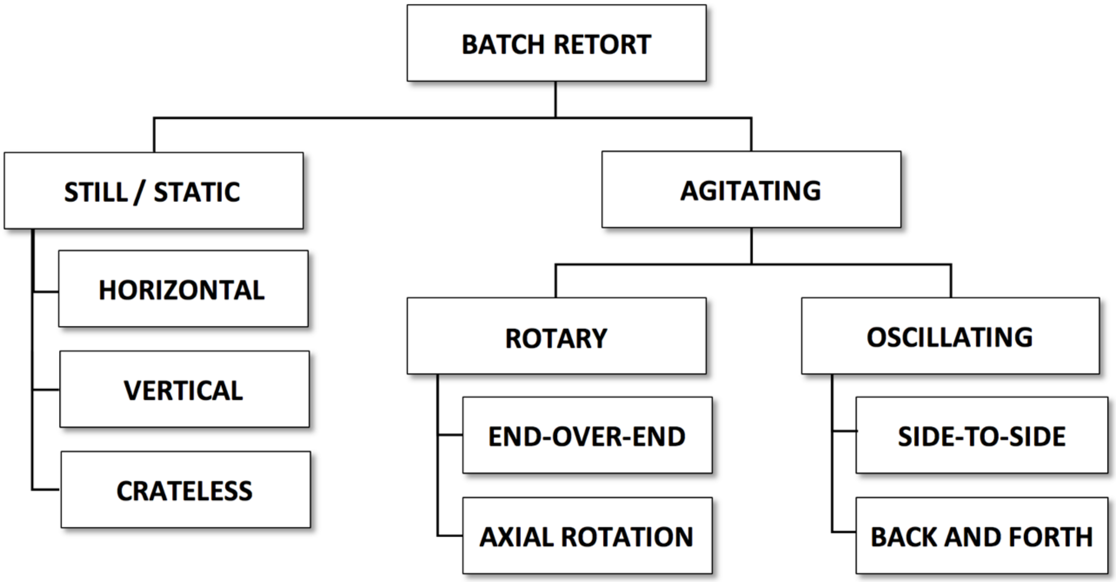

Figure 2.2. Types of batch retort.

For still retorts, containers can be loaded into or unloaded from the pressure vessel using racks, crates, cars, baskets, or trays. Still retorts that are loaded with crates next to each other are called horizontal retorts, while still retorts that are loaded with the crates stacked on top of each other are called vertical retorts. However, there are some models of still retorts that can be operated without using container support systems, which are referred to as crateless retorts (Black & Barach, 2015).

Obviously, a batch-operated thermal process is not an energy-efficient method of processing because of the energy wasted in heating and cooling the retort for every batch of food products. Continuous food processing is a more preferable method where high-volume products are being processed (Featherstone, 2015). Continuous retorts, in addition to improving energy efficiency, help increase throughput and lower manpower cost. Continuous retorts can be classified into hydrostatic (or static) or rotary (or agitating) retorts (Kumar & Sandeep, 2014).

Thermal processing in hydrostatic retorts occurs in a processing chamber that is maintained at an elevated temperature and pressure. Since there are no doors or valves sealing off the processing chamber from the atmosphere, the pressure is maintained and counterbalanced by the weight of water in the infeed and discharge legs. Therefore, a maximum process temperature of hydrostatic retorts is limited by the maximum height of the water legs (Black & Barach, 2015). Figure 2.3 provides an illustration of the operating principle of a hydrostatic retort.

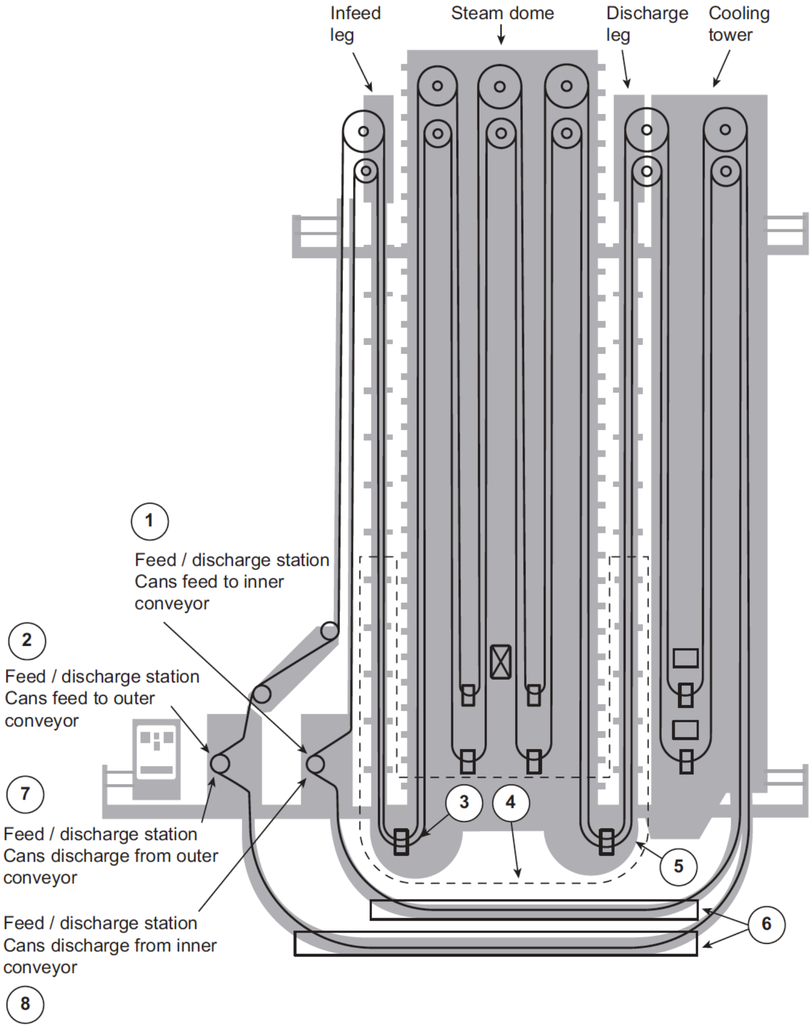

Figure 2.3. Operating principle of a hydrostatic retort (Featherstone, 2015).

Unlike a hydrostatic retort where extensive use of vertical space is required, continuous rotary retorts provide product agitation and container handling using a spiral track on the inside circumference along the length of the retort's horizontal cylindrical chamber, like an auger as shown in Figure 2.4 (Black & Barach, 2015). In spite of the benefits of continuous rotary retorts, this system typically requires a cylindrical container with limited variation in diameter and height for each application. This is due to its lack of flexibility in container handling using a spiral track (Sun, 2012).

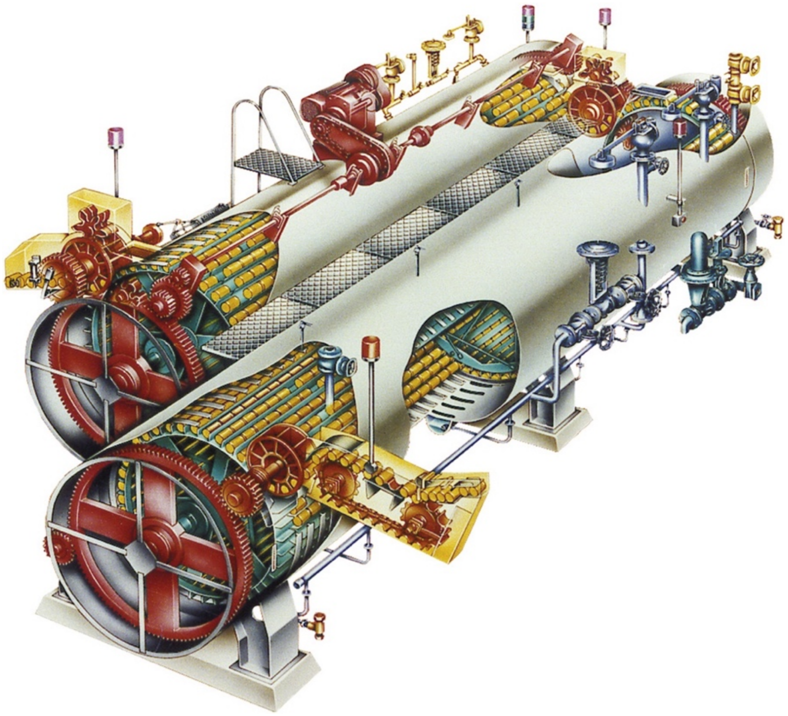

Figure 2.4. Cut-away view of continuous rotary retort (JBT, 2018).

In addition to the design of product handling and agitation method, another classification system of a retort process involves the type of the heating medium used. Steam is the most common heating medium for thermal processing. Even when water is used as the heating medium, steam is generally used to heat the water (Black & Barach, 2015). There are four steam-based processes typically used in sterilizing food products: saturated steam, water immersion, water spray (including cascading water), and steam-air process. Since there is no retort process that fits all the applications, it is necessary to choose the optimum retort process for a particular product and container (Williams, 2018).

Saturated steam retorts use pure saturated steam to directly heat the containers. Pure saturated steam is steam free of air, other non-condensable gases, and condensable volatile materials other than steam, excess condensate, or solutes (Featherstone, 2015).

When saturated steam condenses on the outside of the container, latent heat is transferred to the food product. If there is air trapped inside the retort, it forms an insulting boundary film around the containers which prevents the steam from condensing and causes underprocessing of the food. This trapped air also produces a lower temperature in the retort than that obtained with pure saturated steam. Therefore, it is important to remove all air from the retort using incoming steam, which is referred to as venting (Rahman, 2007). This venting procedure leads the saturated steam retort to be one of the systems that use a lot of steam in processing (Williams, 2018).

Though saturated steam retorts are not energy-efficient, they have the ability to process most canned products. Also, this process requires a low capital investment, since overpressure does not need to be used unless containers other than cans are being processed. If overpressure is used in saturated steam retorts, it can only be used in the cooling step since air is not permitted to enter the vessel at any time during sterilization step (Williams, 2018). When the sterilization step is completed, containers are cooled with water, which causes rapid temperature reduction in the retort. At this point, food cools more slowly than the interior of the retort and the pressure within the containers remain high. An overpressure is then required if fragile containers such as glass, rigid plastics, and flexible pouches are being processed in order to prevent internal pressure from bursting the containers (Kumar & Sandeep, 2014; Rahman, 2007).

The water immersion retort is similar to a saturated steam retort in that the product is totally isolated from any influence of cooling air since the product in a water immersion retort is totally submerged in water. Unlike saturated steam retorts where air cannot be introduced into the vessel during sterilization, the water immersion process uses overpressure on top of the water to pressurize the process load, which allows the retort to be able to handle most fragile containers (Williams, 2018). However, the rate of heat transfer from water to product is significantly slower than from steam to product, which leads to longer total time required for each processing cycle (Featherstone, 2015).

A water spray retort is a retort that uses pressurized hot water as a heating medium. To heat the product, a controlled amount of process water is distributed through spray nozzles located along the top and sides of the retort onto the product (Featherstone, 2015). The water spray retort is also an overpressure system which can be used to process a wide variety of container types like a water immersion retort, but the containers are directly exposed to the influence of overpressure air. This process has become one of the most popular types of retorts due to its high flexibility and low equipment cost compared to other types of overpressure retorts. Also, a water spray retort is an energy efficient system when it is configured with a storage tank since sterilizing and cooling water can be reused without chemical treatment for the next process (Williams, 2018).

A cascading water retort is similar to a water spray retort, in which a controlled amount of process water is drawn from the bottom of the retort by a high capacity pump and distributed onto the product. However, a cascading water retort distributes process water through a manifold or a perforated plate in the top of the retort instead of using spray nozzles (Featherstone, 2015).

A steam-air process uses steam as the only heating medium. However, air is introduced together with steam to generate overpressure in the retort vessel in order to enhance its ability to process most types of containers. A large fan is used as a driving force to mix the steam with the air to prevent cold spots in the retort. This fan also helps increase the rate of heat transfer by creating a forced convection which leads to higher process energy efficiency (Williams, 2018).

Food Packaging for Retort Process

The four primary functions of packaging are containment, protection, convenience, and communication. The containment function of packaging provides a major contribution to protecting the environment from the myriad of products that are transported from one place to another. This basic function is not always satisfied as evidenced by the high number of packaged food products that leak their contents, especially around the closures and seals (Robertson, 2013).

Protection is another function of packaging that is often regarded as an essential part of the food preservation process. Packaging is expected to protect its contents from outside environmental influences such as water, water vapor, gases, odors, dust, microorganisms, shocks, vibrations, and compressive forces. Food products only maintain their desired shelf life for as long as the package provides protection. Once the integrity of the package is breached, the product is no longer preserved (Robertson, 2013).

In addition to containment and protection of the products, the two other important functions of packaging are convenience and communication. Since packaging plays an important role in meeting the demands of consumers, packaging of successful products are designed to increase convenience to the consumers such as precooked foods that can be reheated without removing their primary package, products that are portioned by packing into a desirable consumer-size container, and resealable containers that can retain the quality of the product when the package is first opened until completely used. Even with a resealable function, a package should not contain too much product that could deteriorate before being completely consumed by the intended consumers (Robertson, 2013).

Communication is also a function of packaging that helps sell products. Packaging increases the ability of consumers to recognize a product through distinctive shapes, branding, and labeling, which enables supermarkets to function on a self-service basis. Not only is it in the supermarkets where the communication function of packaging is important, but also in warehouses and distribution centers that would become chaotic if secondary and tertiary packages lacked labels or carried incomplete details (Robertson, 2013).

In order to serve these functions effectively, packages are developed with a variety of materials, designs, and techniques for applications. In thermal processing of prepackaged foods, certain types of containers are used to prevent recontamination which are typically categorized into rigid, semi-rigid, or flexible containers. Rigid containers such as metal cans and glass containers retain their shape when filled and sealed and are neither affected by the enclosed product nor deformed by external pressure of up to 10 pounds per square inch (psi) or 0.7 kilogram per square centimeter (kg/cm^2) (Black & Barach, 2015).

Metal materials like tinplate and aluminum are used in the manufacture of rigid containers for foods and beverages due to their mechanical strength, low toxicity, ability to withstand wide extremes of temperature as well as their superior barrier properties to gases, moisture, and light (Robertson, 2013). Cylindrical cans made of tin-plated steel have been used in food preservation for a long time, although lacquered tin-free steels which combine the physical strength and relatively low price of steel with the corrosion resistance of coating are gradually replacing them. In addition to food preservation, metal containers like aluminum cans and thin steel cans with easy open ends are commonly used for beverage packing (Holdsworth & Simpson, 2007).

Glass containers are also widely used for packing foods and beverages due to their advantage of having low interaction with the contents and visibility of the product. However, glass containers typically require careful processing and handling. For example, it is necessary to use the correct overpressure during retorting to prevent the lid from being distorted. Also, it is essential to preheat the glass jars prior to processing in order to prevent temperature shock breakage (Holdsworth & Simpson, 2007).

Another type of container that can be used in thermal processing of prepackaged foods are semi-rigid containers such as plastic cups and trays. When filled and sealed, their shape is not significantly affected by the enclosed product under normal atmospheric temperature and pressure but can be deformed by an external pressure of less than 10 pounds per square inch (psi) or 0.7 kilogram per square centimeter (kg/cm^2) (Black & Barach, 2015). The main requirement for a plastic material used in thermal processing is its ability to withstand the rigors of heating and cooling processes. As in glass container processing, it is necessary for semi-rigid plastic container processing to control the overpressure correctly to maintain a balance between the internal pressure developed during processing and the pressure of the retort chamber (Holdsworth & Simpson, 2007).

Common semi-rigid containers used in retort processing are plastic containers such as cups and trays with heat sealed lids. The container body can be a single or multi-layer composition of polymers, which may be formed through either a blow molding or thermoforming method. The lid material which is designed to be sealed efficiently and allows easy removal by the consumer is typically constructed of multiple layers of polymers and may require an aluminum foil layer in order to increase gas and moisture barrier properties. In addition to semi-rigid plastic containers, paperboard packages are also used in a retort processing. The basic construction of paperboard packages is a multilayer laminate of polymers, paperboard, and often aluminum foil. Other than this basic structure, some paperboard packages have resealable pour spouts for consumer convenience (Black & Barach, 2015).

In order to reduce packaging weight and space required in storage and transportation, which can result in reduced shipping and warehousing costs, flexible containers have been developed and replaced some applications of the traditional rigid packaging in the food industry. The flexible containers' shape, when filled and sealed, is significantly affected by the enclosed product. Pouches and bags are common forms of flexible containers which are primarily composed of single or multi-layers of different types of materials such as plastic polymers and barrier materials, for example, aluminum foil and silicon oxide (Black & Barach, 2015).

Retortable Flexible Pouch

In the 1950s, the U.S. Army promoted the concept of retortable flexible pouches which were lightweight, easy-to-pack, and shelf-stable food containers for use in field rations in order to eliminate the heavier, traditional can. Research continued through the 1960s which led retortable flexible pouches becoming a new type of commercial shelf-stable food container. In the late 1960s, retortable flexible pouches were widely accepted and used widespread throughout many countries such as the United States, Italy, and Japan. Today, in terms of food packaging, the retortable flexible pouch is considered to have the most potential as an alternative to the traditional can due to several advantages (Canadian Food Inspection Agency, 2002):

1. The thinner material used to make flexible pouches transfers heat faster than the material used in traditional can manufacturing, which permits reduced heating time in food processing. Lower heating time reduces the risk of overcooking the product, thereby producing better color, firmer texture, and less nutrient loss in the food product. Less heating time not only produces a higher quality food product but also results in energy savings for food processing.
2. Labels can be printed directly into a layer prior to lamination or sandwiching to complete a flexible pouch structure, making it permanent.

3. Flexible pouches are distribution friendly due to their lightweight and ability to be flattened, causing lower transportation costs, less storage space required for pre-filled pouches, and less disposal space required for post-consumer pouches.

Although the use of retortable flexible pouches provides significant benefits, there are also disadvantages that need to be considered (Canadian Food Inspection Agency, 2002):

1. Flexible pouch packing systems often require a higher capital investment for their more complex machinery and have a slower filling speed than conventional can packing systems.
2. Thermal processing of flexible pouches is more complicated than for metal cans since the additional processing parameters, such as residual air in the pouch, pouch thickness, and retort overpressure, must be controlled carefully.
3. Flexible pouches are easily punctured, thereby requiring more careful product handling. They also may require over-wrapping for distribution.

In order to perform its functions properly, the retortable flexible pouch is designed to withstand thermal processing temperatures while also possessing the ability to form and maintain its hermetic seal. Additionally, flexible pouches are often required to meet specific performance needs, such as having barrier to oxygen, moisture, and light; durability to protect the product shelf-life throughout processing, storage, and distribution of the product; and resistance to container-product interaction. These challenging requirements for the retortable flexible pouch are difficult to achieve with discrete materials (Black & Barach, 2015; Holdsworth & Simpson, 2007).

The use of multilayer material in flexible pouch manufacturing creates a single pouch structure that combines the different properties of individual base materials in order to meet design requirements (Dixon, 2011). For example, a flexible pouch based on cast polypropylene (PP) film can be heat sealed to contain a food product and prevent recontamination of that product after thermal processing, but it will stick to the sealing jaw during heat sealing. The addition of an outside layer using a polymer with higher temperature resistance, such as oriented polyamide (PA or Nylon) film provides the ability to heat seal the pouch without the issue of the sealant sticking to the sealing jaw. Table 2.1 compares several properties of a selection of web materials.

Table 2.1. Comparison of properties of web materials (Dixon, 2011).

Material	Thickness*	Tensile Strength	Light Barrier	Heat Sealing	Heat Resistance	Deadfold
Paper	40-70 g/m^2	+++	+	0	++++	++
Aluminum Foil	6.3-12 µm	+	++++	0	++++	++++
Oriented PP film	15-30 µm	+++	0	0	++	+
Metallized oriented PP film	15-20 µm	+++	+++	0	++	+
Oriented PET film	12-19 µm	+++	0	0	+++	+
Metallized oriented PET film	12 µm	+++	+++	0	+++	+
Oriented PA (Nylon) film	12-20 µm	++++	0	0	+++	+
Blown LDPE film	30-70 µm	+	0	++++	+	+
Cast PP film	40-70 µm	++	0	++++	+	+
EVOH layer**	3-10 µm	N/A**	0	0	+	+

*Given thicknesses are used for strength comparison.

**EVOH would not be used on its own; must be supported by other layers.

Note: - The more pluses, the better the material displays that property.

- "0" indicates a complete lack of the property.

To develop a multilayer flexible pouch structure, web materials are chosen for individual layers to achieve specific performance properties required by a target application, which include barrier properties, abuse resistance, sealability, machinability, and consumer appeal. Barrier properties are required to keep moisture, gases, light, flavor, or grease from entering or leaving the package. Barrier properties may be characterized by measuring water vapor and oxygen permeation through the material (Ebnesajjad, 2013). The different barriers provided by commonly used base materials are illustrated by Figure 2.5, which shows their permeabilities to water vapor and oxygen (Dixon, 2011).

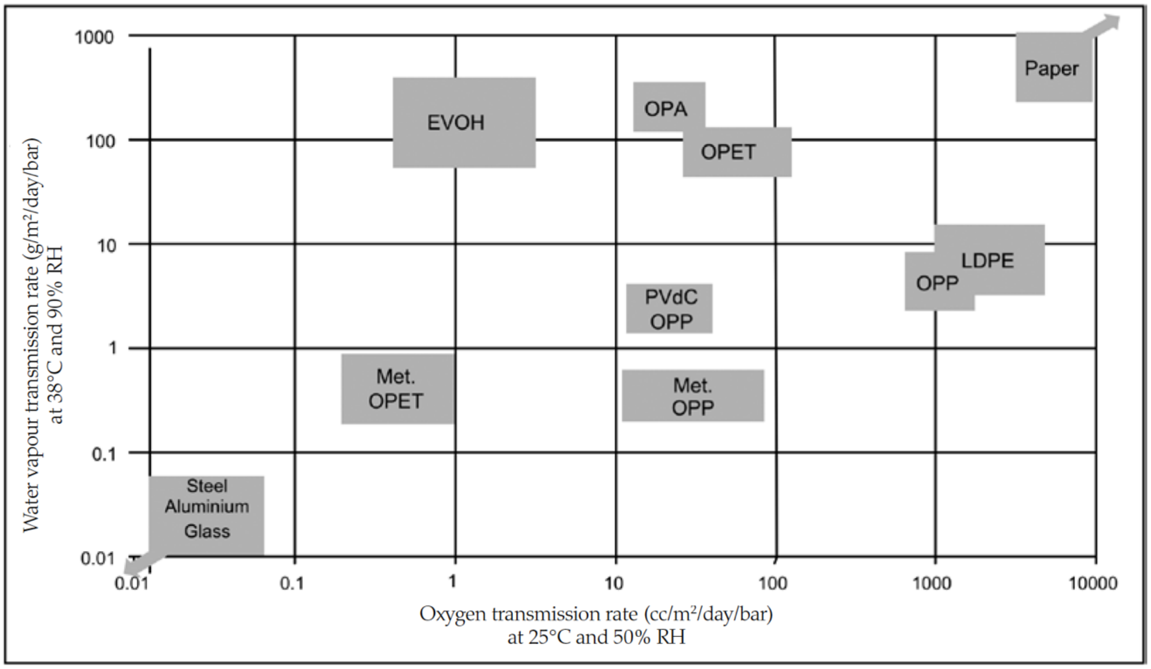

Figure 2.5. Permeabilities to water vapor and oxygen of base materials (Dixon, 2011).

Aluminum foil is one of the most common barriers used in food packaging due to its excellent barrier properties. Nevertheless, aluminum foil is a relatively fragile material, which often results in the degradation of barrier during normal handling of the package. To prevent barrier degradation during package handling, aluminum foil is typically protected by other materials; for example, laminating polyethylene on both sides of the thin aluminum foil. These protecting layers help to cushion aluminum foil against mechanical damage in the form of cracking and pinholes which would reduce barrier effectiveness. In addition to aluminum foil, another form of an aluminum layer in food packaging is metallized films, which have a thin layer of soft aluminum deposited on their surface. However, this thin layer of aluminum can be easily scratched and damaged with a consequent increase in permeability. Therefore, the metallized layer must also be laminated to protect it from mechanical damage and preserve its barrier (Dixon, 2011).

Besides barrier properties, abuse resistance is another property that needs to be considered in flexible pouch structure design to prevent damage to the packaging material and its contents during shipping and storage. Abuse resistance is typically measured through mechanical properties such as puncture resistance, tear strength, impact strength, and modulus, which can be found in film data sheets. However, most published film data sheets are developed from monolayer films which do not account for any interactions between layers, the influence of fabrication variables, or orientation. Some unfavorable interactions lead to interlayer destruction such as when a very ductile layer is adhered to a brittle layer resulting in the film exhibiting the properties of the brittle layer. Additionally, mechanical property data from monolayer film can be misleading since some properties lack a linear relationship between mechanical property and film thickness (Ebnesajjad, 2013).

To enhance sealability, a layer with good sealability is typically coated, laminated, or co-extruded to be used as the innermost layer of the retortable flexible pouch. This allows the package to be made at high speeds and keep the product secure by preventing the package seals from failing. Sealability is commonly characterized by heat seal and hot tack strength, heat seal and hot tack initiation temperatures, seal-through-contamination performance, caulkability, and seal integrity. Hot tack strength refers to the seal strength while the seal is still in the molten state, which is important information for not only when the product drops into the package immediately after sealing, but also for horizontally filled packages involving gussets where the spring-back nature of the folded film creates an opening force. Caulkability refers to the ability of the sealant material to flow in order to fill in gaps around folds, wrinkles, or product contaminants (Ebnesajjad, 2013).

Other than the ability to contain and protect food products by considering barrier properties, abuse resistance, and sealability, flexible packaging is required to provide good machinability. A well-designed flexible package structure with good machinability allows the packaging films to be easily run on high-speed automatic packaging equipment with low scrap rates. Machinability is largely characterized by the film's modulus, thickness, seal properties, and coefficient of friction (Ebnesajjad, 2013).

In the retailers' point of view, flexible pouches may also need to provide some additional performance properties such as eye-catching graphics that help sell the product and the proper physical form for display purposes since package appearance is an important factor driving product preference by consumers. Consumer appeal is commonly related to print quality and package gloss. Additionally, film thickness and modulus may also impact consumer appeal (Ebnesajjad, 2013).

To serve these specific performance properties, reduce cost, and reduce number of processes, multilayer flexible pouches with different structures are developed to be used in retort processing. One common retortable flexible pouch structure is a lamination containing oriented polyethylene terephthalate (PET) film for printability, biaxially oriented polyamide (PA or Nylon) film for toughness, aluminum foil for oxygen barrier, and a polypropylene (PP) sealant film. This type of retortable flexible pouch may contain food items like tuna, pet food, and soup. The food items are held at elevated temperature during retort processing; therefore, the packages must remain intact at elevated temperatures. Besides temperature resistance, toughness, seal strength, and barrier properties are critically important (Ebnesajjad, 2013). These retortable flexible pouches may be either supplied as pre-made pouches or formed from roll-stock using form/fill/seal equipment in the food processing plant. Food processors use pre-made pouches which generally permit an increased line speed over processes that use roll-stock since mechanical issues and steps of converting roll-stock to pouches at the food plant are eliminated (Black & Barach, 2015; Holdsworth & Simpson, 2007).

Food Safety for Retortable Flexible Pouch

In addition to properties required by a target application, such as environmental conditions to which the food product will be exposed during distribution, storage conditions after packing, and compatibility of the package with the thermal processes, food safety criteria must also be considered when developing a retortable flexible pouch for food applications. Food safety criteria are required to ensure the stability of the food product with respect to the deteriorative chemical, biochemical, and microbiological reactions that can occur in a package (Robertson, 2013).

A major food safety concern is the nature and composition of the specific packaging material as well as its potential effect on the quality and safety of the packaged food as a consequence of the migration of components from the packaging material into the food. Components that are transferred to the food as a result of contact or interaction between food product and the package material are often referred to as migrants. In fact, migration is a two-way process, since constituents of the food can also migrate into the packaging material, which is referred to as absorption; for example, the scalping of flavor compounds from fruit juices by plastic containers (Robertson, 2013).

To protect public health by controlling the adulteration of food, U.S. legal requirements are published in the Code of Federal Regulations (CFR). The CFR contains broad areas of regulation including the control of packaging material composition by specifying the amount of additive that can be used and types of polymer to which it can be added. The regulations also contain time-temperature-solvent conditions for short-term migration simulations. The selection of conditions depends on the type of food, the conditions of use, and the thermal treatment applied to the package after filling (Robertson, 2013).

Other than migration-related regulations, standards for the condition of food containers are included in the CFR to ensure that food packages are in a condition that is safe to be used. This condition is defined in the CFR as “the degree of acceptability of the container with respect to freedom from defects which affect the serviceability, including appearance as well as usability, of the container for its intended purpose". In order to make sure that food containers meet this condition, standards for condition of food containers, which include sampling and inspection procedures as well as defect types and classification criteria, shall be applied (Agricultural Marketing Service USDA, 2013).

Defects in Retortable Flexible Pouch

In the CFR, defects of food containers are enumerated and classified according to the degree to which the individual defect affects the serviceability, usability, or appearance of each container type. Food container types mentioned in these standards include metal, composite, glass, plastic, flexible, and unitizing containers as well as other rigid and semi-rigid containers. In order to clarify types of food containers, common examples for each container type are given in the CFR. Examples of flexible containers are plastic, cellophane, paper, textile, and laminated multi-layer pouches. Since retortable flexible pouches are considered a type of flexible containers, understanding flexible containers' defects helps to reduce defects in retortable flexible pouch manufacturing (Agricultural Marketing Service USDA, 2013).

There are 59 defects of flexible containers listed in standards for condition of food containers as shown in Table 2.2. To identify the severity of a defect, commonly used terms are defined as follows (Agricultural Marketing Service USDA, 2013):

1. Critical defect is "a defect that seriously affects, or is likely to seriously affect, the usability of the container for its intended purpose".

2. Major defect is “a defect that materially affects, or is likely to materially affect, the usability of the container for its intended purpose”.
3. Minor defect is “a defect that materially affects the appearance of the container but is not likely to affect the usability of the container for its intended purpose”.
4. Insignificant defect is “a flaw in the container that does not materially affect the appearance and does not affect usability of the container for its intended purpose”. The CFR suggests “when performing examinations, insignificant defects shall not be recorded”.

Table 2.2. Defects of flexible containers (Agricultural Marketing Service USDA, 2013).

Defects	Categories		
	Critical	Major	Minor
Type or size of container or component parts not as specified	None permitted		
Closure not sealed, crimped, stitched, or fitted properly:			
(a) Heat processed primary container	1		
(b) Non-heat processed primary container		101	
(c) Other than primary container			201
Dirty, stained, or smeared container			202
Unmelted gels in plastic			203
Torn or cut container or abrasion (non-leaker):			
(a) Materially affecting appearance but not usability			204
(b) Materially affecting usability		102	
Moldy area	2		
Individual packages sticking together or to shipping case (tear when separated)		103	
Not fully covering product		104	
Wet or damp (excluding ice packs):			
(a) Materially affecting appearance but not usability			205
(b) Materially affecting usability		105	
Over wrap (when required):			
(a) Missing		106	
(b) Loose, not sealed, or closed			206
(c) Improperly applied			207
Sealing tape, strapping, or adhesives (when required):			
(a) Missing		107	
(b) Improperly placed, applied, torn, or wrinkled			208
Tape over bottom and top closures (when required):			
(a) Not covering stitching		108	
(b) Torn (exposing stitching)		109	
(c) Wrinkled (exposing stitching)		110	
(d) Not adhering to bag:			
1. Exposing stitching		111	
2. Not exposing stitching			209
(e) Improper placement			210

Table 2.2. (*Continued*) Defects of flexible containers (Agricultural Marketing Service USDA, 2013).

Defects	**Categories**		
	Critical	**Major**	**Minor**
Product sifting or leaking:			
(a) Non-heat processed		112	
(b) Heat processed	3		
Flexible pop-top:			
(a) Poor seal (wrinkle, entrapped matter, etc.) reducing intact seal to less than 1⁄16-inch	4		
(b) Short pull tab (materially affecting usability)			212
(c) Missing pull tab		113	
(d) Torn pull tab (materially affecting usability)			213
Missing component (straw, etc.)			214
Two part container (poly lined box or bag in box):			
(a) Outer case torn			215
(b) Poly liner:			
1. Missing	5		
2. Improper closure		114	
Missing ‘‘zip lock’’ (re-sealable containers)			216
Loss of vacuum (in vacuum-packed)		115	
Pre-formed containers:			
(a) Dented or crushed area			217
(b) Deformed container			218
Missing re-sealable cap		116	
Inner or outer safety seal—missing, torn, poor seal	6		
Air bubble in plastic		117	
Thermostabilized products (includes but not limited to tubes, pouches, etc.):			
Foldover wrinkle in seal area (thermostabilized pouches):			
(a) Extends through all plies across seal area or reduces seal less than 1⁄16-inch	7		
(b) Does not extend through all plies and effective seal is 1⁄16-inch or greater			219
Incomplete seal (thermostabilized pouches)	8		
Non-bonding seal (thermostabilized pouches)	9		

Table 2.2. (*Continued*) Defects of flexible containers (Agricultural Marketing Service USDA, 2013).

Defects	**Categories**		
	Critical	**Major**	**Minor**
Laminate separation in body of pouch or in seal within 1⁄16-inch of food product edge:			
(a) If food contact layer is exposed	10		
(b) If food contact surface is exposed after manipulation or laminate separation expands after manipulation		118	
(c) If lamination separation is limited to isolated spots that do not propagate with manipulation or is outer ply separation in seal within 1⁄16-inch of food product edge of seal			220
Flex cracks (cracks in foil layer only)			221
Swollen container	11		
Blister (in seal) reducing intact seal to less than 1⁄16-inch	12		
Compressed seal (overheated to bubble or expose inner layer) reducing intact seal to less than 1⁄16-inch	13		
Stringy seal (excessive plastic threads showing at edge of seal area)			222
Contaminated seal (entrapped matter) reducing intact seal to less than 1⁄16-inch	14		
Seal creep (product in pouch ‘‘creeping’’ into seal) reducing intact seal to less than 1⁄16 inch	15		
Misaligned or crooked seal reducing intact seal to less than 1⁄16-inch	16		
Seal formed greater than 1-inch from edge of pouch (unclosed edge flaps)			223
Waffling (embossing on surface from retort racks; not scorable unless severe)			224
Poor or missing tear notch (when required)			225

Waffling Defect

As mentioned previously, a minor defect is a defect that has no adverse effect on the hermetic seal and does not result in the loss of container integrity which would consequently represent a potential public health risk. Effects of minor defects are limited to cosmetic issues, such as stringy seals (the presence of plastic threads emerging at the edges of the cutoff seal), deformed or dented containers, and waffling (embossing caused by racks during thermal processing that appears on the surface of the pouch). These defects may arise in the various stages of container manufacturing and food processing, which include forming, filling, sealing, thermal processing, and handling, before the container reaches the consumer (Canadian Food Inspection Agency, 2002; Heldman, 2003).

Waffling defects are defined by the CFR as “embossing on pouch surface from retort racks”. However, the CFR will consider embossing as a minor defect only when it is severe. As well as the United States CFR, the Flexible Retort Pouch Defects Identification and Classification Manual published by the Canadian Food Inspection Agency (CFIA) provides detailed clarification of retortable pouch defects. The CFIA defined waffling defect as “heavy embossing of the retort tray rack pattern on the surface of the pouch body, from contact with the racks during thermal processing” and considered waffling defect as a minor defect. Additionally, the CFIA claims that waffling defects are caused by pouch expansion against the racks during thermal processing, which leads to a heavy impression on the surface of the pouch material (Agricultural Marketing Service USDA, 2013; Canadian Food Inspection Agency, 2002).

Previous Research on Flexible Pouch Defects

Researchers have continued to study flexible pouch defects in order to improve flexible pouch quality and develop more efficient defect detection as evidenced by several studies found in the literature. After a thorough review of the related literature, it is obvious that researchers mainly focused on critical and major defects which often result in potential public health risks. Since low quality of the seal area is one of the major parts that leads to flexible pouch integrity failure, several published papers related to seal area defects detection are available.

In 1998, Raum et al. presented a non-destructive method to detect channel defects in the seal region of flexible pouches using integrated backscatter ultrasound imaging. It was observed that this method has the ability to detect a 10 μm diameter channel defect (Ruam, Ozguler, Morris, & O'Brien, 1998). In the same year, Ozguler et al., who were co-researchers in the first study, improved an ultrasonic imaging method using a pulse-echo technique. This pulse-echo technique is a non-destructive method to detect not only micro-leaks but also contamination in the seal area of retortable pouches (Ozguler, Morris, & O'Brien, 1998).

In 1999, Ozguler et al. proved that there is a direct relationship between the defect size and image contrast value. They have shown that different defect types and packaging materials have a significant impact on image contrast value. A new technique using image contrast values has been presented as a more reliable sensing method to rapidly detect micro-leaks and contamination in the seal area (Ozguler, Morris, & O'Brien, 1999). In 2001, Ozguler et al. showed that the use of ultrasonic backscattered amplitude integral (BAI) value by itself is not capable of detecting critical and major defects, such as nonbonding, wrinkles, and bubbles distributed within the seal area. The authors presented a new technique using a combination of the mean BAI value and coefficient of variation of the BAI value in detecting defects in seals of flexible pouches. This technique has the potential of providing a real-time, online control by sensing whether a seal without nonbonding, wrinkles, and bubbles has been achieved (Ozguler, Morris, & O'Brien, 2001).

In 2015, D’Huys et al. presented a method to detect seal contamination in heat sealed flexible packaging using active infrared thermography. This method uses the heat of the sealing bars as an excitation source. The cooling profile of contaminated seals was recorded shortly after sealing. High resolution digital images served as a reference to quantify contamination and then the processed thermal images were mapped to these references. In this study, the authors compared six thermal image processing methods based on a single frame, a fit of the cooling profile, thermal signal reconstruction, pulsed phase thermography, principal component thermography, and a matched filter (D'Huys, Saeys, & De Ketelaere, 2016).

In 2016, Morris presented another technique using infrared thermography method called Dynamic Scanning Infrared Thermography (DSIRT). In this study, the samples were placed in relative motion to a laterally positioned infrared laser, inducing heating through the plane of the seal. The emergent thermal artifact on the observed side was sensed using either a bolometer camera or a thermopile sensor with thermal anomalies indicating potential defects (Morris, 2016).

Since waffling defects are considered a minor defect that do not result in potential public health risks, no published research papers related to waffling defect have been found. However, there is a need to understand the effect of retort processing on the severity of the waffling problem, which leads to an opportunity to develop retortable pouches that fit each retort process in order to reduce customer claims on flexible pouch appearance issues. Also, food processors will have the ability to create new retort processes with optimized temperature, overpressure, and sterilization time settings to reduce waffling.

Research Objective

The objective of this research was to determine the relationship between three retort process critical parameters (temperature, overpressure, and sterilization time) and the measured depth of the surface impression (waffle) from contact with the retort rack for a specific grade of retort pouch and retort rack design. Understanding the relationship between these retort parameters and waffling defects will allow pouch manufacturers and food processors to be able to predict the severity of their waffling problem when there are changes to a retort process. Pouch manufacturers will then know limitations of each pouch product used in a specific retort process and have an opportunity to develop new grades of retortable pouches which successfully work in retort process ranges that are not covered by existing grades. Food processors will have the ability to know if another grade of retort pouch is required for each newly developed scheduled process.

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CHAPTER THREE

MATERIALS AND METHODS

Retortable Pouch Preparation

Retortable pouches used for this experiment were four side seal pouches constructed of polyethylene terephthalate (PET) film 12 µm / aromatic laminating adhesive / aluminum foil 9 µm / aromatic laminating adhesive / biaxially oriented nylon (BON or BOPA) film 15 µm / aromatic laminating adhesive / retortable cast polypropylene (RCPP) film 80 µm (Prepack Thailand, Samut Sakhon, Thailand). The pouches were unprinted with a size of 200 mm in width and 300 mm in length. Pouches were initially sealed on three sides (manufacturer's seals) prior to being filled with 1,000 mL of room temperature water. The fourth seal (processor's seal) was made using a semi-automatic impulse sealer at 155°C for 1 second (Model CA300; Fuji Impulse, Osaka, Japan). The filled and sealed pouches were punctured at one corner using a hypodermic needle before the air was pulled out with a syringe to minimize headspace variation. The punctured corner of each pouch was sealed using the impulse sealer at 155°C for 1 second to ensure that the pouches were hermetically sealed.

Retort Processes

A horizontal water spray retort was used for this experiment (Model AO-142; Surdry, Biscay, Spain). The maximum allowing working pressure (MAWP) for this retort is 71.1 psig at 149°C, which covers the experimental ranges of 26-46 psig at 111-131°C. The retort was set to static mode during the experiment to eliminate motion as a variable. The filled pouches were placed horizontally on the retort rack (Fig. 3.1). The retort racks were designed to have 360 holes per square foot and each of these holes has a diameter of 0.5 inch to ensure that the heating medium can flow thoroughly.

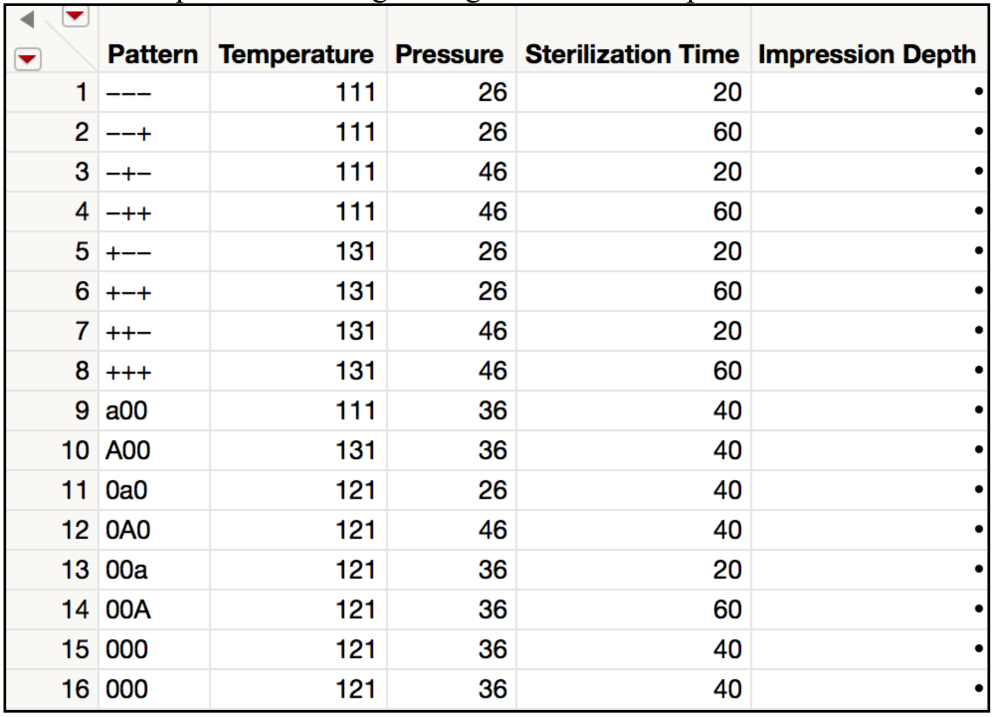
Figure 3.1. Pouch arrangement on the retort rack.

The multiple regression methodology was applied to the experiment with one response (impression depth) and three factors (temperature, overpressure, and sterilization time). In order to cover the experimental range for each factor, the retort processes were designed using:

- Three different temperature values (111°C, 121°C, 131°C)
- Three different overpressure values (26 psig, 36 psig, 46 psig)
- Three different sterilization time values (20 mins, 40 mins, 60 mins)

The Design of Experiment (DOE) used in this experiment was the central composite model, which requires a total of 16 retort processes to run (Table 3.1).

Table 3.1. Experimental design using the central composite model.



	Pattern	Temperature	Pressure	Sterilization Time	Impression Depth
1	−−−	111	26	20	•
2	−−+	111	26	60	•
3	−+−	111	46	20	•
4	−++	111	46	60	•
5	+−−	131	26	20	•
6	+−+	131	26	60	•
7	++−	131	46	20	•
8	+++	131	46	60	•
9	a00	111	36	40	•
10	A00	131	36	40	•
11	0a0	121	26	40	•
12	0A0	121	46	40	•
13	00a	121	36	20	•
14	00A	121	36	60	•
15	000	121	36	40	•
16	000	121	36	40	•

Sample Preparation

After the pouches were retorted, a corner of each pouch was cut to drain the water. The drained pouches were hung to be dried overnight prior to sample preparation. A grid pattern of 2 inches by 2 inches in size was drawn on the surface of the dried pouches where the waffling defects were observed (Fig. 3.2). The grid lines were cut by a paper trimmer to ensure that the waffling area was not affected.

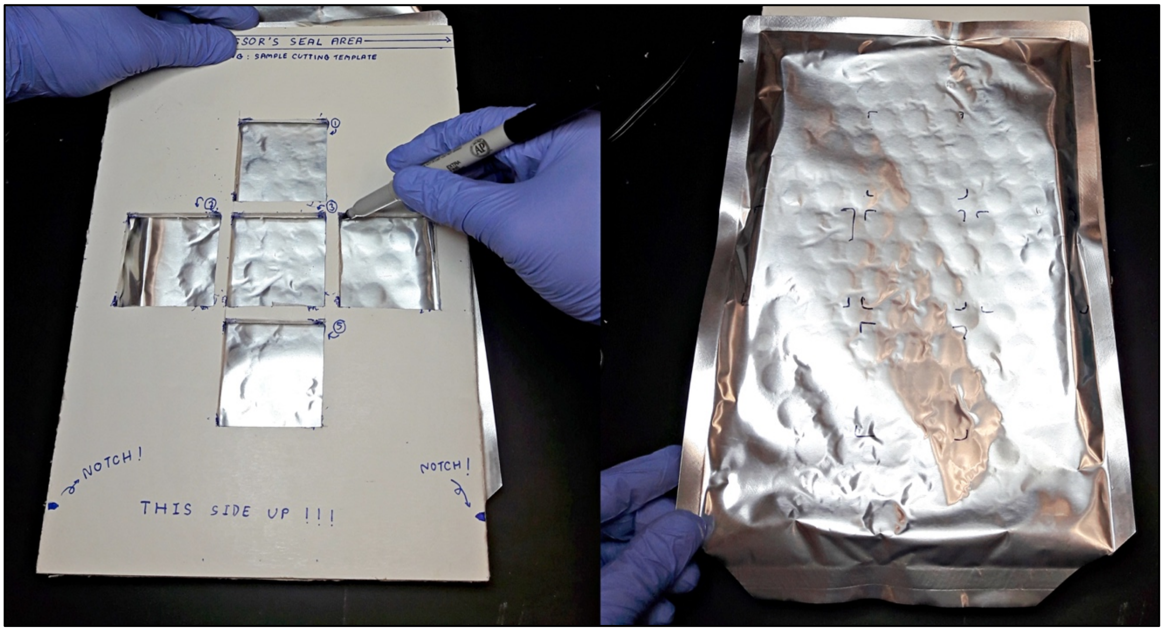


Figure 3.2. Drawing a grid pattern on the surface of the pouch.

Glass microscope slides of 2.25 inches by 2.75 inches in size were used as sample bases due to their complete flatness and uniform height. A layer of flat, double-sided tape was adhered on the glass microscope slide before the sample could be placed on the slide (Scotch double-sided permanent tape). A pair of forceps was used to place samples on the slides to minimize sample interference from handling.

Sample Measurement

To identify a measuring path, two points were drawn on each side of a circular impression (waffling defect) using a fine point permanent marker. The center point of the circular impression must be in the middle between these two points. A confocal laser scanning microscope with an objective lens of 5X was used to measure the dimension of the waffling defect (Model LEXT OLS 3000; Olympus, Tokyo, Japan). The stitching mode, which was designed to string multiple 3D-images together to create one large image, was required due to the measuring area being larger than the microscope's field of view. A stitched 3D-image (Fig. 3.3) was converted into a profile chart where its dimensions could be measured using measuring tools in Olympus LEXT software. Reference lines were placed at the highest point and the lowest point of the profile chart (Fig. 3.4). Impression depth of a waffling defect was determined by measuring the difference in height between the two reference lines.

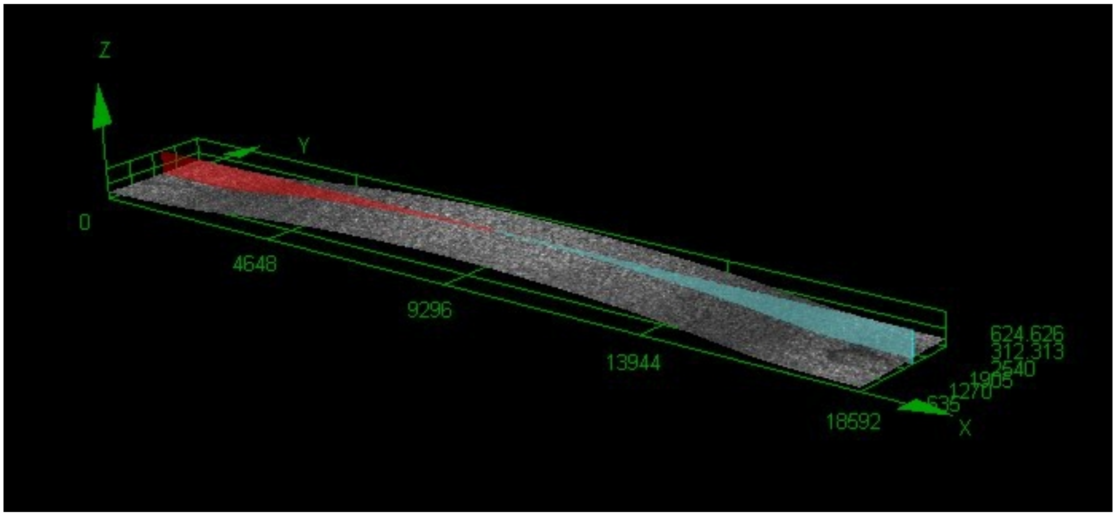


Figure 3.3. Sample of a stitched 3D-image.

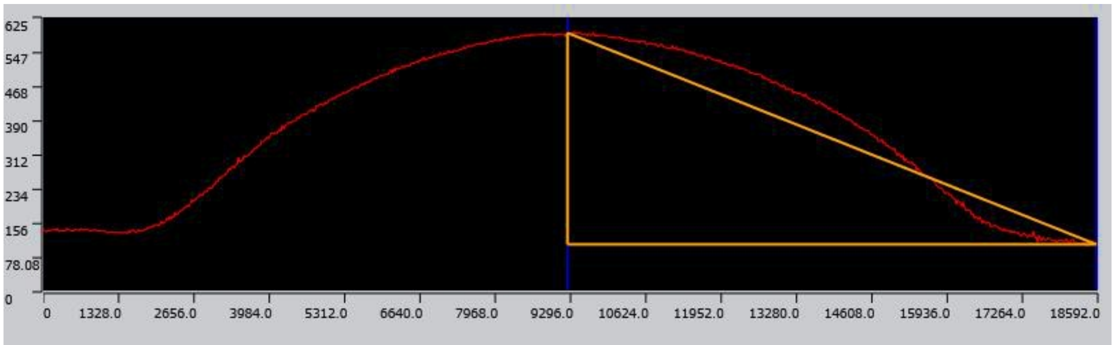


Figure 3.4. Measuring impression depth using reference lines.

Statistical Analysis

The measurements were made on 30 waffling defects of each retort process and there were 16 retort processes in this experiment. Therefore, a total number of 480 data points were used in the statistical analysis. Statistical analysis software, SAS studio version 3.7 (SAS Institute, Cary, NC), was used in comparing and recognizing the difference between the results. The t-test procedure (PROC TTEST), a function of SAS studio designed to perform hypothesis tests in comparing two population means, was used to determine the effect of temperature, overpressure, and sterilization time on depth of a surface impression or the severity of a waffling defect. A significance level of 0.05 or a confidence level of 95% was used in the hypothesis tests.

The response surface methodology (RSM), which is a collection of mathematical and statistical techniques based on the fit of a polynomial equation to the experimental data, was used to determine the relationship between independent variables (temperature, overpressure, and sterilization time) and dependent variable (depth of surface impression). The response surface regression procedure (PROC RSREG), a function of SAS studio designed to use the method of least squares to fit quadratic response surface regression models, was used to determine an equation expressing the relationship which is useful for predicting the severity of waffling defect for a given set of independent variables. Another statistical analysis software named JMP version pro 13.2.0 (SAS Institute, Cary, NC), which has a response surface methodology (RSM) function, was also used to analyze the experimental data and plot a three-dimensional surface response diagram based on the regression equation to demonstrate the relationship.

CHAPTER FOUR

RESULTS AND DISCUSSION

Effect of Retort Temperature on the Severity of Waffling Defects

The effect of retort temperature on the severity of waffling defects was determined by comparing population means of measured impression depth values between waffling defects occurring in low temperature retort processing (111°C) and waffling defects occurring in high temperature retort processing (131°C). To make sure that a specific range of retort process factor combinations was covered in the comparison, four experiments were conducted separately based on different overpressure and sterilization time settings. Combinations of overpressure settings (low overpressure at 26 psig or high overpressure at 46 psig) and sterilization time settings (short sterilization time at 20 minutes or long sterilization time at 60 minutes) were used to conduct the experiments.

Table 4.1 shows the changes in the average and standard deviation values of measured impression depth of waffling defects based on the different retort temperatures for all four experiments. There was a significant difference in measured impression depth between the low temperature setting (111°C) and high temperature setting (131°C) at a level of significance of 0.05 ($p<0.05$). Further analysis of the results indicated that the higher temperature setting resulted in higher measured impression depth ($p<0.05$) for all four experiments (Table 4.2).

Table 4.1. Effect of temperature on average and standard deviation values of measured impression depth.

Combination	Avg Depth (μm)		Std. Dev. (μm)	
	at 111°C	at 131°C	at 111°C	at 131°C
Case 1.1: Low Overpressure - Short Time	116.6	427.0	47.3	63.2
Case 1.2: Low Overpressure - Long Time	122.2	472.1	45.6	123.5
Case 1.3: High Overpressure - Short Time	153.8	187.4	58.9	48.8
Case 1.4: High Overpressure - Long Time	155.5	218.0	50.7	91.0

Table 4.2. Effect of temperature on the severity of waffling defects (at α=0.05).

Overpressure	**Sterilization Time**	
	Short (20 mins)	**Long (60 mins)**
Low (26 psi)	**CASE 1.1** Higher Temp results in Higher Impression Depth	**CASE 1.2** Higher Temp results in Higher Impression Depth
High (46 psi)	**CASE 1.3** Higher Temp results in Higher Impression Depth	**CASE 1.4** Higher Temp results in Higher Impression Depth

Case 1.1: Effect of temperature at low overpressure and short sterilization time.

The distribution curves and box plots (Fig. 4.1) compare the data sets of the impression depth values from the different retort temperatures when the pouches were retorted at low overpressure (26 psig) and short sterilization time (20 minutes). There was a significant difference (p<0.05) in measured impression depth regarding the retort temperatures used. Higher average impression depth (p<0.05) was observed for samples processed at a high retort temperature of 131°C (427.0 ± 63.2 microns) compared to at a low retort temperature of 111°C (116.6 ± 47.3 microns). Even though there was a standard deviation gap of 15.9 microns between the two data sets, the equality of variances test showed that the samples from different retort temperatures were equal in variance (p>0.05).

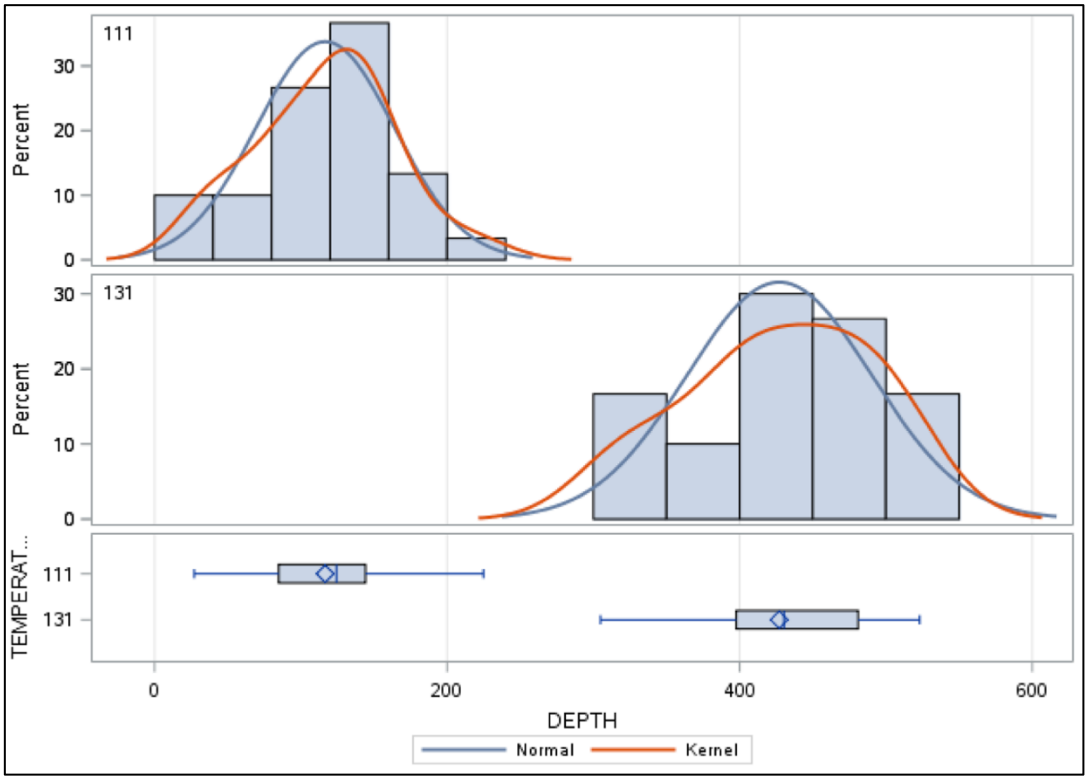


Figure 4.1. Distribution of impression depth values over different temperatures (111°C and 131°C) at low overpressure (26 psig) and short sterilization time (20 mins).

Case 1.2: Effect of temperature at low overpressure and long sterilization time.

The difference in the impression depth value between data sets from different retort temperatures when the pouches were retorted at low overpressure (26 psig) with longer sterilization time (60 minutes) is demonstrated in the distribution curves and box plots (Fig. 4.2). The measured impression depth of the samples processed at a retort temperature of 111°C was statistically different ($p<0.05$) from the samples processed at 131°C. The average impression depth of the samples retorted at 131°C (472.1 ± 123.5 microns) was statistically higher ($p<0.05$) than the average impression of the samples retorted at 111°C (122.2 ± 45.6 microns). The equality of variances test did show that the samples from different retort temperatures were not equal in variance ($p<0.05$). There was a higher variance (*p*<0.05) in the impression depth value for samples processed at a high retort temperature of 131°C compared to at a low retort temperature of 111°C.

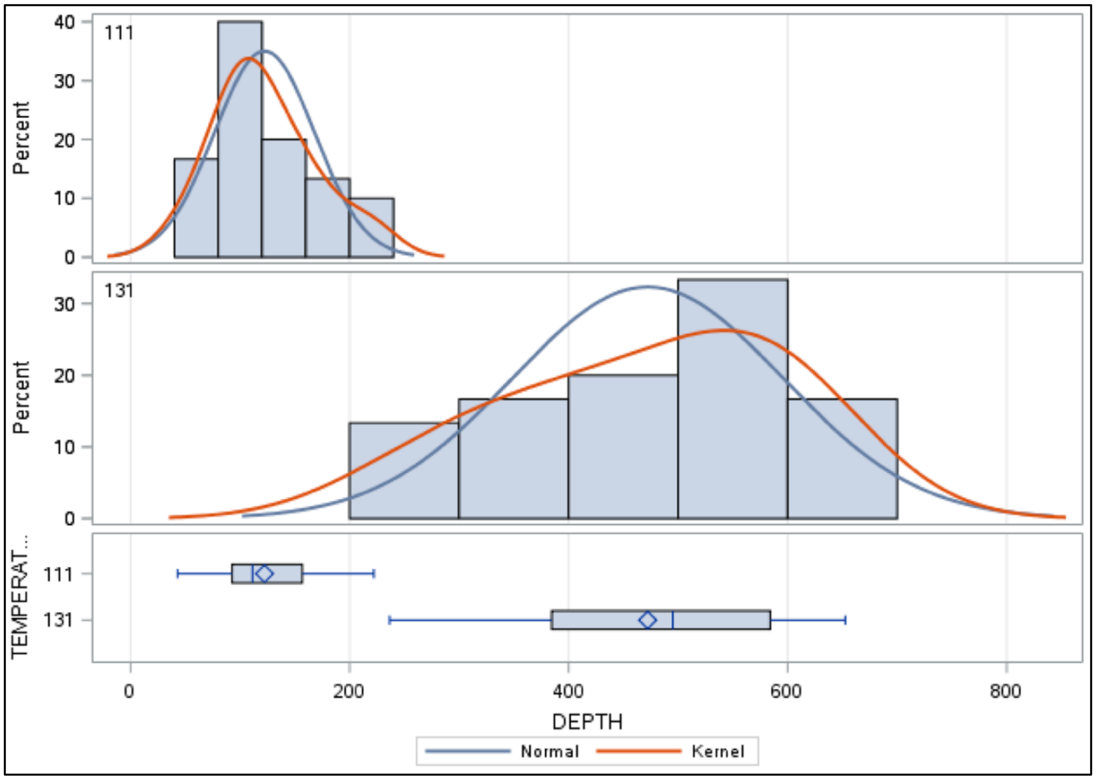


Figure 4.2. Distribution of impression depth values over different temperatures (111°C and 131°C) at low overpressure (26 psig) and long sterilization time (60 mins).

Case 1.3: Effect of temperature at high overpressure and short sterilization time.

After the pouches were retorted at a higher overpressure of 46 psig with a short sterilization time of 20 minutes, the data sets of impression depth values from the different retort temperatures were compared using distribution curves and box plots to see if there was any difference (Fig. 4.3). There was a significant difference (*p*<0.05) in measured impression depth regarding the retort temperatures used. The average impression depth of the samples processed at 131°C (187.4 ± 48.8 microns) was statistically higher (*p*<0.05) than the samples processed at 111°C (153.8 ± 58.9 microns).

With a standard deviation gap of 10.1 microns between these two data sets, the statistical analysis using the equality of variances test showed that the samples from the two retort temperatures were equal in variance ($p>0.05$).

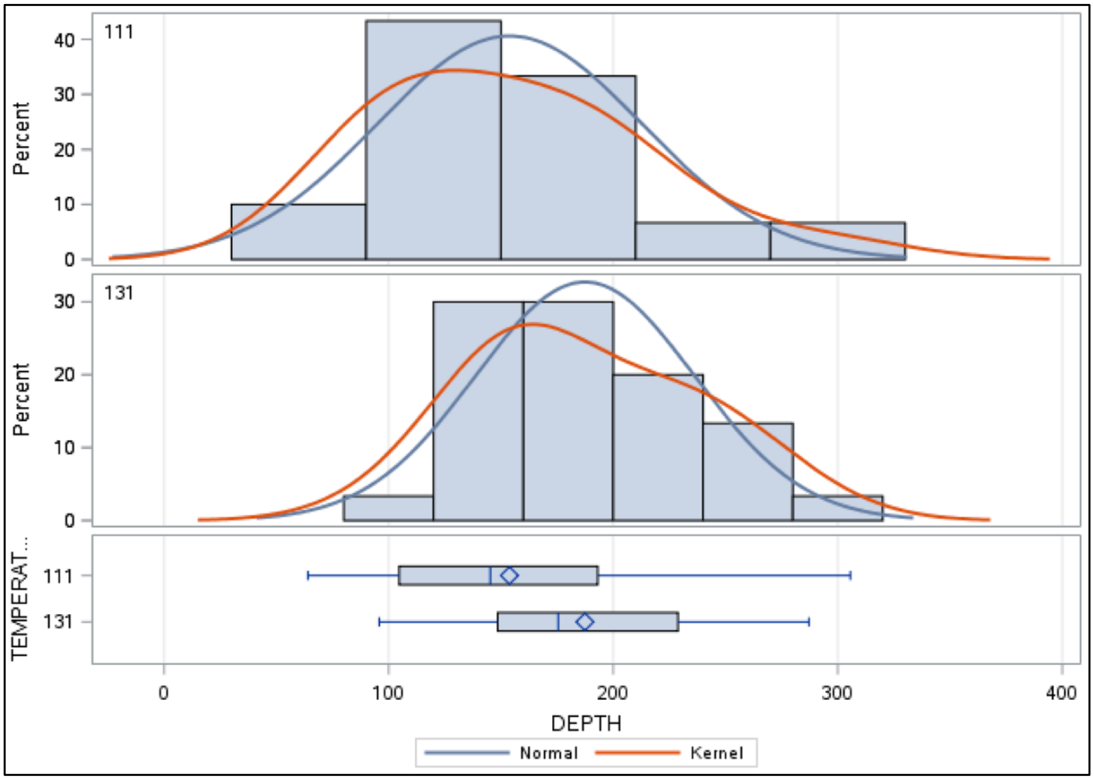


Figure 4.3. Distribution of impression depth values over different temperatures (111°C and 131°C) at high overpressure (46 psig) and short sterilization time (20 mins).

Case 1.4: Effect of temperature at high overpressure and long sterilization time.

The distribution curves and box plots (Fig. 4.4) compare the data sets of the impression depth values from the different retort temperatures when the pouches were retorted at high overpressure (46 psig) and long sterilization time (60 minutes). There was a significant difference ($p<0.05$) in measured impression depth regarding the retort temperatures used. Higher average impression depth ($p<0.05$) was observed for samples processed at 131°C (218.0 ± 91.0 microns) compared to 111°C (155.5 ± 50.7 microns).

With a difference between the standard deviation values of 40.3 microns, the equality of variances test did show that the samples from different retort temperatures were not equal in variance ($p<0.05$). There was a higher variance ($p<0.05$) in the impression depth value for samples processed at a high retort temperature of 131°C compared to at a low retort temperature of 111°C.

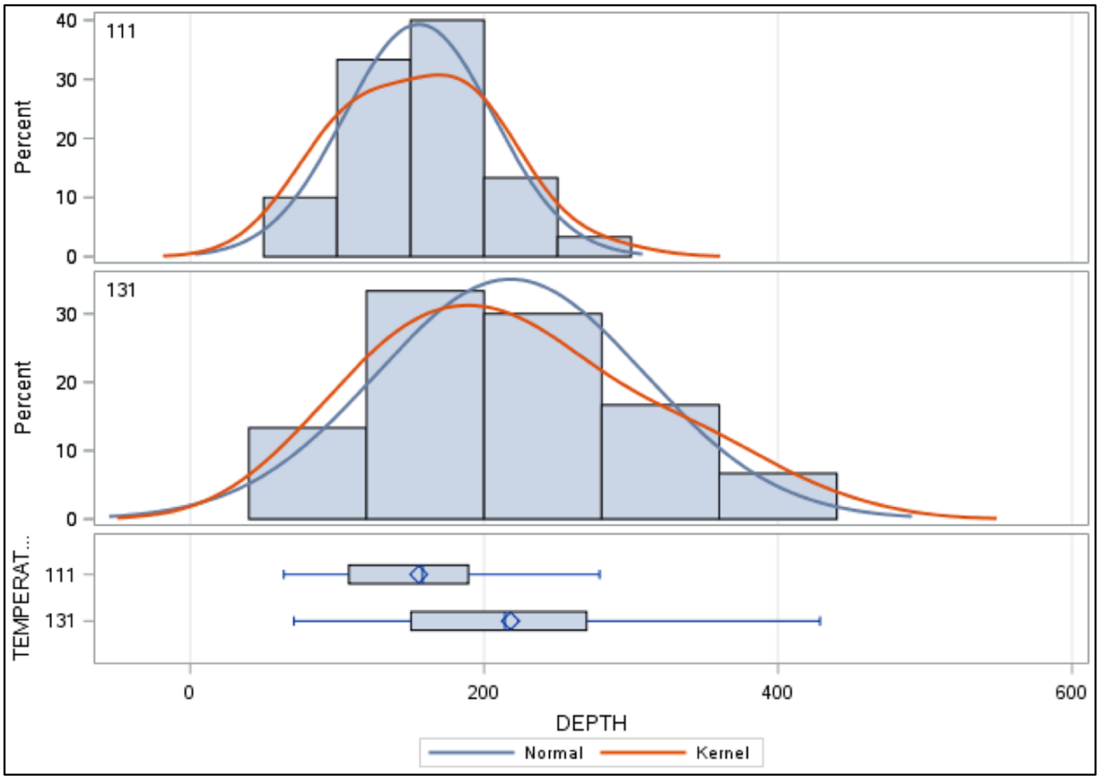


Figure 4.4. Distribution of impression depth values over different temperatures (111°C and 131°C) at high overpressure (46 psig) and long sterilization time (60 mins).

The results from the experiments showed that the higher temperature setting resulted in higher measured impression depth ($p<0.05$) in all of the tested overpressure and sterilization time combinations. These results can be attributed to a common characteristic of the polymers used in the flexible pouch for this experiment, which is that the flexural strength changes as a function of temperature. Flexural strength is the ability of a material to resist deformation from bending forces applied perpendicular to its longitudinal axis (Campo, 2008).

Since polyethylene terephthalate (PET), polyamide (Nylon), and polypropylene (PP) are semi-crystalline polymers, which have both crystalline and amorphous regions, they combine the strength of crystalline polymers with the flexibility of amorphous polymers. Their characteristics depend on their degree of crystallinity because higher crystallinity results in a harder and more thermally stable material. When semi-crystalline polymers are heated above their glass transition temperature, amorphous regions in semi-crystalline polymers are ductile and able to deform plastically. Even though their ductility increases with higher temperature, semi-crystalline polymers remain in a solid state until a given quantity of heat is absorbed to a point where they rapidly change into a low viscosity liquid, also known as the melting temperature. Therefore, when heating the semi-crystalline polymers to temperatures between their glass transition temperature (T_g) and melting temperature (T_m), they slowly lose their flexural strength as the temperature increases (Tripathi, 2002).

Since the retort temperatures used for this experiment ranged from 111°C to 131°C, the polymers used in the flexible pouch were heated to a temperature between their T_g and T_m (Table 4.3) (Ghosh, 2002). Therefore, the heated polymer layers in the flexible pouches slowly decreased their flexural strength and consequently decreased their ability to resist deformation from the weight of the product, which presses the pouch surface onto the retort rack. This deformation led to the presence of surface impressions on the pouch, also referred to as waffling.

Table 4.3. T_g and T_m of the polymers used in the flexible pouch for the experiment.

Polymer	T_g, °C	T_m, °C
Polyethylene terephthalate (PET)	69	254
Polycaprolactam (Nylon 6)	50	215
Polypropylene (PP)	-20	176

The results from the experiment also showed that there were two cases with a difference in variance ($p<0.05$) between the samples retorted at a low temperature of 111°C and at a high temperature of 131°C. Case 1.2 and Case 1.4 were both retorted at a long sterilization time of 60 minutes, but at different overpressure settings of 26 psig and 46 psig, respectively. Further analysis showed that when samples were retorted for a long sterilization time (60 minutes), the samples retorted at a high temperature (131°C) had a higher variance ($p<0.05$) of impression depth values than the samples retorted at a low temperature (111°C) at any overpressure setting. Therefore, not only the impression depth but also the variation in severity of the waffling defects was affected by the temperature setting when samples were retorted for a long sterilization time.

From a production point of view, variation is a disparity between an actual measure of a product characteristic and its target value. Excessive variation is outside of the acceptable limits established for a product specification, which can lead to product rejection. For this reason, a key manufacturing performance objective is the establishment of stable and predictable processes that limit variation to what can be described as random, minimum variation around target values. In this study, the difference in the variance of the waffling defect severity can be used to determine the more preferable retort processes in terms of quality control. A retort process with a smaller variance of the waffling defect severity results in greater consistency, predictability, and quality, which tends to minimize waste and produce products that perform consistently over time (Nordmeyer, 2018; Wachs, 2018).

Effect of Retort Overpressure on the Severity of Waffling Defects

The effect of retort overpressure on the severity of waffling defects was determined by comparing population means of measured impression depth values between waffling defects occurring in low overpressure (26 psig) retort processing and waffling defects occurring in high overpressure (46 psig) retort processing.

Table 4.4 shows the changes in average and standard deviation values of measured impression depth of waffling defects based on the different overpressures for all four experiments. There was a significant difference in measured impression depth between the low overpressure setting (26 psig) and high overpressure setting (46 psig) at a level of significance of 0.05 ($p<0.05$). Further analysis of the results indicated that the higher overpressure setting resulted in higher measured impression depth ($p<0.05$) when samples were retorted at a low temperature (111°C). However, the results were different when samples were retorted at different temperatures. A higher overpressure setting resulted in lower measured impression depth ($p<0.05$) when samples were retorted at a high temperature (131°C) (Table 4.5).

Table 4.4. Effect of overpressure on average and standard deviation values of measured impression depth.

Combination	Avg Depth (µm)		Std. Dev. (µm)	
	at 26 psi	at 46 psi	at 26 psi	at 46 psi
Case 2.1: Low Temperature - Short Time	116.6	153.8	47.3	58.9
Case 2.2: Low Temperature - Long Time	122.2	155.5	45.6	50.7
Case 2.3: High Temperature - Short Time	427.0	187.4	63.2	48.8
Case 2.4: High Temperature - Long Time	472.1	218.0	123.5	91.0

Table 4.5. Effect of overpressure on the severity of waffling defects (at α=0.05).

Temp	**Sterilization Time**	
	Short (20 mins)	**Long (60 mins)**
Low (111°C)	**CASE 2.1** Higher Pressure results in Higher Impression Depth	**CASE 2.2** Higher Pressure results in Higher Impression Depth
High (131°C)	**CASE 2.3** Higher Pressure results in Lower Impression Depth	**CASE 2.4** Higher Pressure results in Lower Impression Depth

Case 2.1: Effect of overpressure at low temperature and short sterilization time.

The distribution curves and box plots (Fig. 4.5) compare the data sets of the impression depth values from the different overpressures when the pouches were retorted at a low temperature (111°C) and short sterilization time (20 minutes). There was a significant difference (p<0.05) in measured impression depth regarding the overpressures used. Higher average impression depth (p<0.05) was observed for samples processed at a high overpressure of 46 psig (153.8 ± 58.9 microns) compared to at a low overpressure of 26 psig (116.6 ± 47.3 microns). Even though there was a standard deviation gap of 11.6 microns between the two data sets, the equality of variances test showed that the samples from different retort temperatures were equal in variance (p>0.05).

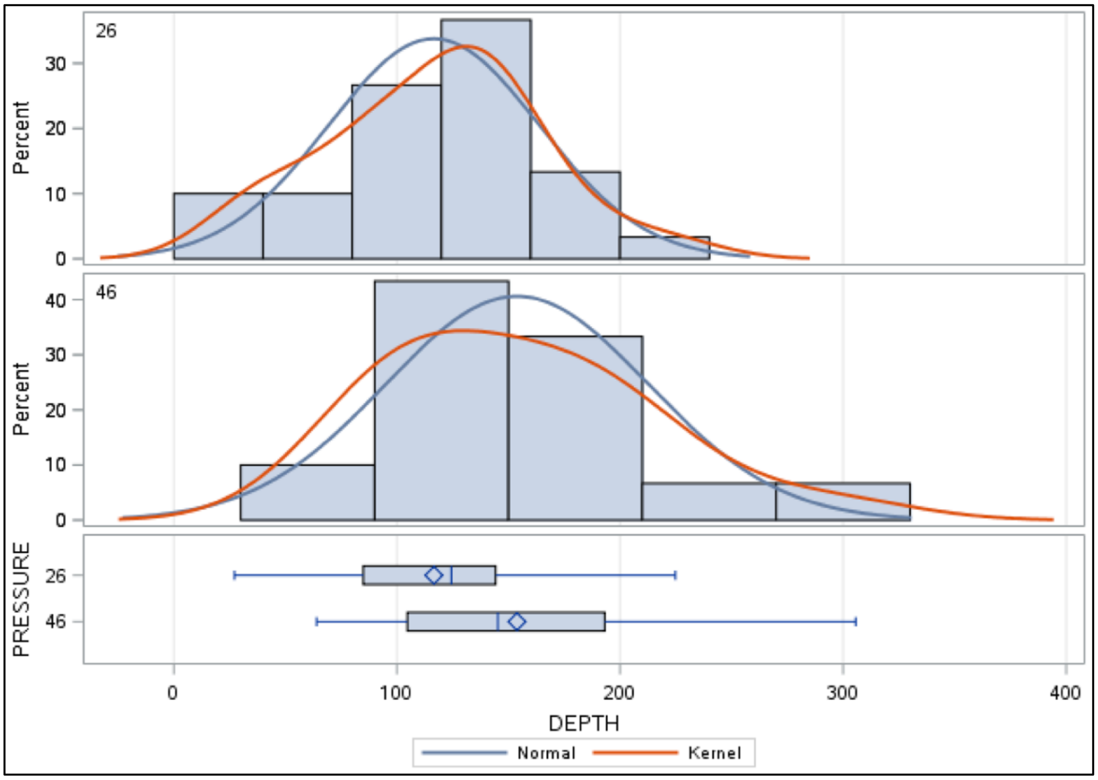


Figure 4.5. Distribution of impression depth values over different overpressures (26 psig and 46 psig) at low temperature (111°C) and short sterilization time (20 mins).

Case 2.2: Effect of overpressure at low temperature and long sterilization time.

Impression depth value differences between the data sets of different overpressures when the pouches were retorted at low retort temperature (111°C) with longer sterilization time (60 minutes) are shown in the distribution curves and box plots (Fig. 4.6). The measured impression depth of the samples processed at an overpressure of 46 psig was different ($p<0.05$) from the samples processed at 26 psig. The analysis showed that the average impression depth of the samples retorted at 46 psig (155.5 ± 50.7 microns) was statistically higher ($p<0.05$) than the average impression of the samples retorted at 26 psig (122.2 ± 45.6 microns). With a standard deviation gap of only 5.1

microns, the equality of variances test did show that these samples from different overpressures were equal in variance (*p*>0.05).

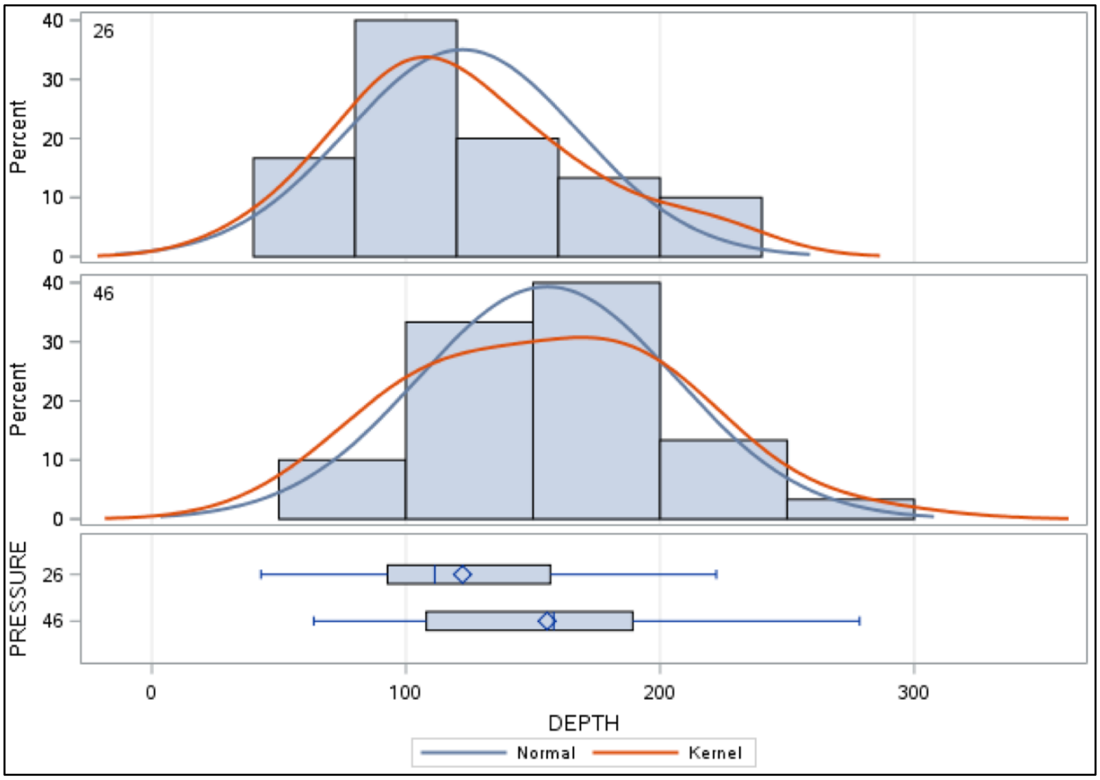


Figure 4.6. Distribution of impression depth values over different overpressures (26 psig and 46 psig) at low temperature (111°C) and long sterilization time (60 mins).

Case 2.3: Effect of overpressure at high temperature and short sterilization time.

After the pouches were retorted at a higher temperature of 131°C with a short sterilization time of 20 minutes, the data sets of impression depth values from the different overpressures were compared using distribution curves and box plots to determine if there was any difference (Fig. 4.7). A significant difference (*p*<0.05) was seen in measured impression depth regarding the overpressures used. Further analysis showed different results from previous experiments. The average impression depth of the samples processed at an overpressure of 46 psig (187.4 ± 48.8 microns) was lower (*p*<0.05) than the samples processed at 26 psig (427.0 ± 63.2 microns). There was a standard deviation gap of 14.4 microns between the two data sets, nevertheless, the equality of variances test showed that the samples from different retort temperatures were equal in variance (*p*>0.05).

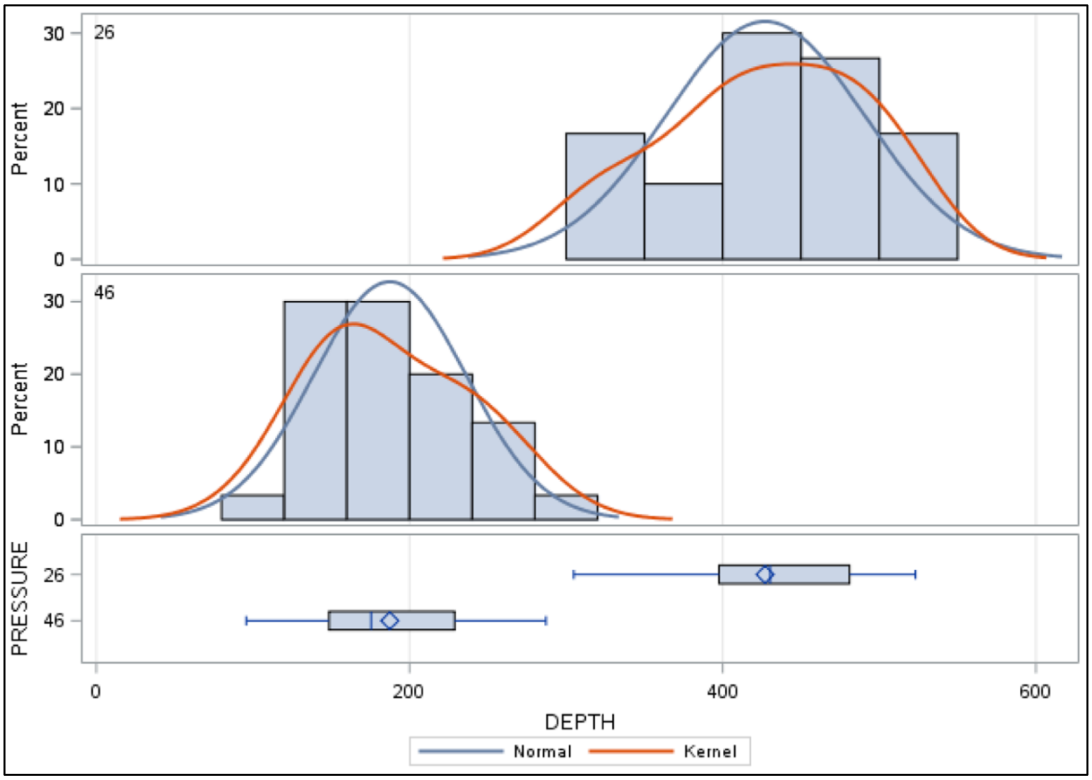


Figure 4.7. Distribution of impression depth values over different overpressures (26 psig and 46 psig) at high temperature (131°C) and short sterilization time (20 mins).

Case 2.4: Effect of overpressure at high temperature and long sterilization time.

The distribution curves and box plots (Fig. 4.8) compare the data sets of the impression depth values from the different overpressures when the pouches were retorted at high temperature (131°C) and long sterilization time (60 minutes). There was a significant difference (*p*<0.05) in measured impression depth regarding the overpressures used. Further analysis showed similar results as Case 2.3, even when the long sterilization time was used.

A lower average impression depth ($p<0.05$) was seen for the samples processed at a high overpressure of 46 psig (218.0 ± 91.0 microns) compared to at a low overpressure of 26 psig (472.1 ± 123.5 microns). Even though there was a standard deviation gap of 32.5 microns between the two data sets, the equality of variances test showed that the samples from different overpressures were equal in variance ($p>0.05$).

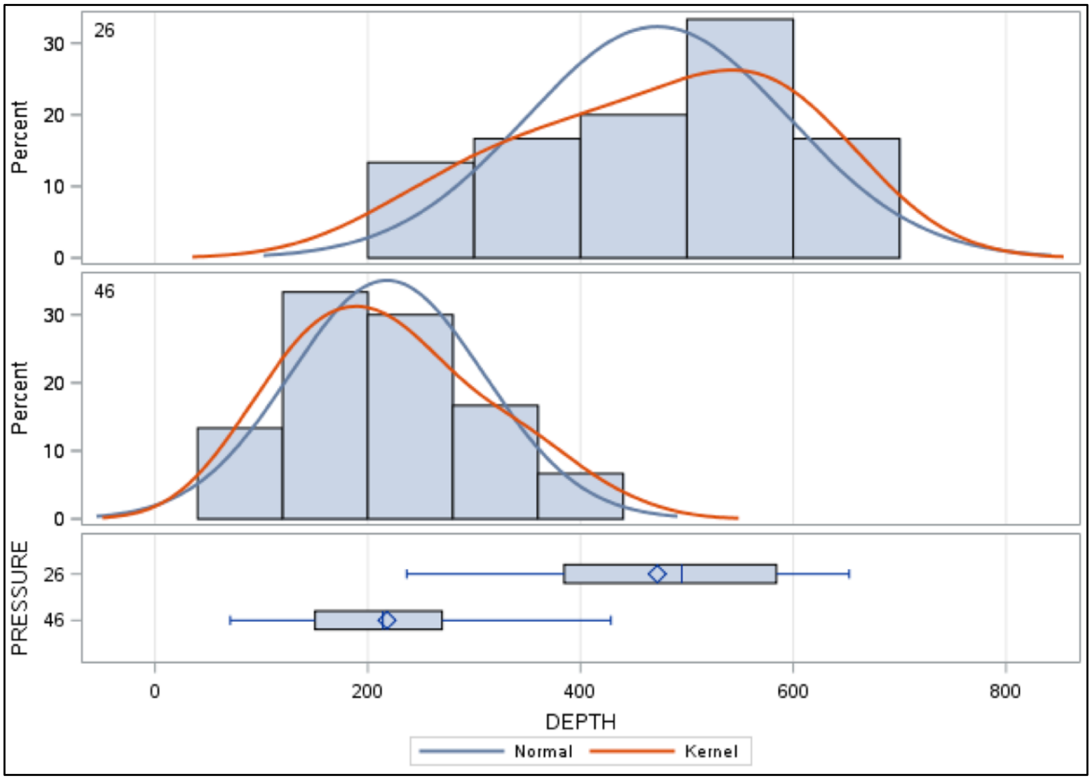


Figure 4.8. Distribution of impression depth values over different overpressures (26 psig and 46 psig) at high temperature (131°C) and long sterilization time (60 mins).

There were two cases in which the higher overpressure setting resulted in a higher measured impression depth ($p<0.05$). Case 2.1 and Case 2.2 were both retorted at a low temperature of 111°C but at a different sterilization time of 20 minutes and 60 minutes.

The results can be attributed to a common phenomenon that occurs in thin-walled pressure vessels. A hermetically sealed flexible pouch becomes an internally pressurized thin-wall vessel when the thermal expansion of the product and residual air within the pouch occurs during retort processing. In Figure 4.9, a thin square plate is used to represent a small element of a pouch's surface to demonstrate the stresses that act upon it. The red arrows (σ) represent the direction of normal stresses acting perpendicular to each neighboring small element, while the black arrows (P) represent the stresses acting on the surface of the pouch (Ibrahim, Ryu, & Saidpour, 2015).

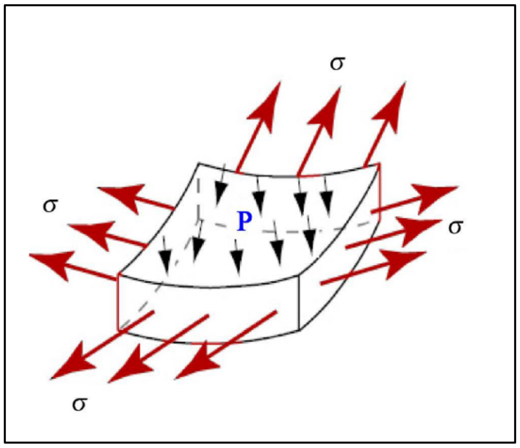


Figure 4.9. Stresses in an element of pouch surface when internally pressurized.

When the thermal expansion of products and residual air occur, the stresses acting on the inside of bottom surface of the pouch (P) are the weight of the product and the force from the internal pressure. Since pressure expands in all directions, when a pouch is internally pressurized, it causes the pouch to bulge outward in all directions. Therefore, all surfaces of the pouch are acted on by the force from the internal pressure. Since a four side seal pouch has a seal on each of its four edges, when both surfaces of the pouch are acted on by internal pressure, it forms a shape similar to a traditional bed pillow (Fig. 4.10).

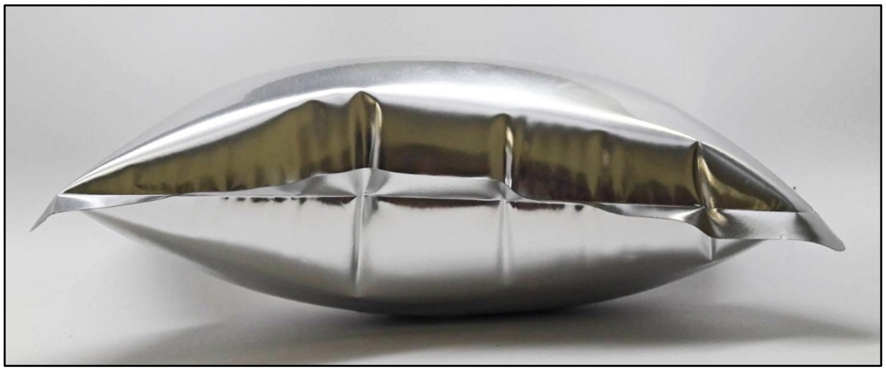


Figure 4.10. Internally pressurized pouch.

As a force component of normal stresses (σ) is in the opposite direction of the stresses acting on the inside surface of the pouch (P), the two forces will act equally to maintain force equilibrium, which will cancel each other out and result in a net force of zero. Therefore, when the pouch was hermetically sealed, higher internal pressure results in higher normal stresses (σ) on the pouch surface until the forces are in equilibrium. When the force of the normal stresses (σ) is higher, it pulls the material more towards its direction and reduces the influence of the product weight that pushes the bottom surface of the pouch through the holes of retort rack pattern, which results in less surface impression and less severity of waffling defects.

When overpressure is introduced, force from external pressure is applied onto the outside surface of the pouch which acts directly against the internal pressure. This allows the internal and external forces to reach equilibrium at a lower force of normal stresses (σ), where the influence of the product weight remains and pushes the bottom surface of the pouch through the holes of retort rack resulting in a waffling pattern. Therefore, at low retort temperature (111°C) higher overpressure results in higher severity of waffling defects ($p<0.05$).

However, when the pouches were retorted at high temperatures, different results were observed. In Case 2.3 and Case 2.4, the higher overpressure setting resulted in a lower measured impression depth ($p<0.05$). These two cases were both retorted at a high temperature of 131°C, but at a different sterilization time setting of 20 minutes and 60 minutes. These results agree with a common behavior of semi-crystalline polymers, which is the flexural strength as a function of temperature as mentioned previously.

When heating semi-crystalline polymers to a temperature between their glass transition temperature (T_g) and melting temperature (T_m), they slowly lose their flexural strength as the temperature increases and consequently decrease their ability to resist deformation. This allows the force from the product weight to push the bottom surface of the pouch through the holes of the retort rack to form the waffling pattern more easily, resulting in the presence of surface impression on the pouch or waffling defect.

When deformation of the bottom surface of the pouch occurred, it interfered with the ability of the pouch surface to be pulled towards the direction of normal stresses (σ) and resulted in the occurrence of severe wrinkles on the pouch surface (Fig. 4.11). Losing the ability to be pulled in the normal stress (σ) direction reduces influence of the normal stresses acting against the force of internal pressure and product weight on the bottom internal surface of the pouch (P) and results in a more severe surface impression.

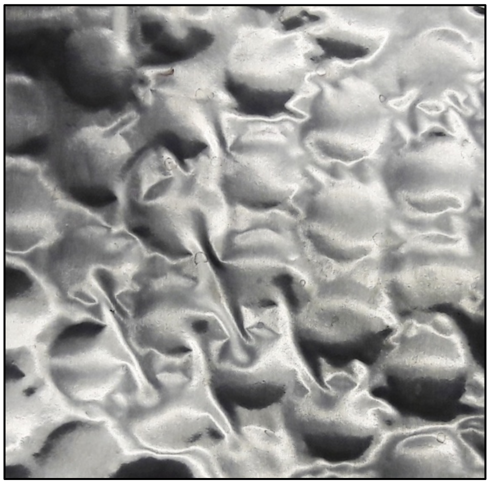


Figure 4.11. Wrinkles on pouch surface.

When overpressure is introduced, the force from external pressure is applied onto the outside surface of the pouch, which acts directly against the internal pressure that causes a severe surface impression. Therefore, at a high retort temperature (131°C), where the deformation due to the loss of flexural strength occurs, higher overpressure results in lower severity of waffling defects ($p<0.05$).

In addition, the results from all the experiments showed no significant difference in variance ($p>0.05$) between the samples retorted at a low overpressure of 26 psig and at a high overpressure of 46 psig. Therefore, the change in only overpressure settings of the retort process, which showed no significant change in variance of waffling defect severity, did not significantly affect the ability to control the waffling defect.

Effect of Sterilization Time on the Severity of Waffling Defects

The effect of sterilization time on the severity of waffling defects was determined by comparing population means of measured impression depth values between waffling defects occurring in short sterilization time (20 minutes) retort processing and waffling defects occurring in long sterilization time (60 minutes) retort processing.

Table 4.6 shows the changes in average and standard deviation values of measured impression depth of waffling defects based on the different sterilization time settings for all four experiments. There was a significant difference in measured impression depth between a short sterilization time setting of 20 minutes and a long sterilization time setting of 60 minutes ($p<0.05$) only if the samples were retorted at a high temperature and a low overpressure. Further analysis of the results indicated that the longer sterilization time resulted in higher measured impression depth ($p<0.05$) when samples were retorted at a high temperature of 131°C with a low overpressure of 26 psig. Different results were observed when samples were retorted at different temperature and overpressure combinations. There was no significant difference in average measured impression depth ($p>0.05$) between the different sterilization time settings when samples were retorted either at a low temperature (111°C) or at a high overpressure setting (46 psig) (Table 4.7).

Table 4.6. Effect of sterilization time on average and standard deviation values of measured impression depth.

Combination	Avg Depth (μm)		Std. Dev. (μm)	
	at 20 mins	at 60 mins	at 20 mins	at 60 mins
Case 3.1: Low Temperature - Low Ovepressure	116.6	122.2	47.3	45.6
Case 3.2: Low Temperature - High Ovepressure	153.8	155.5	58.9	50.7
Case 3.3: High Temperature - Low Ovepressure	427.0	472.1	63.2	123.5
Case 3.4: High Temperature - High Ovepressure	187.4	218.0	48.8	91.0

Table 4.7. Effect of sterilization time on severity of waffling defect (at α=0.05).

Temp	**Overpressure**	
	Low (26 psi)	**High (46 psi)**
Low (111°C)	**CASE 3.1** Time has no significant effect on Impression Depth	**CASE 3.2** Time has no significant effect on Impression Depth
High (131°C)	**CASE 3.3** Longer Time results in Higher Impression Depth	**CASE 3.4** Time has no significant effect on Impression Depth

Case 3.1: Effect of sterilization time at low temperature and low overpressure.

The distribution curves and box plots (Fig. 4.12) compare the data sets of the impression depth values from the different sterilization time settings when the pouches were retorted at low retort temperature (111°C) and low overpressure (26 psig). The average impression depth for the samples processed at a long sterilization time of 60 minutes was 122.2 ± 45.6 microns, while it was 116.6 ± 47.3 microns for the samples processed at a short sterilization time of 20 minutes. However, there was no significant difference (p>0.05) in measured impression depth regarding the sterilization time settings used. There was a gap of only 1.7 microns between standard deviation values of the samples retorted at different sterilization time settings. These samples with minor difference in standard deviation values were proven to be equal in variance (p>0.05) using the equality of variances test.

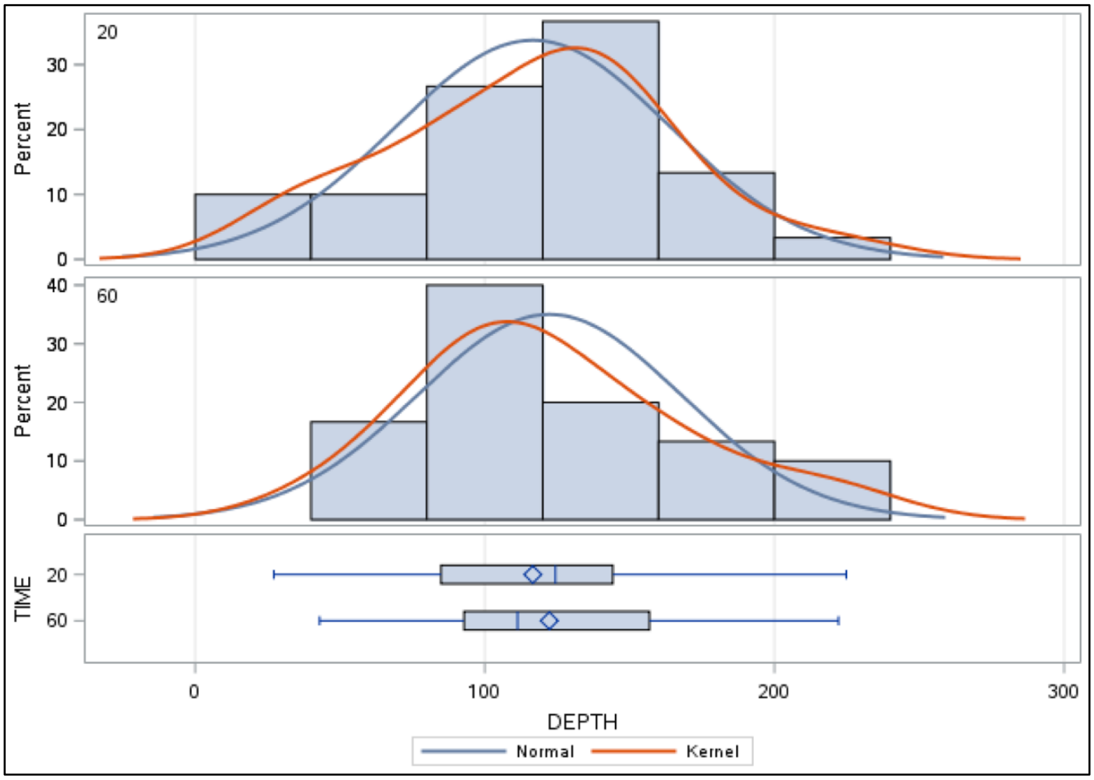


Figure 4.12. Distribution of impression depth values over different sterilization time (20 mins and 60 mins) at low temperature (111°C) and low overpressure (26 psig).

Case 3.2: Effect of sterilization time at low temperature and high overpressure.

The difference in the impression depth value between data sets from different sterilization time settings when the pouches were retorted at a low retort temperature of 111°C with a higher overpressure of 46 psig is demonstrated in the distribution curves and box plots (Fig. 4.13). The average of impression depth value from the samples processed at a sterilization time of 60 minutes and 20 minutes were 155.5 ± 50.7 microns and 153.8 ± 58.9 microns, respectively. The average impression depth of the samples retorted at different sterilization time settings were not different ($p > 0.05$). There was a gap of only 8.2 microns between standard deviation values of the samples retorted at different sterilization time settings. The equality of variances test did show that the samples from different sterilization time settings were equal in variance ($p > 0.05$).

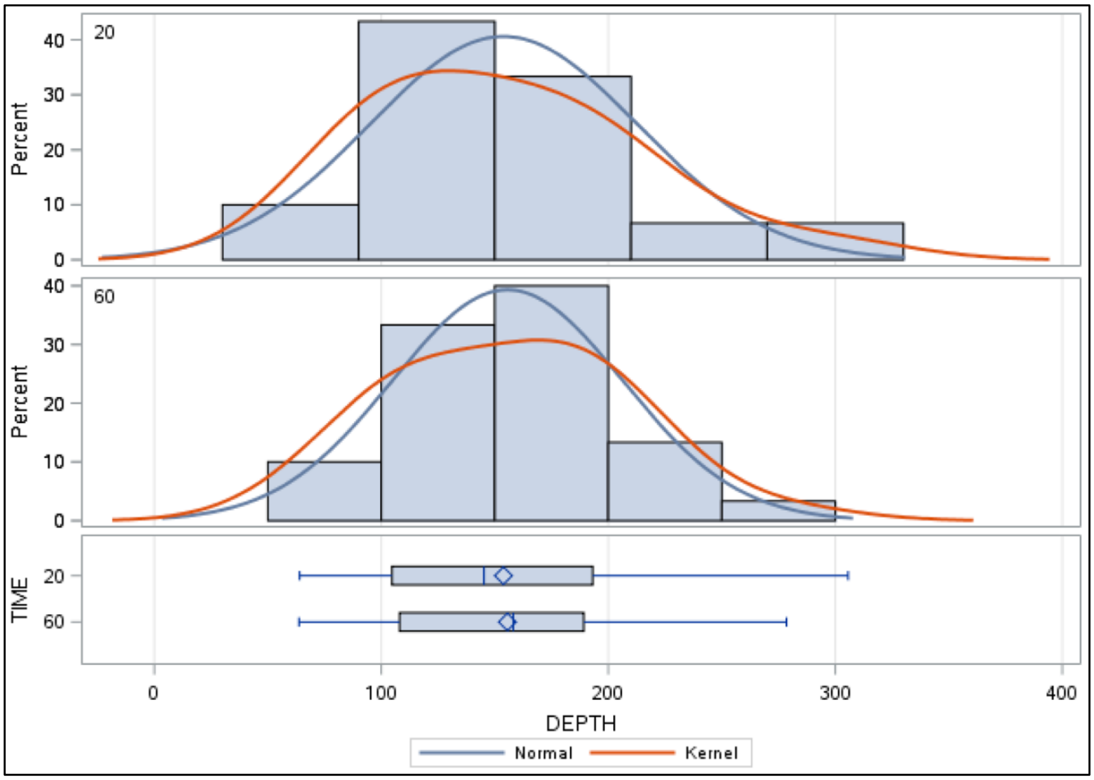


Figure 4.13. Distribution of impression depth values over different sterilization time (20 mins and 60 mins) at low temperature (111°C) and high overpressure (46 psig).

Case 3.3: Effect of sterilization time at high temperature and low overpressure.

After the pouches were retorted at a higher temperature of 131°C with a low overpressure of 26 psig, the data sets of impression depth values from the different sterilization time settings were compared using distribution curves and box plots to see if there was any difference (Fig. 4.14). Statistical analysis showed different results from previous experiments. The significant difference in measured impression depth regarding the sterilization time settings used was shown ($p < 0.05$) in the analysis. The average impression depth of the samples processed at a long sterilization time of 60 minutes was

472.1 ± 123.5 microns and found to be statistically higher (*p*<0.05) than the samples processed at a short sterilization time of 20 minutes with an average impression depth of 427.0 ± 63.2 microns. There was a gap of 60.3 microns between the standard deviation value of the data sets retorted at different sterilization time settings. The equality of variances test showed that the samples from different sterilization time settings were not equal in variance (*p*<0.05). There was a higher variance (*p*<0.05) in the impression depth value for samples processed at a long sterilization time of 60 minutes compared to at a short sterilization time of 20 minutes.

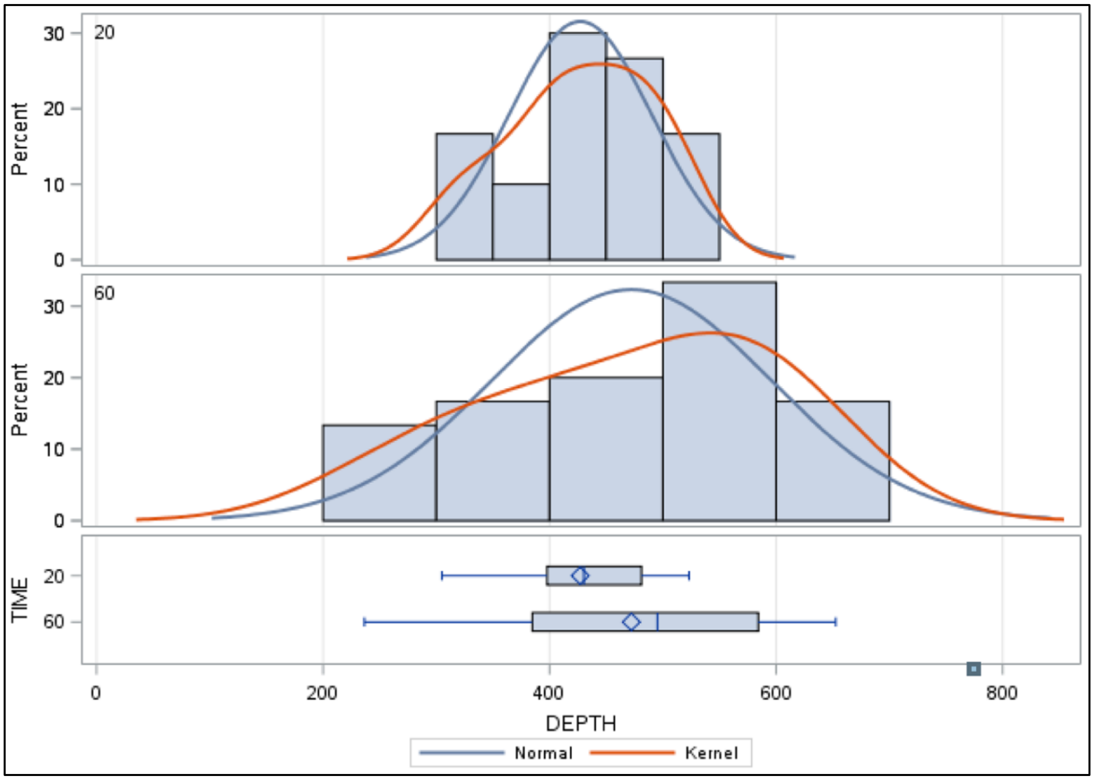


Figure 4.14. Distribution of impression depth values over different sterilization time (20 mins and 60 mins) at high temperature (131°C) and low overpressure (26 psig).

Case 3.4: Effect of sterilization time at high temperature and high overpressure.

The distribution curves and box plots (Fig. 4.15) compare the data sets of the impression depth values from the different sterilization time settings when the pouches were retorted at high temperature (131°C) and high overpressure (46 psig). The average impression depth for the samples processed at a long sterilization time of 60 minutes was 218.0 ± 91.0 microns, while it was 187.4 ± 48.8 microns for the samples processed at a short sterilization time of 20 minutes. Even though there was a gap of 30.6 microns between the average impression depth value of the two data sets, there was no significant difference ($p>0.05$) in measured impression depth. The standard deviation between the data set of samples had a difference of 42.2 microns. The samples from different sterilization time settings were not equal in variance ($p<0.05$). There was a higher variance ($p<0.05$) in the impression depth value for samples processed at a long sterilization time of 60 minutes compared to at a short sterilization time of 20 minutes.

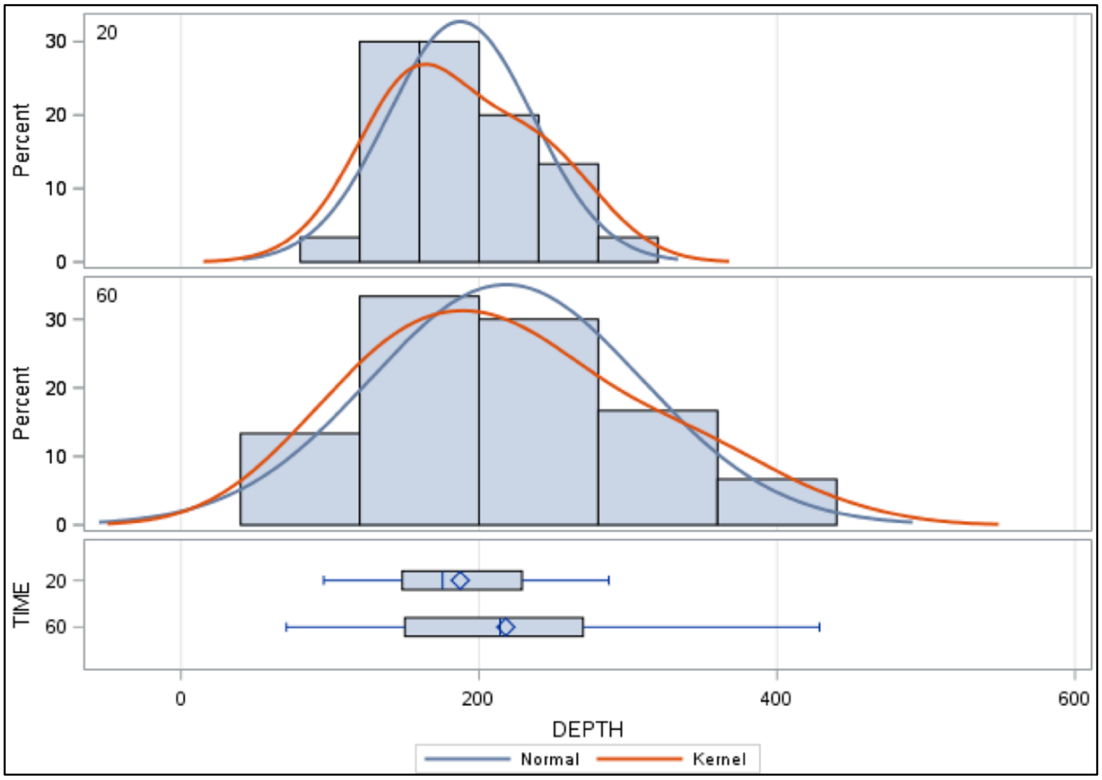


Figure 4.15. Distribution of impression depth values over different sterilization time (20 mins and 60 mins) at high temperature (131°C) and high overpressure (46 psig).

The result from the experiments showed that the longer sterilization time resulted in higher measured impression depth ($p<0.05$) when samples were retorted at a high temperature of 131°C with a low overpressure of 26 psig. This result agrees with a common behavior of polymers when they are exposed to a level of stress for a certain time period, which is called creep or time-dependent deformation.

Creep is the tendency of a solid material to deform permanently under the influence of mechanical stresses. Since creep is the result of the inherent viscoelastic nature of polymers, when the polymers used in flexible pouches are subjected to a constant load, they deform continuously. The materials will continue to deform slowly with time until rupture or yielding causes failure. The degree of creep depends on the type of polymer, exposure temperature, magnitude of applied stress, and exposure time. For example, creep is intensified when polymers are subjected to heat and becomes more severe as they come closer to their melting temperatures (Jansen, 2015; Mantell, 2004).

Therefore, when samples were retorted at a high temperature (131°C) and a low overpressure (26 psig), the combination of a high amount of heat and a high magnitude of stress from internal pressure applied to the flexible pouch resulted in more severe creep behavior or time-dependent deformation. Since the significant difference in impression depth value between short sterilization time and long sterilization time setting was expected in the experiment that has a severe time-dependent deformation, it was found only in the samples retorted at a high temperature of 131°C with a low overpressure of 26 psig.

The results also showed that there were two cases with a difference in variance ($p<0.05$) between the samples retorted at a short sterilization time of 20 minutes and at a long sterilization time of 60 minutes. Case 3.3 and Case 3.4 were both retorted at a high temperature of 131°C, but at different overpressure settings of 26 psig and 46 psig, respectively. Additional analysis showed that when samples were retorted at a high temperature (131°C), the samples retorted for a long sterilization time of 60 minutes had a higher variance ($p<0.05$) of impression depth values than the samples retorted for a short sterilization time of 20 minutes at any overpressure setting. Therefore, a combination of a high retort temperature of 131°C and a long sterilization time of 60 minutes resulted in a high variance of waffling defect severity ($p<0.05$), which means it is a less preferable retort process in terms of quality control (Wachs, 2018).

Relationship Between Retort Processing Factors and Waffling Defect Severity

The relationship between retort processing factors and waffling defect severity was determined using response surface regression. To ensure that a specific range of retort processing conditions was covered in determining the relationship, the data obtained from sixteen retort runs with different combinations of processing factors based on central composite experimental design was used in the calculation. The resulting prediction expression, as seen in Equation 4.1, can be used to predict the average impression depth value (severity of waffling defect) using three processing factors as predictor variables, which are retort temperature (T), overpressure (P), and sterilization time (t).

$$Predicted\ Impression\ Depth = 0.25T^2 + 0.19P^2 + 0.04t^2 - 0.71TP + 0.04Tt - 0.01Pt - 28.32T + 67.86P - 7.86t + 383.73 \qquad (4.1)$$

The hypothesis tests were used to gain an understanding of importance of the linear effect, the quadratic effect, and the effects of the cross-products with each of the other two factors. The results showed that linear and cross-product terms were significant ($p<0.05$) in predicting dependent variable (impression depth), while quadratic terms are not significant ($p>0.05$). Additionally, the factor ANOVA indicated that only retort temperature and overpressure were the factors with significant overall effects on the severity of waffling defects ($p<0.05$), while sterilization time is not significant ($p>0.05$).

This result agrees with the three-dimensional surface response plots, which show that sterilization time is less significant than retort temperature and overpressure in predicting the severity of waffling defects. Each data point on the plots indicates the averaged actual value of impression depth from each retort process combination in this study. The shaded region shows the resulting prediction from the equation. As the predicted values come closer to the actual values, the shaded region comes closer to the points on the plot. The surface response plot (Fig. 4.16) shows the effect of only retort temperature and overpressure on impression depth value. Although the effect of sterilization time is not included in the prediction, the shaded region falls close to all the data points.

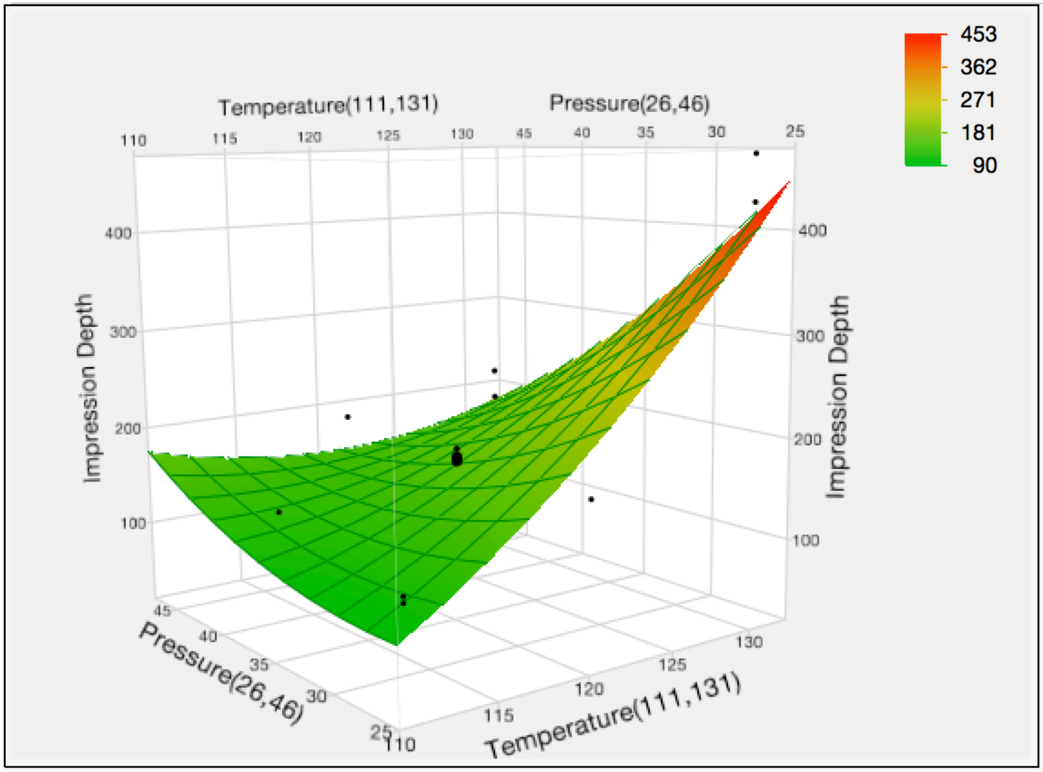


Figure 4.16. Effect of temperature and overpressure on impression depth.

The surface response plot (Fig. 4.17) shows the effect of only temperature and sterilization time on impression depth value. Since the shaded region falls close to the data points when the retort temperature is in a range of 111°C to 121°C and away from the data points when retort temperature comes closer to 131°C, the prediction based only on the effect of temperature and sterilization time is not effective at high retort temperatures. Additionally, sterilization time tends to show less effect on impression depth value, since there is no significant difference in the distance between the shaded region and the data points when sterilization time changes.

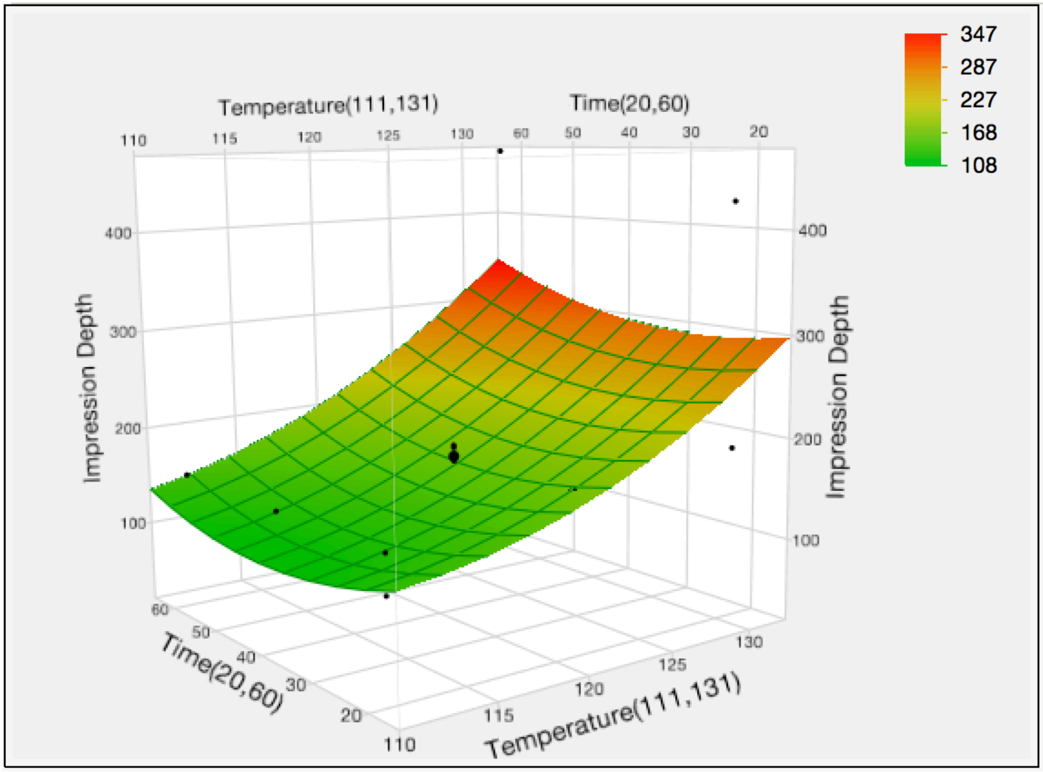


Figure 4.17. Effect of temperature and sterilization time on impression depth.

The surface response plot (Fig. 4.18) shows the effect of only overpressure and sterilization time on impression depth value. Since the shaded region falls close to the data points at a high overpressure of 46 psig and away from the data points when overpressure comes closer to 26 psig, the prediction based only on the effect of overpressure and sterilization time is not effective at low overpressures. In addition, sterilization time tends to show less effect on impression depth value since there is no significant difference in the distance between the shaded region and the data points when sterilization time changes.

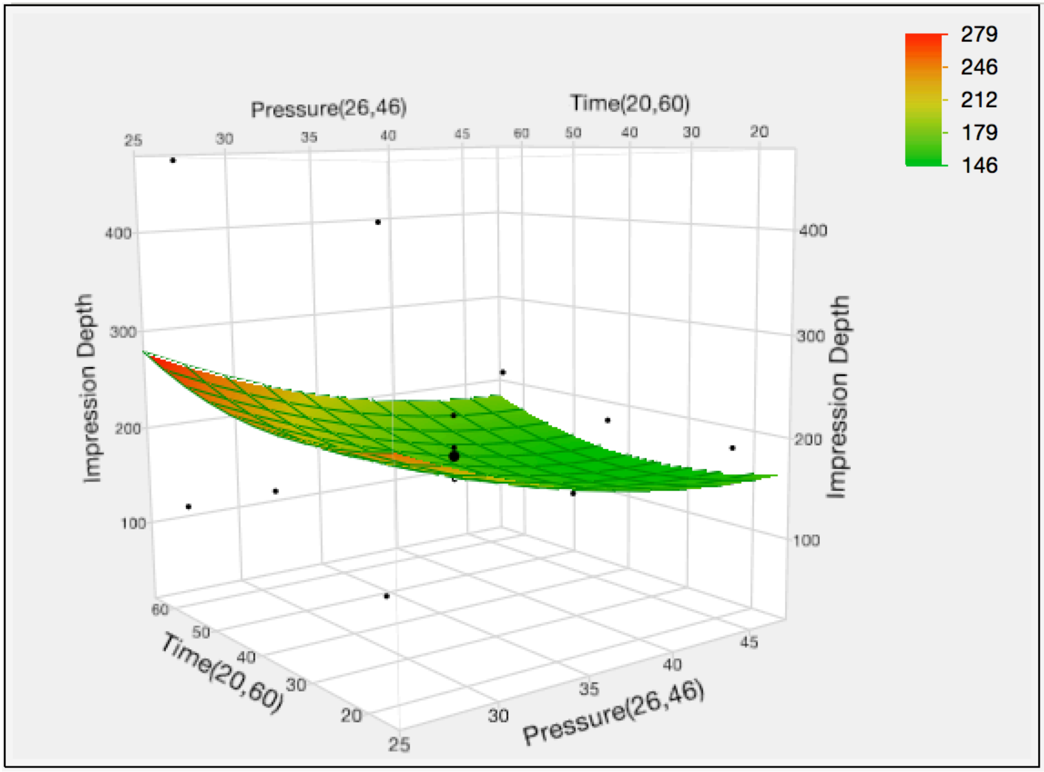


Figure 4.18. Effect of overpressure and sterilization time on impression depth.

However, the lack of fit of the proposed equation was not significant (*p*>0.05), which proved that the resulting equation with all the terms and factors is applicable for predicting impression depth. Therefore, a prediction can be made using this equation and no further experimentation with additional or less variables are required to perform.

In addition to the lack of fit, there are methods to evaluate model accuracy, such as the Actual by Predicted Plot, hypothesis test of overall model (overall F-test), coefficient of determination (R-squared), root-mean-square error (RMSE), and coefficient of variation (CV). The Actual by Predicted Plot (Fig. 4.19) shows the actual impression depth value versus the predicted impression depth value. As the predicted values come closer to the actual values, the points on the scatter plot fall closer around the red line. The shaded region indicates the area of 95% confidence interval of the line, also known as the confidence band (SAS Institute, 2017).

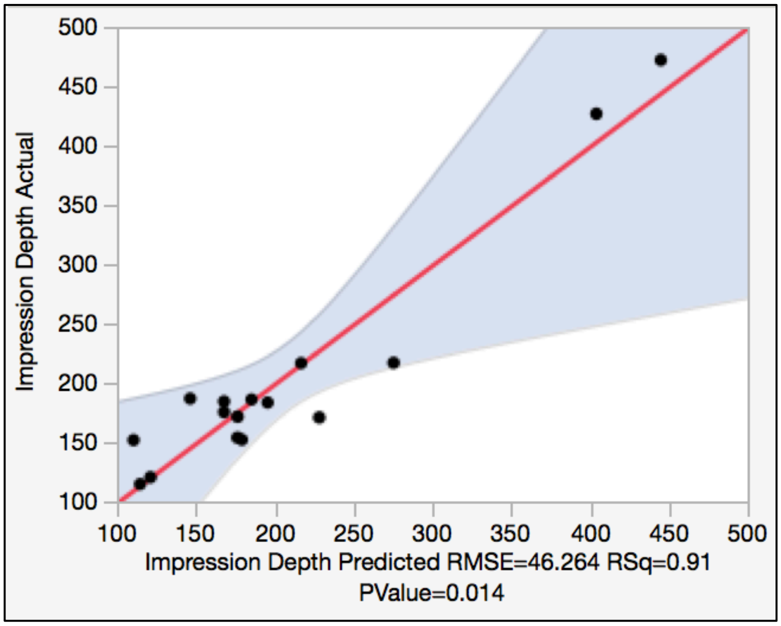


Figure 4.19. Actual by Predicted Plot.

The hypothesis test (overall F-test) determined whether the proposed relationship between the response variable (impression depth) and the set of predictors (retort temperatures, overpressure, and sterilization time) was statistically reliable and useful in its prediction. Since the hypothesis test result showed that the overall model was statistically significant ($p<0.05$), the model predicted impression depth based on the chosen factors well (Grace-Martin, 2018).

Another measure of model accuracy is the coefficient of determination, or R-squared value, which provides an estimate of the strength of the relationship between the regression model and the response variable with a range from 0 to 1. A value closer to 1 means a model is predicting well. This regression model had an R-squared value of 0.91.

However, since the coefficient of determination is fit for use with a completely linear regression model, it should not be used alone to evaluate the regression model in this study (Frost, 2017).

The root-mean-square error (RMSE) is one of the most common statistical measures to indicate the absolute fit of the model to the data by evaluating how close the observed data points are to the model's predicted values. Lower values of RMSE indicate better fit and more accurate the model predicts the response. When the RMSE is normalized by the mean value of the observed data, also known as the normalized root-mean-square error (NRMSE) or coefficient of variation (CV), it becomes unitless and can be used to compare between regression models. The model with the smaller CV has predicted values closer to the actual values. The proposed regression model had an RMSE value of 46.26 microns with a CV of 0.22. Since this model has the smallest RMSE and CV values compared to other models in this study, it predicted the average impression depth value more accurately. Even though both R-squared value and CV are unitless measures that are indicative of model fit, they define model fit in different ways. R-squared evaluates how much of the variability in the actual values is explained by the model, while the CV evaluates the relative closeness of the predictions to the actual values (UCLA: Statistical Consulting Group, 2018).

In summary, the results of the model evaluation methods proved that the proposed equation using three processing factors (retort temperature, overpressure, and sterilization time) as predictor variables was useful in predicting impression depth and no further experimentation with additional or less variables were required.

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CHAPTER FIVE

CONCLUSIONS

Effect of Retort Temperature on the Severity of Waffling Defects

The first objective of this study was to examine the effect of retort temperature on the severity of waffling defects. This study compared and determined the differences in the impression depth values and their variation when different retort temperatures (111°C and 131°C) were used. Higher temperature settings resulted in higher measured impression depth values ($p<0.05$) in all of the tested overpressure (26 psig or 46 psig) and sterilization time (20 minutes or 60 minutes) combinations. This was a result of the heated polymer slowly decreasing its flexural strength. Consequently, the decrease in flexural strength reduced the material's ability to resist deformation from the weight of the product, which presses the pouch surface onto the retort rack.

Also, when samples were retorted for a long sterilization time (60 minutes), the samples retorted at a high temperature of 131°C had a higher variance ($p<0.05$) of impression depth values than the samples retorted at a low temperature of 111°C at any overpressure setting. Therefore, retort processing at a high temperature (131°C) for a long sterilization time (60 minutes) will require a more careful production and quality control due to its high variance of waffling defect severity.

Effect of Retort Overpressure on the Severity of Waffling Defects

The second objective of this study was to examine the effect of retort overpressure on the severity of waffling defects. This study compared and determined the differences in the impression depth values and their variation when different retort overpressures (26 psig and 46 psig) were used. At low temperature (111°C), the higher overpressure resulted in higher severity of the waffling defects ($p<0.05$). If a low overpressure setting is used, the normal stress from internal pressure pulls the material more towards the direction of the normal stress and reduces the influence of the product weight, which results in less surface impression and less severity of the waffling defects. On the other hand, when higher overpressure is used, it results in a lower normal stress and consequently leads to higher severity of the waffling defects due to increased influence of the product weight pushing the bottom surface of the pouch through the holes of the retort rack.

However, a different result was observed at a high temperature (131°C), where deformation due to the loss of flexural strength occurred. In this case, higher overpressure resulted in lower severity of waffling defects ($p<0.05$). This was a result of the pouch's bottom surface deformation that interfered with the ability of the pouch surface to be pulled towards the normal stress direction. This consequently reduced the influence of the normal stress acting against the force of internal pressure and product weight, which resulted in a more severe surface impression. In the case that retort overpressure is introduced, the force from external pressure is applied on the outside surface of the pouch that acts directly against the internal pressure, which causes a severe surface impression.

In addition, the results from the experiments showed no significant difference in variance ($p>0.05$) between the samples retorted at different overpressures. Therefore, the same waffling defect control measures may be used in these retort processes, as long as the overpressure settings are in a range between 26 psig and 46 psig and there is no difference in retort temperature and sterilization time settings.

Effect of Sterilization Time on the Severity of Waffling Defects

The third objective of this study was to examine the effect of sterilization time on the severity of waffling defects. To determine if there were differences in the impression depth values and their variation, different sterilization time settings (20 minutes and 60 minutes) were used in the experiment. Longer sterilization time resulted in higher measured impression depth ($p<0.05$) only if the samples were retorted at a high temperature of 131°C with a low overpressure of 26 psig. There was no statistical difference ($p>0.05$) in measured impression depth in other tested retort temperature (111°C or 131°C) and overpressure (26 psig or 46 psig) combinations.

This result agrees with the behavior of polymers called creep or time-dependent deformation. A significant difference in the impression depth values between short and long sterilization time settings was expected in the condition that allowed a severe time-dependent deformation to occur. Therefore, this significant difference was found only in the samples retorted at a high temperature of 131°C with a low overpressure of 26 psig.

The difference in variance of impression depth values between the samples of different sterilization time settings was also studied. When samples were retorted at a high temperature (131°C), samples retorted for a long sterilization time (60 minutes) had a higher variance ($p<0.05$) of impression depth values than the samples retorted for a short sterilization time (20 minutes) at any overpressure setting. This result agreed with the conclusion of the previous study. Therefore, a combination of a high retort temperature (131°C) and a long sterilization time (60 minutes) will require a more careful production and quality control. This is due to its high variance of waffling defect severity compared to other process setting combinations.

Relationship Between Retort Processing Factors and Waffling Defect Severity

The relationship between retort processing factors and waffling defect severity was explained using a prediction expression as shown in Equation 5.1. The proposed equation can be used to predict the average impression depth value, also referred to as the severity of waffling defects, using three processing factors as predictor variables, which are temperature (T), overpressure (P), and sterilization time (t).

$$Predicted\ Impression\ Depth = 0.25T^2 + 0.19P^2 + 0.04t^2 - 0.71TP + 0.04Tt - 0.01Pt - 28.32T + 67.86P - 7.86t + 383.73 \qquad (5.1)$$

The lack of fit of the proposed equation was not significant (*p*>0.05), which proved that the resulting equation with all the terms and factors is applicable for predicting impression depth. In addition, the hypothesis test (overall F-test) result showed that the overall model was statistically significant (*p*<0.05), which means the model predicts impression depth well based on the chosen factors. The coefficient of determination or R-squared value of this regression model was 0.91, which shows a strong relationship between the regression model and the response variable and indicates that the model can be used to perform a reliable prediction.

In summary, the results of model evaluation methods proved that the proposed equation using three processing factors (temperature, overpressure, and sterilization time) as predictor variables is useful in predicting impression depth and no further experimentation with additional or less variables are required.