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Relating Burst Pressure to Seal Peel Strength In Pouches

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RELATING BURST PRESSURE TO SEAL PEEL STRENGTH IN POUCHES

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ABSTRACT

This work was aimed at studying the relationship between burst pressure and seal peel strength. Past researchers developed a force balance equation based on the analysis of a force diagram to relate burst pressure to seal strength. The theoretical formula $S = P\left(\frac{D}{2}\right)$ (S=Seal Strength, P=Burst Pressure, D=Restraining Plate Gap) has been studied by past researchers with contrasting results.

The theoretical formula was tested by varying dwell time to produce seals of varying strength. Pouches were burst tested at each of these dwell times to obtain burst pressures, burst peeling times, and burst locations. Corresponding peel tests were performed with crosshead speeds that were adjusted to match the peeling time of the burst tests. The peel tests were also performed with adjusted gauge lengths to match the plate gap used in inflation burst testing and compression burst testing.

The data was analyzed by treating the obtained burst pressures as proxy variables for the unobtainable true burst pressures associated with the pouches that were destroyed during peel testing. Dwell time was used as an instrumental variable, and the two-stage least squares method for parameter estimation was used to estimate the slope of the regression.

The estimated slopes of the regressions were compared to the theoretical slope (D/2) for each plate gap using analysis of variance. The results showed that the theoretical relationship suggested by past researchers only worked for a restrained burst test with a plate gap of 0.25in.

Empirical equations were developed from the parameter analysis. The empirical equations were used to calculate predicted burst pressures which were then compared to the burst pressures obtained from testing. The percent difference between the two values ranged from 0% to 28%.

DEDICATION

I would like to dedicate this manuscript to my family. Their support and motivation to continue my education has brought me to where I am today. The comforting support of my mother has gotten me through the tough times and given me the strength to continue. The advice from my father and my sisters has helped me make the correct decisions when needed. I am thankful for my family and all that they have done to help me along the way.

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CHAPTER ONE

INTRODUCTION

Packaging can be defined as the science, art, and technology of enclosing or protecting products for distribution, storage, sale, and use. In early history, packaging was simple and fulfilled the most basic of requirements: to protect, contain, carry, and dispense (Hanlon, Kelsey, & Forcinio, 1998). Packaging consisted primarily of leaves, shells and animal skins used to carry an object from one place to another (Hanlon et al., 1998). However, packaging has grown into a multi-billion dollar industry. It was estimated that worldwide packaging sales reached $485 billion in 2004 (*Market statistics and future trends in global packaging.*2008).

Packaging can be broken down into categories such as "rigid," semi-rigid," and "flexible." These are physical descriptions of packaging and indicate the general packaging form. Flexible packaging is becoming increasingly more common in the marketplace and will continue to grow due to its low cost and ability for greater package design alternatives. Flexible packaging sales were estimated to be $25.5 billion in the United States for 2010 (Flexible Packaging Association, 2011).

The most common style of flexible packaging is the pouch. Pouches are used to contain anything from component parts to food and beverages or medical devices. Pouches can be made from flat and flexible materials including paper, aluminum foil, and plastic films. In most cases, a combination of these materials is required to fully protect the contents from the outside environment. However, a pouch cannot protect and contain the contents without proper seals. Seals are used to form the pouch from the flexible materials, and they are also used to close the pouch after it is filled with its contents.

Seals must be strong enough to withstand any pressure differentials or compression hazards the pouch may encounter. In some cases, the seal must also be peelable to allow for easy opening. There are several ways to test the strength of a seal including inflation burst testing, compression burst testing, and peel testing. Inflation burst testing requires the use of an inflation burst apparatus, which utilizes a needle to puncture a fully sealed pouch and inflate it from the inside. Compression burst testing requires the use of a compression tester to apply a load to the pouch until it bursts. Peel testing requires that one cut a sample of the seal and pull it apart on a tensile tester.

The problem with using different types of tests to determine the strength of a seal is that it is difficult to translate the results of one test into results of another test. There is a need to establish a relationship between peel tests and burst tests in the packaging industry because not all parties involved have access to the same equipment. A pouch maker may not have access to a tensile tester but often has access to a burst tester. A plastic film supplier does not specialize in making pouches and may not have the proper equipment available to produce a pouch for burst testing. Therefore, the film supplier cannot obtain burst pressure values to compare with the pouch maker.

Alternatively, film suppliers often have access to small sealing equipment and tensile testers. This equipment gives them the ability to measure peel strengths of different seals. On the other hand, a pouch filler or pouch maker may only have access to burst testing equipment. The pouch filler may also be more interested in burst testing because it more closely simulates the hazards a pouch will encounter throughout the distribution cycle.

For a film supplier to provide burst pressure data to the pouch maker, or for a pouch maker to provide peel strength requirements to a film supplier, a proper relationship between burst test data and peel test data must be established. The work presented in this document studies a theoretical relationship between the two types of tests derived by past researchers. The equation is based on a force balance model equating the area acted upon during a peel test to the area acted upon during a burst test.

CHAPTER TWO

REVIEW OF LITERATURE

Packaging

Since packaging is such a broad industry, it can be categorized into the following market segments: consumer, institutional, industrial, and military (Hanlon et al., 1998). Consumer packaging is a segment that generally includes packaging for "individual or small family groups" (Hanlon et al., 1998). This segment consists primarily of decorated, small-volume containers, while the institutional segment consists primarily of large, lightly decorated packages (Hanlon et al., 1998). Industrial packaging consists of bulk containers, large drums, corrugated boxes, pallets, crates, and large bags (Hanlon et al., 1998). Lastly, military packaging is a "highly specialized type of protective packaging in which all elements – most particularly product identification and inspection procedures – have been defined by the government and documented in intricate detail" (Hanlon et al., 1998). This is one method of categorizing the packaging industry, but a more simplistic method of categorizing the industry is based on the physical characteristics of packages. For instance, packaging can be reduced to descriptions such as "rigid," "semi-rigid," or "flexible." Rigid packaging is generally comprised of metal or glass and does not bend without permanent deformation or breakage. Semi-rigid packaging has some limited ability to bend and return to original shape. Semi-rigid packaging is typically made of plastic or paperboard. Flexible packaging is usually made of paper, aluminum foil, plastic films, or a combination of the three.

Flexible Packaging

Flexible packaging has been defined as "the manufacture, supply and end conversion of plastic and cellulose films, aluminium foils and papers, which are used separately or in combination for primary (and some secondary) retail food and beverage packaging, plus certain specialist non-food niche sectors, such as tobacco, medical, pharmaceutical and personal care packaging"(Durston, 2006). It should also be noted that flexible packaging can also be present in the tertiary level as stretch or shrink film.

Since the 1970s, there has been a surge in the popularity and use of plastic packaging (Selke, Cutler, & Hernandez, 2004). According to Durston, "Flexible packaging has grown in use over the last few decades, and it is now one of the most widely used forms of packaging, particularly for food products, because of its ability to provide protection and on-shelf appeal in a highly cost-competitive way" (Durston, 2006). Hanlon states, "the fastest growing packaging forms in the world are now, and will be for some time to come, made from flexible materials." It was estimated that, in Europe, the flexible packaging industry had a growth rate of 2% per year in 2001, and about one third of all expenditure on packaging was associated with flexible packaging (Durston, 2006).

However, before a flexible package can be sold to an end user, the materials involved must undergo one or more converting processes (Rauch, 1977). Converting is a process by which a raw or base material is transformed into a material or structure that will provide the necessary physical or chemical properties required for the particular package. According to the Kline Guide to the Packaging Industry, "the converting operations are of two types: (1) coating, laminating, printing or otherwise treating the materials, and (2) the forming of such products as bags, sacks, pouches and envelopes" (Rauch, 1977). These converting processes enhance the properties of the base material to improve "strength, barrier properties to moisture and oxygen, flavor retention, odor resistance, heat-sealability, scuff protection, and machinability" (Hanlon et al., 1998).

Hanlon states that coating is considered to be the "easiest method for improving the characteristics of paper, film, or foil" (Hanlon et al., 1998). He also states, "Coatings also have been applied to many other papers, foils, and plastic films to open up whole new fields of pouch and bag packaging and to enable significant source reduction in the amount of material required to protect and transport both dry and liquid products." Coatings can be separated into four categories: protective overcoats, heat-seal coatings, barrier coatings, and laminating adhesives. In many cases, a single material cannot provide the required barrier, strength, or sealing aspects, and coating is often used to add a desired property to a base material.

Although coating is a common way of achieving desired properties, there are also the options of lamination and coextrusion (Hanlon et al., 1998). Lamination is defined as "the bringing together and bonding of two or more webs by means of some adhesive agent to create a single web" (Hanlon et al., 1998). Lamination can be accomplished in three ways: by means of heat, adhesive, or a combination of the two (Hanlon et al., 1998). The marriage of materials in such a manner results in what is termed a "laminate." A coextrusion, on the other hand, is the combination of two or more plastic materials as they are extruded. When these materials are combined in such a manner, they are often called “structured films” (Hanlon et al., 1998).

According to Hanlon, laminations have been in use since the 1950s, but since the 1980s more combinations of materials have come to the market. The sheer multitude of types of plastic films, paper, and foils has allowed for multiple combinations of materials. Another advantage to the use of flexible packaging is the fact that it has the lowest unit cost of any type of packaging (Selke et al., 2004).

Selke states “the major advantage of flexible packaging is economy.” Flexible packaging is efficient in its use of material and space, which in turn translates into savings on tertiary and quaternary packaging (Selke et al., 2004). The increased utilization of space allows for decreased material requirements for distribution packaging simply because the distribution package can be smaller (Selke et al., 2004). In addition, the storage or warehousing requirements of unfilled flexible packaging is minimal in comparison to unfilled rigid packaging (Selke et al., 2004). For example, the storage of 1000 empty food cans will be much less efficient than the storage of 1000 empty food pouches of the same filled volume. The storage of unfilled pouches can either be on a roll or in a stack, either of which take up little space in comparison to their rigid and semi-rigid counterparts.

Because of advances in technology, combined with the energy and space savings plastics can provide, the use of flexible plastic materials has steadily increased over the past few decades. The ability to provide properties that are unattainable through a single material make coated, laminated, or coextruded materials a viable option for many package designs.

Flexible Pouches and Major Functions of Packaging

Although early packaging was simple and served the basic functions required of it at the time, society has advanced, and longer shelf-lives are needed. In order to achieve longer shelf-lives, the necessity to protect a product from the outside environment became more important. This can be accomplished in a number of ways. Although the permeability of materials is an important factor, without a proper seal, many flexible packages will not be able to protect the product from the outside environment.

Sealing allows for the closure of a package to protect the contents inside from being exposed to the outside environment. In flexible packaging, one way in which this is accomplished is by creating pouches. Hanlon defines a pouch as "a small flexible container made from paper, plastic, or foil materials, which is formed, filled, and sealed on a single machine"(Hanlon et al., 1998). However, Hanlon goes on to state that some machines only fill and seal preformed pouches, and in these cases the pouch is formed on a separate machine, called a form-fill-seal (FFS) machine (Hanlon et al., 1998).

There are several different types of pouches, the most common of which are pillow pouches, stand-up pouches, three-side seal pouches, and four-side seal pouches. Pillow pouches are formed from a single web of film using three seals. A common example of a pillow pouch is a potato chip bag. On a pillow pouch, one seal (back seal) is located at the back while the other two seals are located at the top and bottom. The back seal can either be a fin seal or a lap seal (Figure 1). A lap seal is formed by sealing the inside surface of the web to the outside surface of the web, while a fin seal is formed by sealing the inside surface of the web to the inside surface of the web. The difference between a fin seal and a lap seal is simple to distinguish due to the fact that a fin seal will stick up perpendicularly to the pouch surface, and a lap seal will not (Selke et al., 2004).

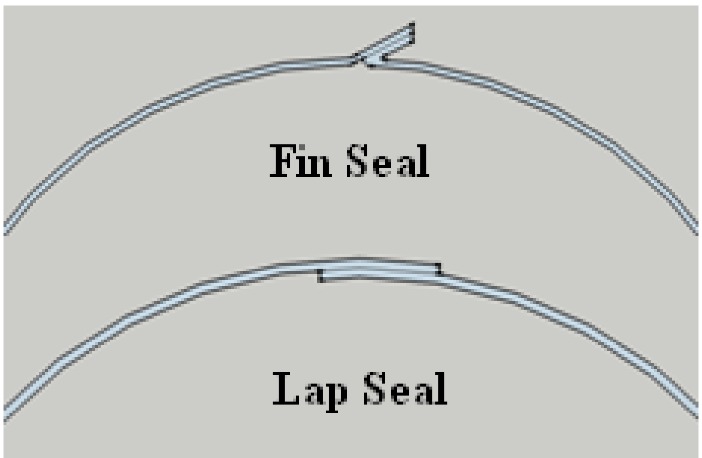

Figure 1. Fin Seal and Lap Seal.

The term "stand-up pouch" refers to the self-supporting structure that allows a pouch to stand in a vertical fashion (Figure 2). These pouches have a bottom gusset, which enables the pouch to form a surface upon which to rest when standing up (Figure 3). Selke describes the bottom gusset in greater detail:

> "Design of the gusset, usually a horizontal bottom gusset, is critical in pouch performance. It can be made from a separate piece of material, sealed to the rest of the pouch, or the pouch material can be folded into a W shape to produce the base. Sometimes the side folds or gussets are used, with an overlapping flat sheet base. Such pouches tend not to stand up as securely as do those containing a

> bottom gusset, so they are used more frequently for dry products than for liquids. In both cases, the design usually depends on the weight of the product to cause the gusset to spread out, so the pouch will stand up only when it contains enough product. If a pouch that can stand up even when empty is desired, a design which provides a rectangular bottom shape, or which can be folded unto such a shape, can be used" (Selke et al., 2004).

Among other reasons, the ability of a stand-up pouch to stand vertically and have a noticeable display area when placed on a store shelf has led to an increase in the use of stand-up pouches. Also, the advancement of closure technology, allowing stand-up pouches to come with threaded closures and zippers has had an effect on the popularity of stand-up pouches (Selke et al., 2004). According to Selke, the market for stand-up pouches grew from 2 billion units in 1998 to over 4 billion units in 2001 and was estimated to reach over 8 billion units by 2006 in North America alone (Selke et al., 2004).

Figure 2. Stand-up Pouch with Zipper.

Figure 3. Bottom Gusset on Stand-up Pouch.

The three-side seal pouch (Figure 4) contains two side seals and a top or bottom seal and is made from a single web of material (Selke et al., 2004). Normally, one side adjacent to the two side seals is folded and does not contain a seal, leaving the third seal to close the pouch, hence the distinction "three-side seal pouch" (Selke et al., 2004). However, Selke states "In some applications, three-side seal pouches are used, but the side with the fold is also sealed, thus blurring the lines between three- and four-side seal pouches" (Selke et al., 2004).

Figure 4. Three-side Seal Pouch.

Four-side seal pouches are pouches often made from two webs of material in which the inside surfaces are sealed to each other on all four sides. Four-side seal pouches allow for the option of using two different materials on each side of the pouch due to the fact that two webs of material are used, however this is not a requirement, and four-side seal pouches can be made from two webs of the same material (Selke et al., 2004).

A common method of forming pouches requires the use of a form-fill-seal machine (Selke et al., 2004). The basic function of a form-fill-seal machine is to form a web into a pouch, which is then filled with product and sealed. The pouches are then cut apart. These processes all occur within the same machine, although the cutting operation can also occur at a separate station (Selke et al., 2004). There is also the option of filling and sealing preformed pouches, in which the preformed pouches have some of the seals but one area is left unsealed until after it is filled with product. In the use of preformed pouches, the pouch is first filled with product, after which the pouch is sealed. The forming and filling operation of preformed pouches is normally accomplished on two separate machines (Selke et al., 2004). Filling and sealing of the pouch can occur on one machine.

There are many different form-fill-seal machines, but they can be classified into one of two groups: vertical form-fill-seal (VFFS) and horizontal form-fill-seal (HFFS). Each of the two classifications is named for the direction in which the pouch moves through the machine (Selke et al., 2004). In VFFS machines, the pouches are formed, filled with product, and sealed, all in a vertical orientation (Figure 5). VFFS machines are preferred when packaging a dry and free-flowing product (Hanlon et al., 1998). Hanlon describes the process of forming and filling pouches in a VFFS machine in greater detail:

> "Here, a single web of film or paper feeds down over a forming shoulder that converts the flat web into a tubular shape around the product feed tube with the two sides of the web overlapping. A vertical heat sealer seals these two edges as the material progresses, product, fed into the tube from volumetric or scale feeders above, falls into the partially formed package, which is then cross-sealed to complete the pouch and severed from the web. The final seal not only forms the closure on the filled package, but also the bottom seal on the next pouch. It should be noted that the cross seals are made through material that varies from two to four thicknesses, the latter in the area of the vertical back seam" (Hanlon et al., 1998).

In HFFS machines (Figure 6), these operations occur in a horizontal orientation (Selke et al., 2004).

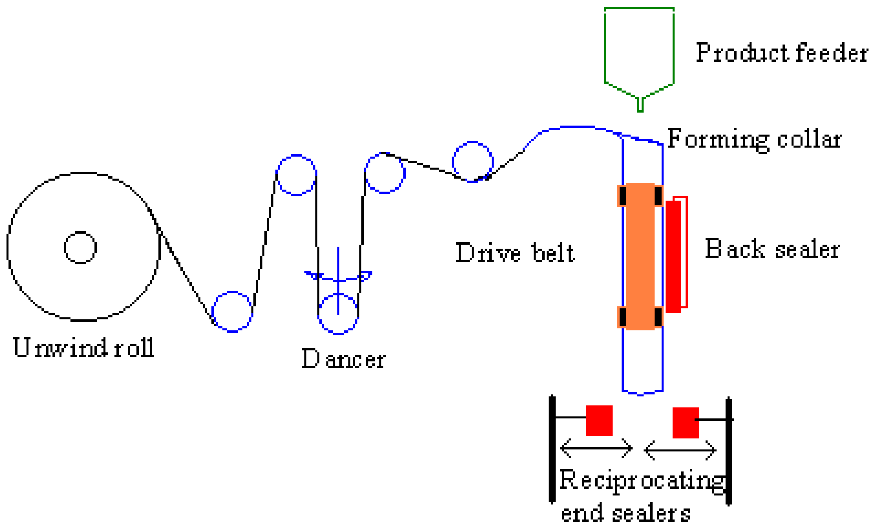

Figure 5. VFFS Schematic.

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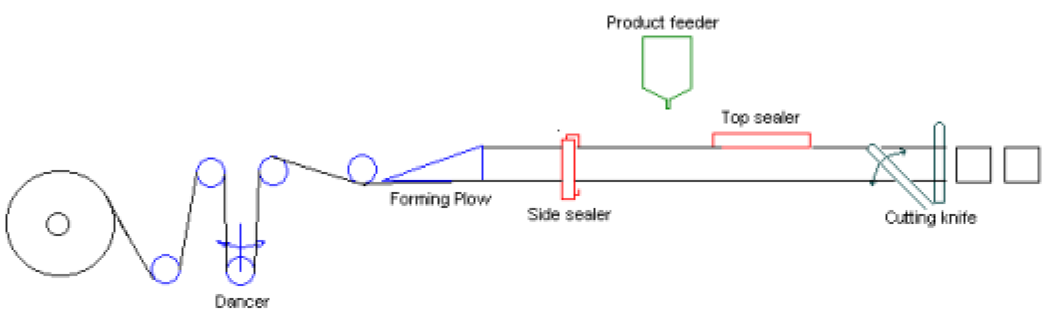

Figure 6. HFFS Schematic.

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Types of Sealing

Sealing is a major component in the formation of a pouch from a flexible web of material. According to Darby, “a seal is a method of closing a package using the flow and adhesion/cohesion properties of at least one plastic material" (Darby, 2011). Each pouch machine uses seals to convert a two-dimensional web to a three-dimensional pouch. Likewise, a pouch filler and sealer will use sealing to close a pouch after filling with product. Sealing technology is complex and encompasses several different methods for creating a seal. The major category of sealing that will be discussed in this document will be heat sealing, which encompasses thermal sealing, impulse sealing, band sealing, hot wire or hot knife sealing, ultrasonic sealing, friction sealing, hot gas sealing and contact sealing, radiant sealing, dielectric sealing, magnetic sealing, and induction sealing (Selke et al., 2004). Heat sealing is "the process by which two structures containing at least one thermoplastic layer are sealed by the action of heat and pressure" (Selke et al., 2004). Heat sealing with flexible structures requires the use of a thermoplastic material at the sealing interface. A thermoplastic material can be reshaped by heat and pressure and can undergo this treatment repeatedly (Selke et al., 2004). In heat sealing, the thermoplastic layer is heated and pressure is applied to create a bond between the two webs of material. The types of flexible materials which can be used in heat sealing are separated into two categories, "supported" and "unsupported" (Selke et al., 2004). Supported flexible structures are structures in which a lamination of at least one non-thermoplastic layer is bonded to at least one thermoplastic layer (Selke et al., 2004). Unsupported flexible structures are structures that consist of one or multiple thermoplastic layers but do not contain a non-thermoplastic layer (Selke et al., 2004).

Heat sealing a flexible structure to create a pouch or another flexible package is often accomplished by applying energy and pressure to the external surface of the flexible structures until the sealing interface begins to flow and molecular entanglement occurs. The most common methods of supplying energy to the inner seal surface are conduction and radiation, of which conduction is most often used (Selke et al., 2004). In addition, convection can also be used to supply energy to the inner seal surface. The flexible structure must have energy applied long enough to cause the sealing interface to heat up to a point where the material begins to flow. Sufficient pressure must be applied to the sealing area to bring the two sides of the seal in intimate contact (Selke et al., 2004). This pressure must be held for a sufficient amount of time, called dwell time, to allow for the thermoplastic layer to flow and create a bond between the two materials (Selke et al., 2004). Finally, the pressure is released after the specified dwell time. At this point, the sealant is often still molten, and the strength of the seal can be measured as "hot tack." Hot tack is the strength of a seal immediately after sealing before cooling has been allowed to take place (Selke et al., 2004).

The most basic heat sealing method is the heated tooling or thermal sealing process (Figure 7). In this process, heated tool surfaces press the two flexible structures together to achieve intimate contact between the sealing surfaces. Simultaneously, the heat from the two sealing tools is conducted into the sealing surfaces allowing the sealing surfaces to soften and fuse into one layer, thus creating a seal. There are several variations of this sealing method, one of which requires that only one of the sealing tool surfaces be heated. Another variation uses heated rollers instead of bars to create seals. In this case, the flexible materials may need to be preheated because the contact time using heated rollers will be much shorter than it would be with hot bars (Selke et al., 2004).

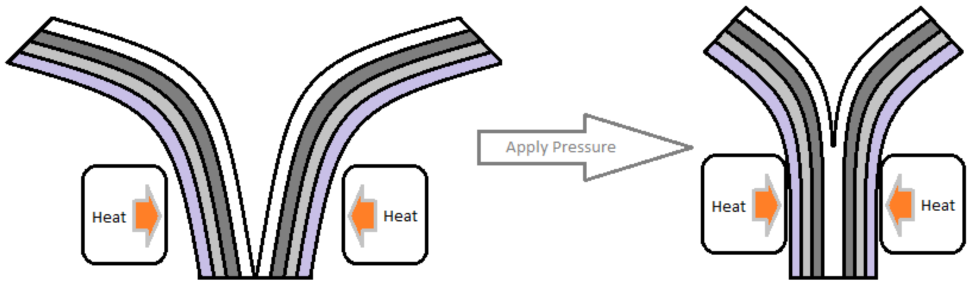

Figure 7. Heated Tooling Sealing.

Impulse sealing is similar to heated tooling sealing in that two seal bars are used to apply pressure and energy to the sealing interface to create a seal. The difference lies in the way the energy is supplied to the interface. In this process, the seal area is heated intermittently by an impulse of electric current which is passed through a nichrome wire ribbon. This method is particularly useful for materials that have very low hot tack, and in some cases, the sealing bars can be cooled by water to allow for quicker cooling of the sealing interface (Selke et al., 2004).

Band sealing is a method of heat sealing in which two moving bands apply pressure to the flexible structure and move the structure along the machine. In this sealing apparatus, the moving bands cause the flexible structure to first pass through a heating zone and then through a cooling zone. This method is useful for sealing preformed pouches, but has the disadvantage of creating wrinkles in sealing area (Selke et al., 2004).

Hot wire or hot knife sealing is a method of heat sealing in which a heated wire or knife is used to seal and cut plastic films apart at the same time. The hot wire or hot knife method is commonly used for Linear Low-Density Polyethylene (LDPE) bags that may be used in applications where a hermetic seal is not required (Selke et al., 2004).

Ultrasonic sealing is a method of sealing in which energy is supplied to the sealing interface using ultrasonic sound waves to vibrate the flexible structures causing friction. The friction causes the sealing surfaces to heat up and begin to melt. This method is useful for applications in which the flexible structure can be damaged by heat or structures that are inefficient to heat the sealing interface using conduction (Selke et al., 2004).

Friction sealing is similar to ultrasonic sealing in that friction is the primary mechanism for creating heat at the sealing interface. However, this method is most commonly used for joining two pieces of rigid or semirigid plastic and is often used for sealing caps to bottles (Selke et al., 2004).

With hot gas sealing, the sealing surfaces are exposed to a flame or heated air. After heating, pressure is applied to the flexible structures using cooled bars to create contact between the sealing surfaces. Contact sealing requires that the sealing surfaces be touched to a heated plate and pressure is applied to bring the surfaces together (Selke et al., 2004).

Radiant sealing is another method of heat sealing which is mostly used for materials that cannot withstand high pressure. This method requires that the sealing surfaces be heated by radiation instead of convection or conduction (Selke et al., 2004).

In the medical device industry, this is a method that is commonly used to seal Tyvek™, which is a spun-bonded high density polyethylene (HDPE) material. It may also be used to seal oriented materials or materials that normally do not seal without a coating (Selke et al., 2004).

Dielectric sealing uses an oscillating high frequency electrical field to apply energy to the sealing surface. This method is only useful for sealing polar flexible materials such as Polyvinyl Chloride (PVC). The mechanism that causes the sealing surface to heat up is explained by the fact that the polar molecules in the material begin to oscillate or bend back and forth because of the oscillating high frequency electrical field attracting the molecules in one direction and then the other very rapidly. This rapid oscillation of the molecules causes a release of energy in the form of heat which is then used to create the seal (Selke et al., 2004).

Magnetic sealing is another method of sealing which uses the oscillation of particles to generate heat. The mechanism by which this method creates heat at the sealing interface involves the use of an oscillating magnetic field which causes magnetic iron particles to oscillate rapidly to create heat for sealing. However, this method does not work on materials that do not have magnetic particles (Selke et al., 2004).

Induction sealing also uses a magnetic field. However, in this case, the magnetic field is used to "induce an electric current in any metal within the field, most often a layer of aluminum foil" (Selke et al., 2004). This electric current actually causes the metal foil to become hotter, which, in turn, heats an adjacent heat seal layer by conduction to create the seal (Selke et al., 2004). It is commonly used for sealing tamper-evident seals on bottles (Selke et al., 2004).

Hazards That Seals Must Withstand

In flexible packaging, there are many hazards that seals must endure before delivery to the consumer. Hazards that can affect the seals on flexible packaging can occur during shipping and transportation, storage / warehousing, and filling / processing. Seals must endure many processes before even leaving the processing facility. For example, on a VFFS machine, the bottom seal of the pouch must be able to withstand the initial stress placed on the sealing area by the product being dropped into the pouch. In a VFFS operation the seals must have good hot tack or they must be allowed enough time to cool before the product is dropped into the pouch to avoid breaking or peeling of the seal. In some cases, the weight of the product alone can be enough to initiate a peel in the sealing area. In retort operations, the seals on pouches must be able to withstand high temperatures and pressures experienced during thermal processing. Another filling operation that can become problematic for seals is hot filling. Hot filling is process by which a liquid product is heated to a temperature at which most of the harmful microbes are killed, and the package (or pouch) is filled. The hot liquid is used to kill any microbes on the package material as well. The heat of the liquid can cause the sealing area to soften and become weaker. In addition to filling operations, pouches must also endure packing operations. This may include hand packing or machine packing. During packing, the seals of the pouches must be able to withstand significant stress when dropped or placed into a corrugated container.

Once the pouches are packed into a corrugated container, the seals must withstand yet another process before leaving the facility. The packed corrugated containers are placed onto a pallet either by machine or by hand. During this process the corrugated containers may encounter shock in the vertical direction or side direction when being placed onto a pallet. Then, the pallet is stretch wrapped and/or banded. After stretch wrapping and/or banding, it is loaded into storage or a trailer for transport to another facility. During the loading or storage operations, the entire pallet can encounter shock horizontally or vertically from fork lift trucks lifting and placing pallets in the desired location. Shock and vibration are also experienced by the pouches inside the corrugated containers, and the pouch seals must be able to withstand this shock and vibration. Pouch seals must also be able to withstand long term static loading conditions when placed in storage. Static loads can be placed on the pouch by anything resting on top of it. For instance, it may be that several pouches are stacked on top of each other when placed in the corrugated container. The bottom pouch must have seals that are able to withstand the force of several pouches placed on top of it without bursting or peeling to a significant degree.

Likewise, the pouch seals will be required to withstand shock and vibration during transport. While the entire pallet is on a trailer and is being transported from one facility to another, the pallet will experience shock and vibration due to the truck and the road upon which it is traveling. Road hazards such as potholes and bumps in the road, as well as acceleration and deceleration of the truck, can cause vertical and horizontal shocks. Acceleration, deceleration, and turning can cause pallets to shift or slide into each other. To prevent shifting and sliding of pallets, proper blocking and bracing with appropriate dunnage material and strapping must be used. Shifting of the pallets can cause horizontal shock, which can be transferred to the pouches. Pouch seals must also be able to withstand dynamic loads. The magnitude of the dynamic load encountered by a pouch is dependent upon the weight of anything on top of the pouch and the shock encountered.

Vibration is also a major concern during transit and can be a cause for failure of a seal. All objects have a natural frequency, which is the frequency at which an object will oscillate with large amplitude even with small external inputs (Hanlon et al., 1998). Packages in trailers experience vibrational frequencies anywhere from 0.08 Hz to 500 Hz during transport (Hanlon et al., 1998). It is possible that a pouch or the packaged product may experience its natural frequency during transport, so the seals must be strong enough to withstand the forces that will be placed on them during this vibration cycle.

However, vibration and shock are not the only concerns during transportation. Some pouches may be sealed at a low altitude and transported across a higher altitude. Such is the case with pouches that may be flown by plane to their destination. There is a change in atmospheric pressure associated with altitude changes. This change in atmospheric pressure can cause a pouch to burst if the seals are not strong enough to withstand the pressure differential of internal pressure to external pressure.

Methods of Testing Pouch Seals

There are many methods for testing seal strength and integrity in flexible pouches. It is important to know the strength and integrity of a seal for ensuring product safety and usability of the pouch. Different applications may require different types of seals. One method for categorizing seals is by grouping them into categories of "peelable" and "fusion" seals.

Seals have several different modes of failure. In a seal with adhesive peel failure (Figure 8), the sealant layer remains adhered to one side of the flexible material and does not split internally. In cohesive peel failure (Figure 9), the sealant layer splits internally, sticking to both sides of the flexible structure. Delamination (Figure 10) occurs when the bond between the sealant layer and the other side of the pouch is stronger than the bond created by the adhesive during lamination or coextrusion. Material break (Figure 11) occurs when the sealing area is stronger than the tensile strength of the material. This can also occur away from the sealing area (Figure 12). Material elongation occurs when the material reaches its yield and begins to stretch (Figure 13). Material elongation can also occur with a slight peel (Figure 14). A material break or elongation is the point at which a seal is no longer considered peelable and is instead considered a fusion seal (*ASTM standard F88*2009).

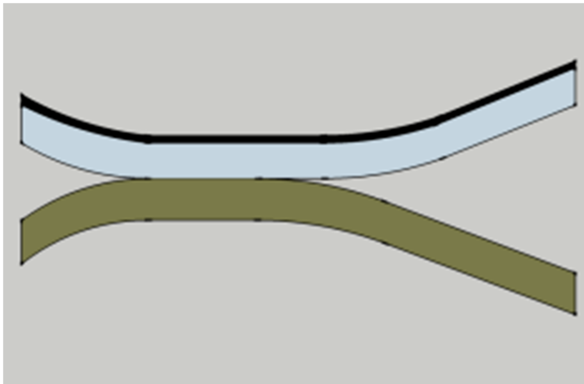

Figure 8. Adhesive Peel Failure.

Drawing adapted from ASTM F88 Figure 4 (*ASTM standard F88* 2009).

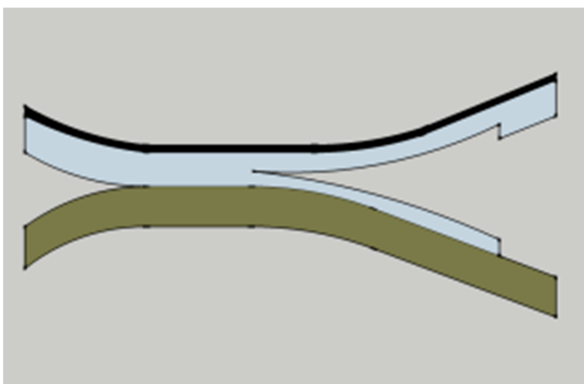

Figure 9. Cohesive Peel Failure.

Drawing adapted from ASTM F88 Figure 4 (*ASTM standard F88* 2009).

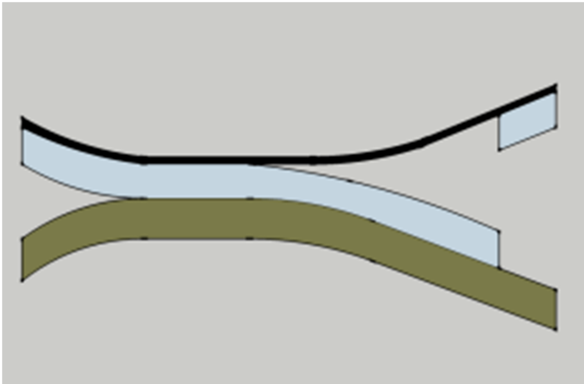

Figure 10. Delamination Failure.

Drawing adapted from ASTM F88 Figure 4 (*ASTM standard F88* 2009).

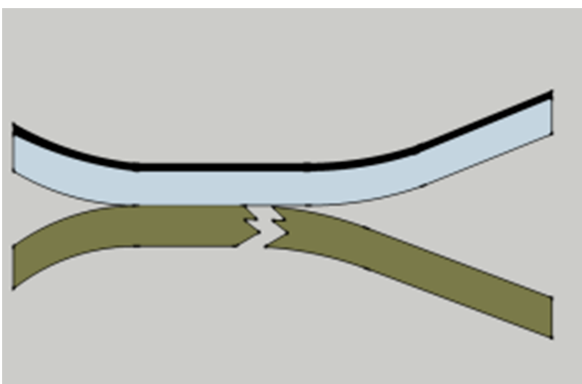

Figure 11. Material Break Failure.

Drawing adapted from ASTM F88 Figure 4 (*ASTM standard F88*2 009).

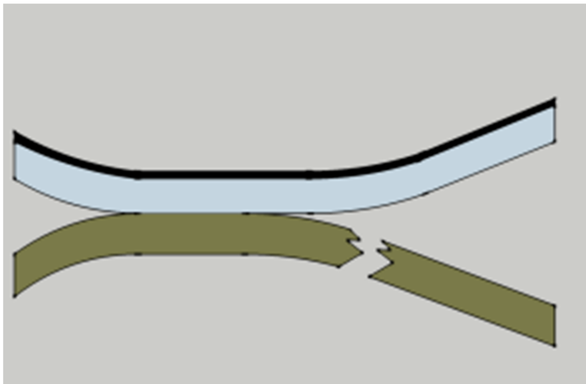

Figure 12. Remote Material Break Failure.

Drawing adapted from ASTM F88 Figure 4 (*ASTM standard F88* 2009).

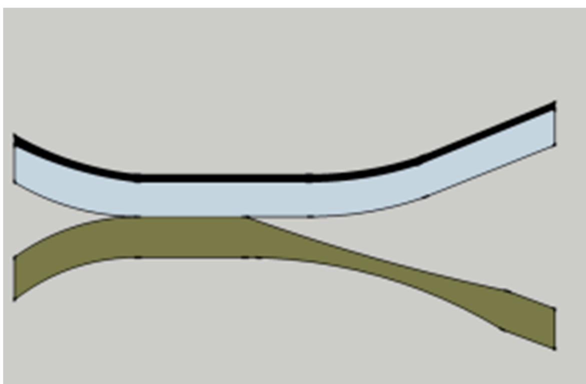

Figure 13. Material Elongation Failure.

Drawing adapted from ASTM F88 Figure 4 (*ASTM standard F88* 2009).

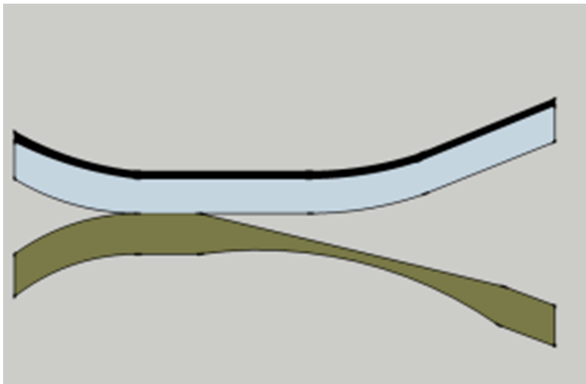

Figure 14. Material Elongation with Slight Peel.
Drawing adapted from ASTM F88 Figure 4 (*ASTM standard F88* 2009).

To test both peelable seals and fusion seals, several test methods have been developed, which can be categorized into burst tests and peel tests. Burst tests use a pressure differential between the outside atmosphere and inside of the pouch to cause a failure in the seal. Peel tests pull sections of the sealing area apart and measure the force required to do so. When using either type of test, it is important to capture the strength of the seal and the mode of failure.

The Standard Test Method for Seal Strength of Flexible Barrier Materials (ASTM F88) is a method in which one-inch wide by (at least) three inch long samples of the sealed material are pulled apart. ASTM F88 specifies that the samples be pulled apart at a constant rate between 8 inches per minute and 12 inches per minute. The resulting force required to pull the sealed sample apart is plotted versus the change in the jaw separation distance of the machine. To accomplish this, the sample is loaded into a tensile testing machine. ASTM F88 specifies a tensile testing machine as “a testing machine of constant rate-of-jaw-separation type" (*ASTM standard F88*2009).The tensile testing machine is then run at the required rate of jaw separation to determine the force required to pull the seal apart.

ASTM F1140, "Standard Test Methods for Internal Pressurization Failure Resistance of Unrestrained Packages," is a set of standards that specifies three test methods used for burst, creep, and creep to failure testing. Since the focus of this study is relating burst pressure values to peel strength, the Test Method A (Burst Test) is the only test method of importance. Test Method A presents two testing apparatus: an open package tester (Figure 15), and a closed package tester (Figure 16) (*ASTM standard F1140* 2007). In Test Method A using an open package tester, the pouch is placed in the apparatus with or without product inside. As the names indicate, an open package tester is used to burst test a pouch with one opened side, whereas a closed package tester is used to burst test a pouch that has all sides sealed.

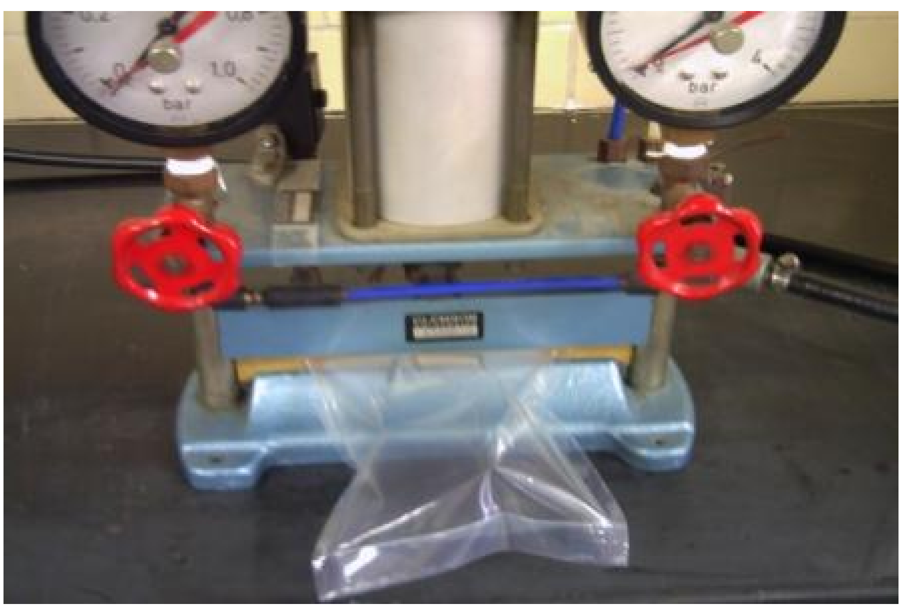

Figure 15. Open Pouch Inflation Burst Tester.

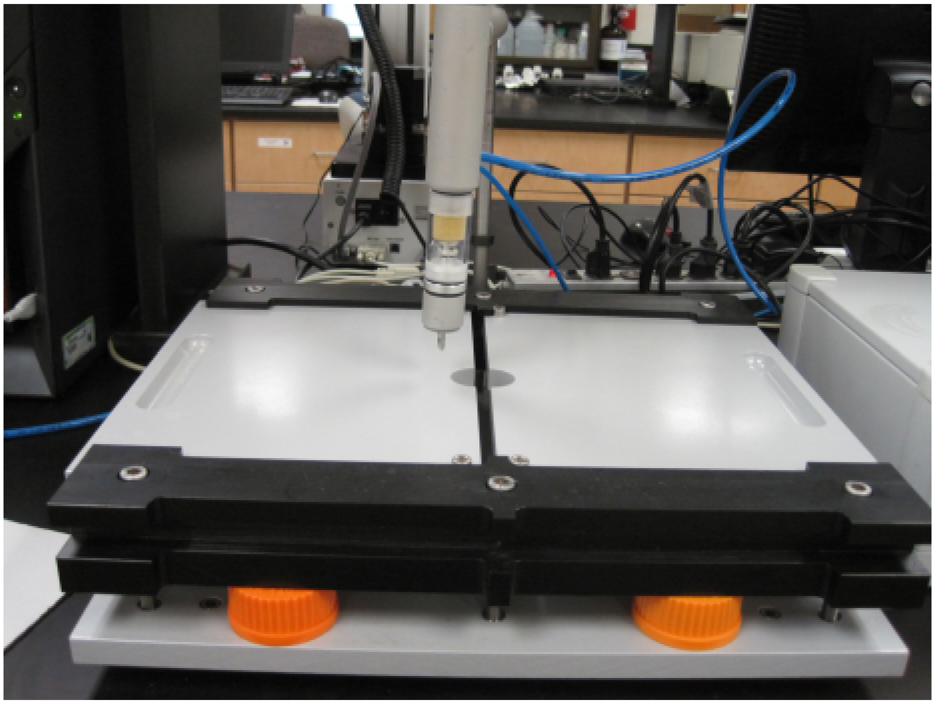

Figure 16. Closed Pouch Inflation Burst Tester.

With the use of an open package tester, the opened side of the pouch is placed between the clamps of the apparatus so that the inflation needle is between the two sides of material. Once placed in the clamp, the testing can begin, which includes inflation of the pouch at a specified rate until burst occurs. This inflation rate should not be greater than the response rate of the pressure indicator to prevent error in final burst pressure readings. There are three configurations used with this type of testing apparatus, which include unsupported, supported by hand, and supported by a plate. In the use of the unsupported configuration, the pouch is left untouched after being placed in the clamp and does not have anything to support it from underneath. ASTM F1140, specifies the supported configurations and states that supported methods limit curling of pouches during testing.

The use of a closed package tester requires that the pouch be completely sealed before testing. In this case, the pouch must be punctured in the center of one of the flat faces of the pouch to allow for insertion of the inflation needle. Once the needle is inserted into the pouch, the inflation of the pouch can occur at a specified rate. The rate of pressurization cannot be greater than the response rate of the pressure indicator to prevent any error in the reading of the final burst pressure.

ASTM F1140 requires that a number of things be reported for each test. First, the type of test must be recorded whether it is a burst test, creep test, or creep to failure test. Another important factor is the testing apparatus used as well as the settings used such as inflation rate. The materials used in the pouch are also of importance, as well as the conditioning parameters and final burst pressure of the pouch.

ASTM F2054, "Standard Test Method for Burst Testing of Flexible Package Seals Using Internal Air Pressurization Within Restraining Plates," is similar to ASTM F1140 but differs in one key aspect. ASTM F2054 requires the use of restraining plates, which are plates used to prevent expansion of the pouch. According to ASTM F2054, this test method has a distinct advantage over ASTM F1140 due to the inclusion of restraining plates (*ASTM standard F2054* 2007). In ASTM F1140, the pouch is unrestrained which means the maximum stress to the pouch occurs where the diameter of the pouch is largest (*ASTM standard F1140* 2007). To focus the stress on the seal area, ASTM F2054 includes the use of restraining plates with different gap specifications.

ASTM F2054 has included guidelines for determining the necessary restraining plate gap. These guidelines specify three options for restraining plate gaps: 25.5mm, 12.7mm, and 6.5mm. These plate gaps must be selected so that at least 60 percent of the surface area of the inflated pouch is in contact with the surface of the restraining plates. To calculate the amount of surface area of the inflated pouch in contact with the restraining plates, the test method specifies Equation (1) below.

$$Z = \left[\frac{x - \left(\pi \times \frac{D}{2}\right)}{x}\right] \times 100 \tag{1}$$

Where:

Z = Percentage of Inflated Pouch Surface in Contact with Restraining Plates

W = Package width dimension (inner dimension from seal to seal),

L = Package length dimension (inner dimension from seal to seal),

D = Plate gap dimension selected,

x = The lesser value of W or L, and

π = 3.141593

Solving Equation (1) for *Z* gives the percentage of the surface of the inflated pouch in contact with the restraining plates. To solve the equation, the plate gap dimension, *D*, must be selected first. If the resulting percentage is greater than 60 percent, the selected dimension is acceptable for testing.

Another testing standard used to burst pouches is one that involves compression instead of internal pressurization. ASTM D 642, "Standard Test Method for Determining Compressive Resistance of Shipping Containers, Components, and Unit Loads," is a testing standard intended for use on corrugated containers and unit loads but is often used in the industry for burst testing of pouches. In such a case, a fixed platen compression tester is used to place a compressive force on the pouch until burst occurs, and the resulting force is recorded (*ASTM standard D642* 2000).

Finally, vacuum burst testing is usually used for leak testing of pouches, but can be used to test burst pressure if the seals are weak enough to fail under the pressure limitations of a vacuum burst testing apparatus. This disadvantage testing is that the apparatus can only create a maximum pressure differential of around one atmosphere. This limitation requires that the seals be weak enough to fail at pressure differentials less than or equal to one atmosphere. In this test, the pouch is placed inside the vacuum chamber and a vacuum is pulled until the pouch bursts. The resulting vacuum pulled up to the bursting point is the burst pressure recorded.

The Need for a Regression Model

Since peel tests and burst tests result in two different measurements for evaluating a seal failure, they each have their specific uses. Burst tests have the advantage of determining the weakest sealing area of a pouch and simultaneously determining the strength of the seal at that location. Burst tests more closely simulate hazards that a pouch may encounter in the distribution, storage, and end-use environment than peel tests. For example, filled and sealed pouches that are placed in a corrugated box will have the weight of the other pouches on top of them, which will create a compressive force. In this case, a compression burst test may be more appropriate for simulating the hazard that these pouches encounter.

On the other hand, pouches that are filled at atmospheric or elevated pressure and transported by air to another location will encounter another type of pressure differential. This occurs because atmospheric pressure decreases as altitude increases. If the atmospheric pressure is less than the pressure inside the pouch, there will be stress placed on the inside surface of the pouch. This stress can cause failures in the seal area, which results in leaking or bursting of pouches. Also, if a pouch were filled with a higher internal pressure than atmospheric pressure, then there would be a similar pressure differential. In this case, the outside pressure would less than the inside pressure even at the same altitude as it was filled. Inflation burst tests more closely simulate pressure differentials than peel tests.

The peel test may be more useful for a company that does not have the ability to make pouches or does not specialize in making pouches. For example, pouch makers will buy the film to make pouches from a film converter. A film converter laminates two or more films together to create one film structure. This film structure is then sold to a pouch maker to process into pouches. In order to provide a pouch maker with technical data regarding the sealability of the material, the film converter often performs peel tests. There are a few reasons why a film converter may choose to use peel tests for determining the sealability of their material. For one, a film converter does not specialize in making pouches and may not have the proper equipment or enough time on hand to make pouches. On top of that, the film converter may not have the equipment to burst test pouches. Additionally, the film converter requires little equipment to create a seal and peel test it. It also requires less material to quickly create a seal versus creating pouches on a pouch-making machine. Peel tests can also be used to create a seal curve. Seal curves help a film converter predict how the material will behave on a pouch making (or other packaging machines) when sealed at several different temperatures, pressures, and dwell times.

A problem occurs when a pouch filler purchases material from a film converter because there is a disparity in the methods of evaluating the sealability of the material. While the film converter often provides data to the pouch filler based on the peel strength values, the pouch filler needs to know what that data means in terms of pouch burst pressures. The lack of agreement on the types of tests to use has created a need to establish a relationship between burst pressure and seal peel strength so that a pouch filler or film converter can easily predict one value from the other.

Past Research

Past researchers have just "scratched the surface" of this topic and have come up with different ways to determine the relationship between burst tests and peel tests. One concept all past researchers used to determine the relationship between the two tests is hoop stress. Before one can understand hoop stress, it must be understood how hoop stress applies to pouches.

A pressurized pouch can be thought of as what engineers refer to as a "thin-walled pressure vessel." A thin-walled pressure vessel is a pressure vessel with a wall thickness that is up to 10 percent of the diameter. To determine the stress in the wall of a thin-walled pressure vessel, the thin-walled assumption is applied and the simplified equation (2) is used.

Hoop Stress in Thin-Walled Pressure Vessels

$$\sigma_H = \frac{RP}{T} \tag{2}$$

Where:

σ_H = Hoop Stress (PSI)

R = Radius of the Pressure Vessel (inches),

P = Internal Pressure (PSI),

T = Thickness of the Wall of the Pressure Vessel (inches)

Hoop stress (σ_H) occurs in the circumferential direction and not in the longitudinal direction, as can be seen in Figure 17. It is a measure of the force the wall of the vessel (in this case the pouch) receives across an area. The reason this stress is applicable is because at the time of burst, an idealized pouch forms a cylinder. If the sealing area were isolated, it could be seen that the sealing area would form a semi-cylindrical shape at burst Thus, the principles of hoop stress may apply. This approach is used to develop force diagrams that explain the stresses a seal will encounter during bursting situations.

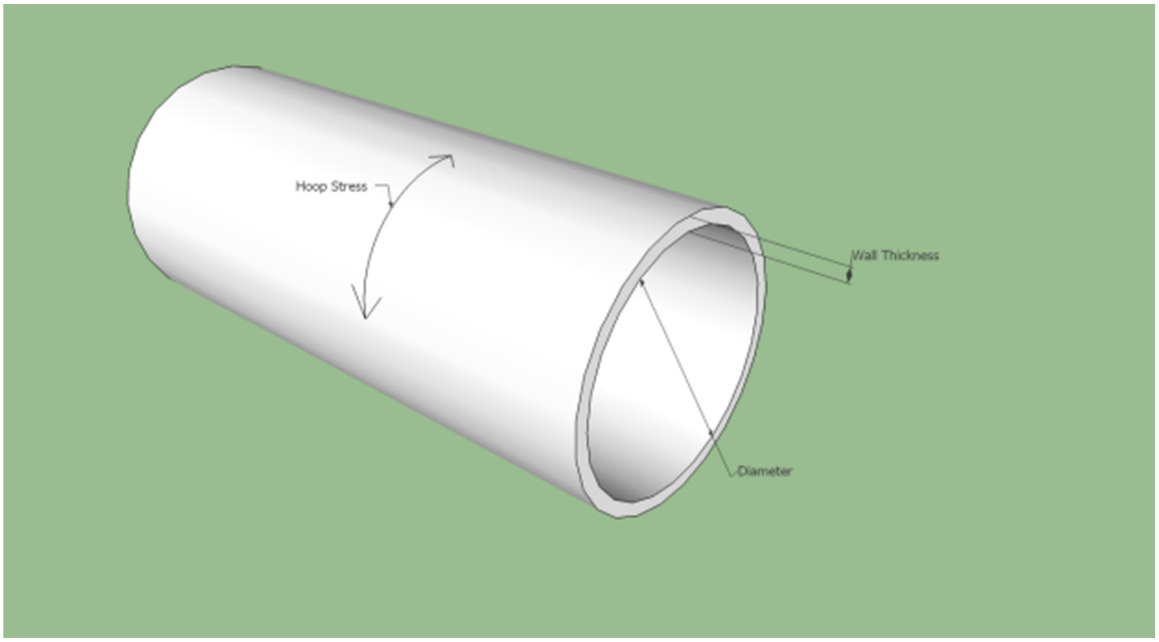

Figure 17. Pressure Vessel and the Associated Stresses.

The first attempt made to determine the relationship between burst pressure to peel strength involved what Thomas P. Wachala regarded as an ideal pouch with a designed peel strength of 1 lbf/in. According to Wachala, the medical device industry has established a “de facto minimum peel force standard of one pound per inch”(Wachala, 1991).

Wachala states that to establish the relationship between burst pressure and peel strength, “one must equate a force acting on a surface with a force acting on a line.” In order to do this, Wachala analyzed a force diagram (

Figure 18) of an “ideal package with an infinitely small height and a rigid, nondeformable surface”(Wachala, 1991). As can be seen from the diagram, the package has a length denoted as L and width denoted as W. This results in the package having a seal perimeter of 2 (L + W). Analyzing the force diagram in

Figure 18 will result in Equation

(3) for the force action upon the entire seal (Wachala, 1991).

$$F_{seal} = 1\frac{lb}{in}\ x\ perimeter$$

$$= 1\frac{lb}{in}\ x\ 2(L + W) \tag{3}$$

Where:

$\mathrm{F_{seal}} = \text{The force acted upon the seal } \left(\frac{\mathrm{lb}}{\mathrm{in}}\right)$

$\text{Perimeter} = \text{Length(in)x Width (in)}$

$\text{L} = \text{Length (in)}$

$\text{W} = \text{Width (in)}$

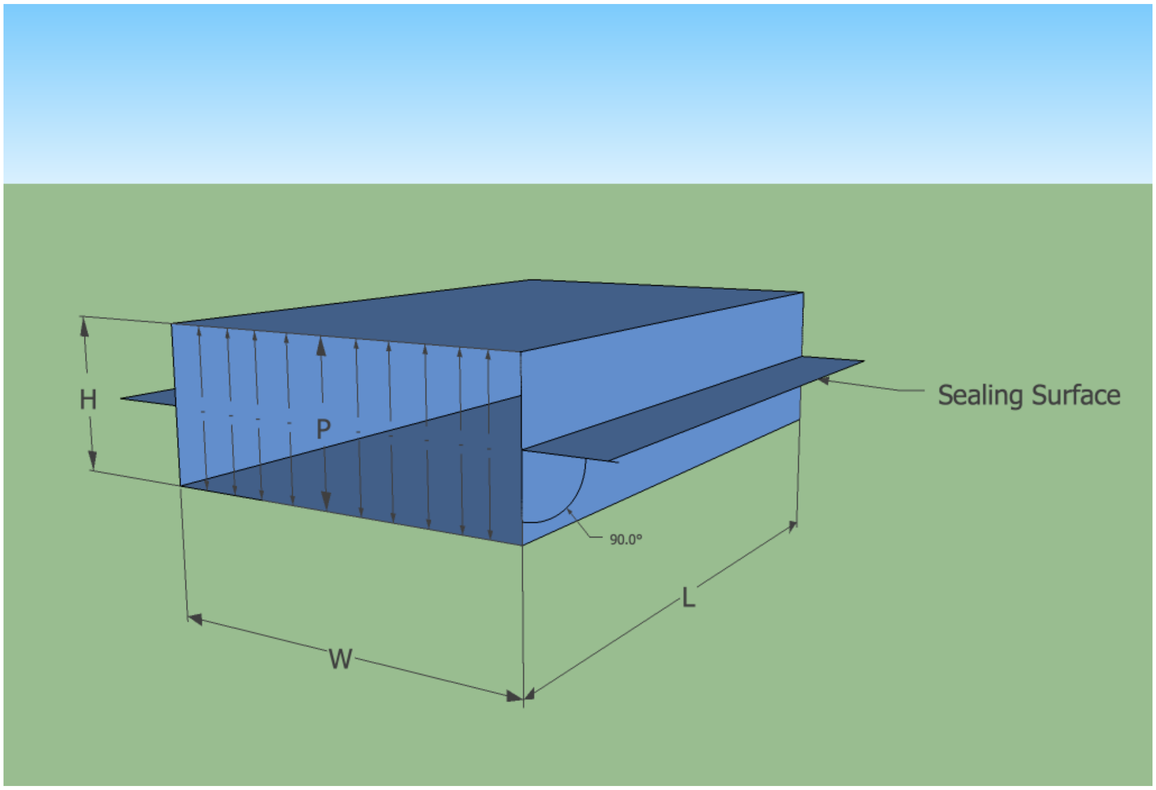

Figure 18. Force Diagram.

Adapted from Wachala's Figure 2 (Wachala, 1991)

Wachala also developed Equation (4) based on

Figure 18 for the force acting upon the surface of the package. His derivation is based on the assumption that when "the pressure is increased to the edge of seal failure, the rigid container is in equilibrium and the force attempting to pull the package apart is balanced by the seal strength." Accordingly, he set Equation (3) equal to Equation (4) to derive Equation (5) for his relationship between burst pressure and peel strength.

$$F_{surface} = P \; x \; area$$

$$F_{surface} = P\ x\ (L\ x\ W) \tag{4}$$

Where:

P = internal pressure (PSI)

Area = Length (in) x Width (in)

L = Length (in)

W = Width (in)

Derivation of Equation (5)

$$F_{seal} = F_{surface}$$

$$1\frac{lb}{in} x\ 2\ (L + W) = P\ (L + W)$$

$$1\ x\ 2\ (L + W) = P\ (L\ x\ W)$$

$$P = \frac{2\ (L + W)}{(L\ x\ W)}$$

$$P = \frac{Perimeter}{Area} \tag{5}$$

Where:

P = Burst Pressure

L = Length

W = Width

Unfortunately, pouches are not comprised of rigid materials, so material deformation comes into play. In fact, Wachala noted that pouches will take on a more curved shape when inflated. Wachala then considered another ideal situation in which an inflated pouch forms a circular cross-section seen in

Figure 19. In this case, the tangent to the sealing area is 90°, and the width of the pouch is now equal to the diameter of the circle (Wachala, 1991). The original width of the deflated pouch becomes half the perimeter of the deflated pouch (Equation (6)). Accordingly, the circumference of the circular cross-section is found by using Equation (7). Setting Equation (6) equal to Equation (7) and solving for the diameter (D) will result in the equation for the diameter of the circular cross-section (Equation (8)).

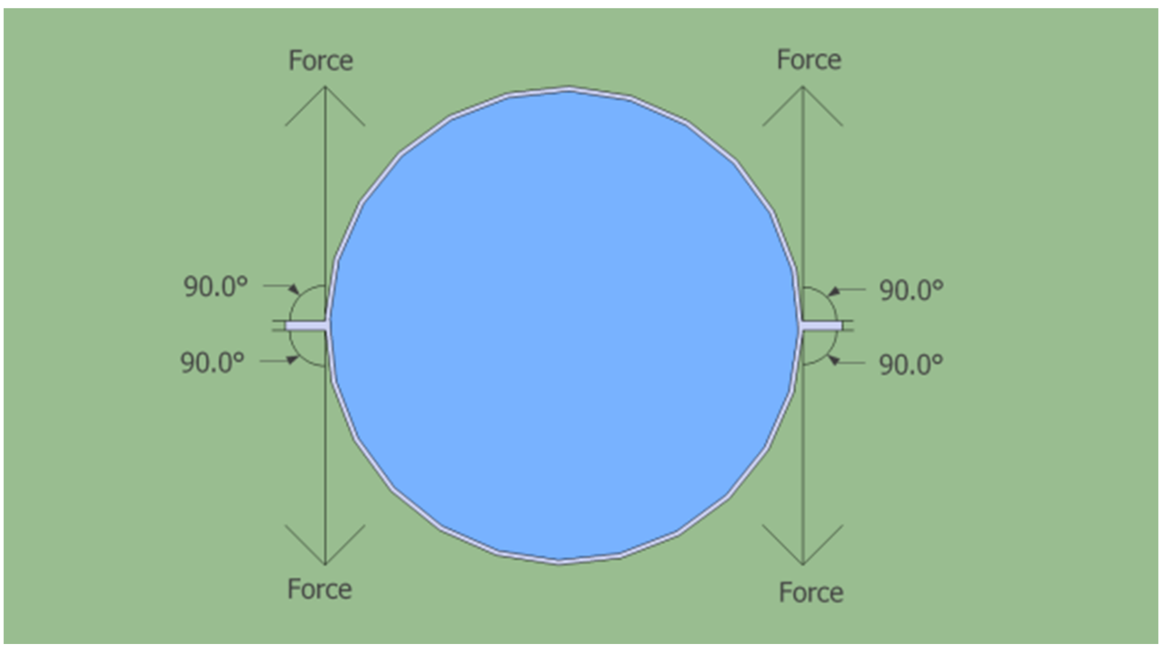

Figure 19. Cross Section of an Ideal Pouch.
Adapted from Wachala's Figure 4 (Wachala, 1991)

(6)

Where:

W = Width (in)

(7)

Where:

D = Diameter (in)

(8)

Where:

W = Width (in)

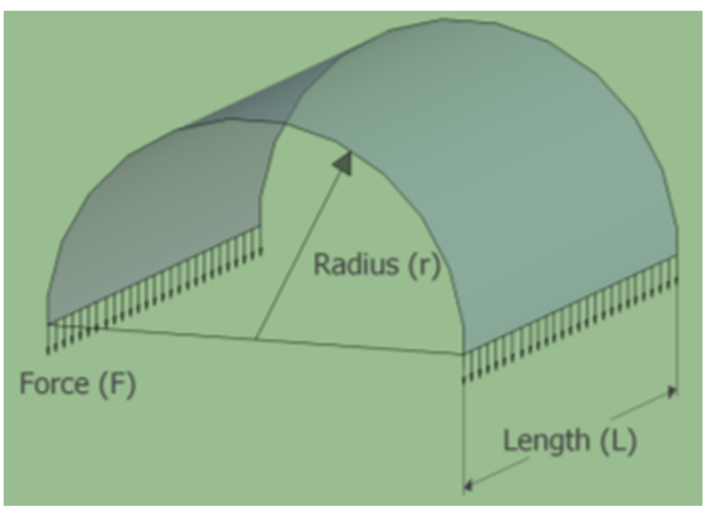

Figure 20. Stress in Thin-walled Cylinders.

Adapted from Wachala's Figure 5 (Wachala, 1991)

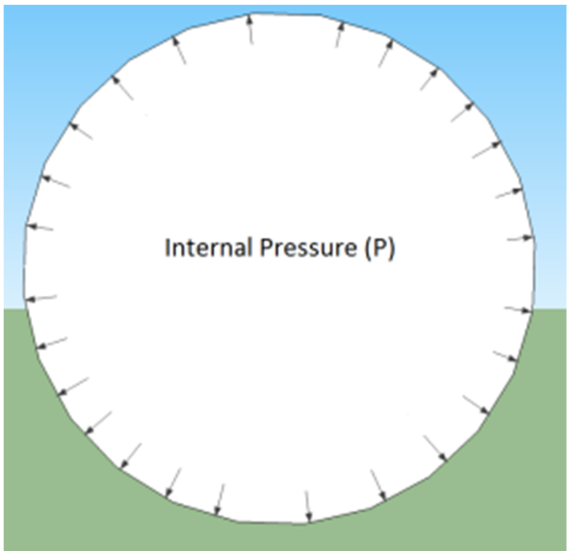

Figure 21. Cross Sectional View of Pouch with Internal Pressure.

Adapted from Wachala's Figure 5. (Wachala, 1991)

Using the diagrams of an ideal pouch that forms a cylinder in Figure 20 and Figure 21, the equation for the force acted upon the sealing interface is Equation (9).

— (9)

Where:

P = Internal Pressure

d = Diameter of Circular Cross-Section (in)

L = Length of Pouch (in)

W = Width (in)

However, Wachala notes that pouches do not form a perfectly circular cross-section at burst, and the tangent to the sealing surface is not 90° as would be assumed using the assumption that a pouch forms a cylinder. This concept is visually explained in Figure 22.

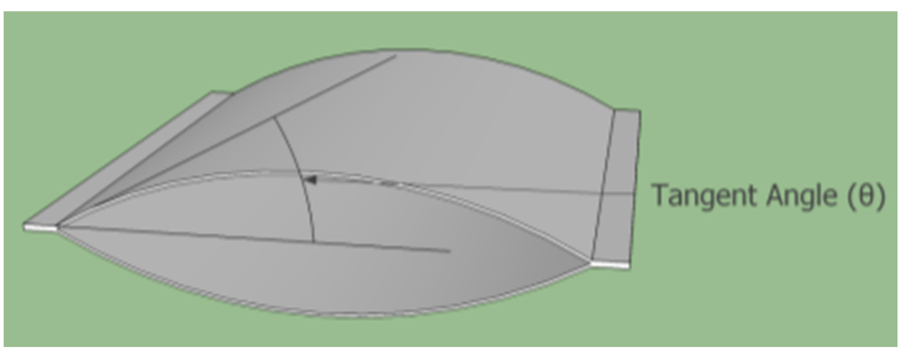

Figure 22. Angle of Tangent to Sealing Surface.
Adapted from Wachala's Figure 6 (Wachala, 1991).

At this point in Wachala's study, he states that the "direct correlation between burst and peel is beginning to deteriorate." However, he states that an equation for the ideal situation in which a pouch forms a cylinder can be used to correlate burst pressure to peel strength, but the error may reach 20 – 30% (Wachala, 1991). To account for this correlation problem, Wachala suggests performing what he refers to as a "hybrid correlation" (Wachala, 1991).

A hybrid correlation is a method in which Equation (5) is used to determine the predicted burst pressure for pouches of various sizes. Then, pouches are burst tested and the actual burst pressure is plotted on the same graph as the predicted burst pressures. According to Wachala, when the predicted burst pressure curves are studied, a quadratic equation (Equation (10)) is found to have the best fit. The three coefficients, a, b, and c, from the quadratic equation along with the length and width values for the pouch in question are used in Equation (11) to determine a correction factor. After the correction factor is determined, Equation (12) is used to obtain a more accurate burst pressure prediction.

$$ax^2 + bx + c = 0 \qquad (10)$$

$$Correction\ Factor\ (K) = \frac{L^2}{aL^2 + bLW + cW^2} \tag{11}$$

Where:

L = Length (in)

W = Width (in)

$$Predicted\ Burst\ Pressure = \frac{Perimeter}{Area}\ x\ K \tag{12}$$

In 1993, a study entitled "Relationship between Seal Strength and Burst Pressure for Pouches" by Kit L. Yam, Jack Rossen, and Xuan-Fei Wu was published. In this study, Yam presented a force diagram (Figure 23) for pouches at burst in a restrained burst testing apparatus. He analyzed the diagram to determine the forces occurring in the y-axis to obtain Equation (13) below. Then, this equation was integrated from 0 to π/2 or 0 to 90 degrees using the integral in Equation (13). Next, Yam explains that the seal peel strength (S) can be used in place of F_y, and burst pressure (P_b) can be used in place of internal pressure (P) to obtain Equation (15). Like Wachala, Yam also assumes a cylindrical geometry at burst (Yam, Rossen, & Wu, 1993).

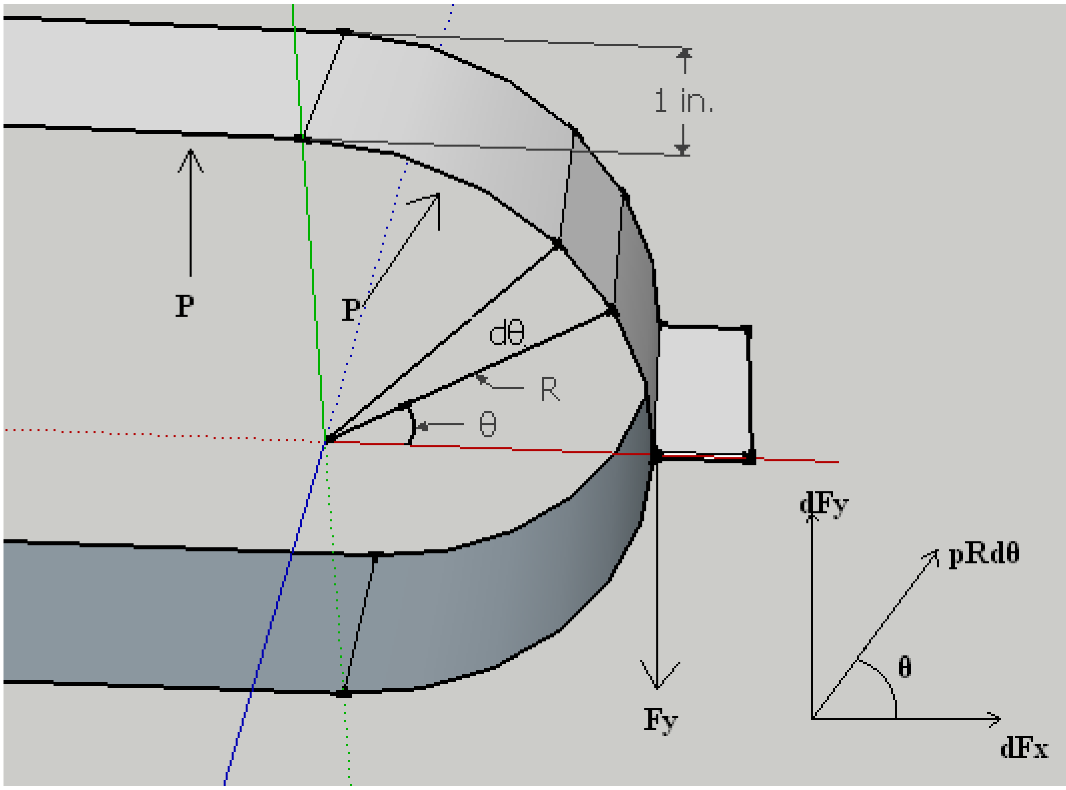

Figure 23. Forces Acting Upon Pouch In Restrained Burst Test.

Drawing Adapted from Yam's Figure 2 (Yam et al., 1993).

Forces Occurring in Y-axis

$$dF_y = PRsin\theta d\theta \tag{13}$$

Integral of Forces Occurring in Y-axis

$$F_y = \int_0^{\pi/2} PRsin\theta d\theta \ = PR \tag{14}$$

Predicted Seal Peel Strength Based on Burst Pressure

$$S = P_b R \qquad (15)$$

Yam's study involved the use of meal ready-to-eat (MRE) pouches to test the validity of Equation (15). MRE's are shelf stable meals used in the military. In order to test the equation, Yam heat sealed the open end of each pouch at 250°C and 60 PSI for 1 second. Then, burst tests were performed at a several different restraining plate gaps and inflation rates. The inflation rates were adjusted so that specific burst peeling times could be obtained. Eight pouches were burst tested at six different burst peeling times for a total of 48 burst tests. In addition, peel testing was performed using 8 samples per burst peeling time totaling 48 peel tests. The speed at which the jaws of the tensile tester move apart, or the crosshead speed, was calculated using Equation (16). The data collected from the peel tests were then used in Equation (15) to predict the burst pressures. The observed burst pressures were then plotted against the predicted burst pressures (Yam et al., 1993).

Tensile Peeling Time

$$t_p = 60\Delta L/v \qquad (16)$$

Where:

ΔL = Elongation at Seal Strength (in.)

v = Crosshead Speed (in./min.)

t_p= Burst Peeling Time (s)

Yam stated that seal peel strength "increases linearly with the logarithm of the crosshead speed." Accordingly, the seal peel strength decreased as tensile peeling time increased due to the inverse relationship between tensile peeling time and crosshead speed. Yam's data also indicated that, as tensile peeling time decreases, its influence on seal peel strength will increase. Likewise, the burst pressure has an inverse relationship with the burst peeling time. According to Yam, shorter peeling times have a greater effect on burst pressure and seal peel strength than longer peeling times. Yam concluded that the predicted burst pressures were in good agreement with the observed burst pressures (Yam et al., 1993).

Another article entitled "Comparing Tensile and Inflation Seal-Strength Tests for Medical Pouches" studied the relationship of seal peel strength and burst pressure as well (Franks & Barcan, 1999). First, Franks and Barcan developed testing to study the effects of the following variables on the burst pressure of pouches:

1. Restraining Plate Gaps – Unrestrained, ¼ in, ½ in, ¾ in, and 1 in.
2. Length-to-width Ratio of Pouch
3. Flow Rate or Burst Peeling Time
4. Open vs. Closed Pouch
5. Nonporous vs. Porous Pouch Materials
6. Pouch Size – Small, Medium, Large
7. Package Style – Pouch, Tray, Strip Bag
8. Adhesive Material – Peelable, Heat Weld (Franks & Barcan, 1999).

However, initial testing was limited to studying peelable, nonporous pouches with varying length-to-width ratios with the same surface area. These initial tests were done to study the effect of various restrained plate gaps (as well as unrestrained) on inflation burst pressure. Initial testing measured the effect of length-to-width ratios on burst pressure and the location of seal failure (Franks & Barcan, 1999).

Further testing was designed to determine the sensitivity of the burst testing apparatus. This was performed by first sealing the pouches at varying temperature, pressure, or dwell times to result in different seal strengths. Then, some sealed pouches were peel tested, and the others were burst tested. The burst testing was performed using an unrestrained apparatus and a restrained apparatus with plate gapes of ¼, ½, ¾, and 1 inch (Franks & Barcan, 1999).

Franks and Barcan found that the restraining plate gap distance had the greatest influence on the burst pressure of the pouches he studied. They determined that there were significant differences between each plate gap distance but found no significant difference between open-ended and closed pouch configurations. They also found no significant difference between the variability of unrestrained burst tests and the variability of restrained burst tests. Their data also showed a linear regression of burst pressure versus seal peel strength with an R^2 value of 0.9422. The seal temperature was varied to obtain different seal strengths.

In conclusion, Franks and Barcan found that "inflation burst tests using restraining plates offer the advantage of identifying the lowest-seal-strength area of packages." They explain this result by concluding that burst tests allow for "a more uniform distribution of stresses on the package seals than other tests do." According to Franks and Barcan, "manufacturers can confidently develop the relationship between these tests and apply the tests for process control" (Franks & Barcan, 1999).

A more recent study entitled "Correlation of Peel and Burst Tests for Pouches" written by Rosamari Feliu-Baéz, Hugh E. Lockhart, and Gary Burgess was in 2001 (Feliú-Baéz, 2001). In this study, the authors attempted to verify the same relationship between seal peel strength and burst pressure as Yam. The researchers drew their own force diagram and analyzed the forces to develop Equation (17). It can be noted that Equation (17) is the same as Equation (15) in terms of diameter instead of radius.

Predicted Burst Pressure Based on Seal Peel Strength

$$P = 2S/D \qquad (17)$$

Where:

P = Predicted Burst Pressure

S = Seal Peel Strength

D = Restraining Plate Gap Distance

The researchers first made some predictions about their experiment based on the equation they were studying. They predicted that the burst pressure would increase as the seal peel strength increases. They also predicted that the burst pressure would increase with decreasing plate gap dimensions. Finally, they predicted that the burst pressure does not depend on the area of the pouch in contact with the restraining plates.

To test the validity of the equation, the researchers used 6 inch x 10 inch Tyvek and "plastic" pouches. The study did not note the composition of the "plastic" layer. First, burst tests were performed. The number of pouches burst tested was not specifically stated, but the data tables indicate that 9 pouches were burst tested. The burst testing was performed at restraining plate gaps of 0.25, 0.5, and 1 inch. Additionally, the inflation rate was adjusted to machine settings of 1, 5, and 9 for each restraining plate gap tested. The burst peeling time was measured with the use of a stopwatch.

Peel testing was performed on 1-inch wide samples of each of the seals denoted below in Figure 24 by A, B, D, and E. One sample for each seal was cut from each of 8 pouches totaling 32 samples. Like Yam, Baéz adjusted the crosshead speed for each sample tested according to Equation (18) to achieve the same peeling time as the burst peeling time. The gauge length was also adjusted, according to Equation (19), so that gauge lengths of 0.40, 0.80, and 1.60 inches were used. For each peel test, the peak seal strength and the tensile peeling time were recorded. The data obtained was used to determine predicted burst pressure values according to Equation (17). The predicted burst pressure was calculated using two different values of seal peel strength: S_{min} and S_{total}. S_{min} was the average of the minimum peak seal peel strength found for each pouch. For instance, if seal A had the lowest peak peel strength on every pouch tested, S_{min} would be the average of all the peak peel strength values obtained from seal A on the pouches. S_{total} was the average of all peak peel strength values obtained from each pouch.

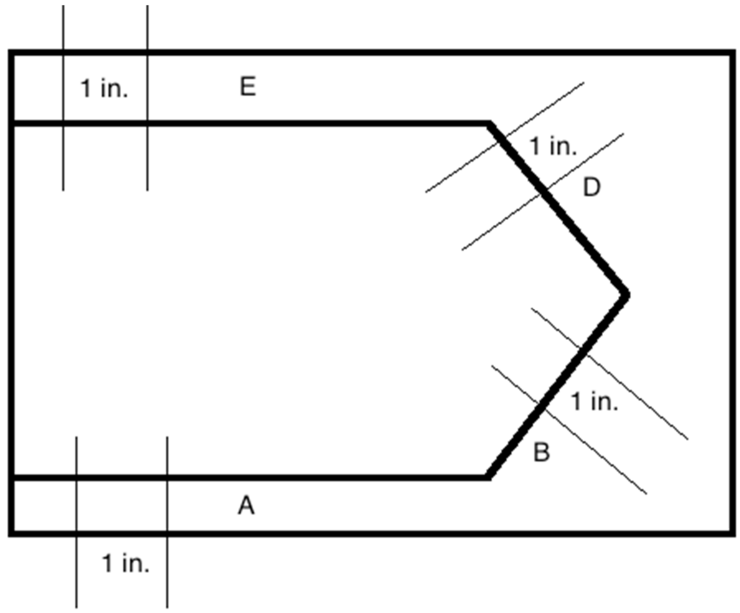

Figure 24 - Seals on Chevron Pouch.

Adapted from Baéz's Figure 5 (Feliú-Baéz, Lockhart, & Burgess, 2001).

Crosshead Speed

(18)

Where:

v = Crosshead Speed (in/min)

= Twice the Seal Width (in)

Burst Peeling Time (s)

Gauge Length

$$Gauge\ Length = \frac{\pi D}{2} \qquad (19)$$

Where:

D = Restraining Plate Gap Dimension(in)

The results of this study showed that Equation (17) grossly overestimated the burst pressure of a pouch. The research indicated that the overestimation decreased as the restraining plate gap increased. From this, the researchers conclude that the "the theoretical correlation between peel and burst test did not work for the Tyvek/plastic chevron seal pouches that were tested in this experiment" (Feliú-Baéz et al., 2001). They also noted that this conclusion differs from the results of Yam's study, which indicated a good correlation between the two.

They explained possible reasons why the formula failed including the seal geometry and size of the pouches not being the same as the pouches used in Yam's study. According to Baéz the "seal perimeter does not take the same load at all points because it deforms differently around the seal perimeter." They indicate that wrinkled areas do not take any stress, and sharp corners be points at which stress is concentrated (Feliú-Baéz et al., 2001). Also the samples tested for the peel testing may not have been representative of the true peel strength because a weak area may have been missed during the sample cutting process. Additionally, they note that materials will elongate or break at a lower value when using lower inflation rates or lower crosshead speeds. Finally, they conclude that Equation (17) "is not universally applicable" (Feliú-Baéz et al., 2001).

In another study published by Baéz, entitled "Correlating Peel and Burst Tests for Sterile Medical Device Packages," she reported her findings and explored another theoretical relationship. In her new theoretical model, she attempted to incorporate package size into the equation. Her previous studies showed that package size had a significant effect on the restrained burst pressure of a pouch (Feliú-Baéz, 1998). Therefore, she hypothesized that the accuracy of Yam's theoretical model (Equation (15)) could be improved by incorporating the pouch size into the equation.

One thing to consider, as Baéz pointed out, is that the dimensions of a pouch change as the pouch is inflated. For example, there is a length, width, and depth measurement associated with each pouch. The depth measurement is effectively zero before inflation. Once inflation occurs, the depth increases. However, to account for the increase in depth, the length and width must decrease.

In her study, Baéz first wanted to determine whether the crosshead speed and gauge length had any effect on the peel strength of her materials. It was determined that there was no significant effect, so she did not control the burst peeling times and the tensile peeling times to be the same (Feliú-Baéz, 1998).

Baéz developed a theoretical model that incorporated the change in package dimensions due to inflation (Equation(20)). She noted that the new theoretical model would not be any better than Kit Yam's original equation because it has a correction factor that is greater than 1 (Equation (21)). She stated that the correction factor being greater than 1 would cause greater overestimation of the burst pressures than they would with Yam's equation.

$$P = 2\,S/D\,[(L_o + W_o - 1.142D)(L_o + W_o - 2.142D)] \quad (20)$$

$$Correction\ Factor = [(L_o + W_o - 1.142D)(L_o + W_o - 2.142D)] \quad (21)$$

Where:

$L_o = Uninflated\ Length\ of\ Pouch\ (in)$

$W_o = Uninflated\ Width\ of\ Pouch\ (in)$

$D = Restraining\ Plate\ Gap\ (in)$

$S = Seal\ Peel\ Strength\ \left(\frac{lb}{in}\right)$

$P = Burst\ Pressure\ (PSI)$

Since Equation (20) did not improve the accuracy of the theoretical model, she developed several empirical models and performed multiple regression analysis (Feliú-Baéz, Lockhart, & Burgess, 2003). The empirical models were used to calculate a predicted burst pressure which was compared to the burst pressure obtained during testing. The results showed that there was a 1 to 7% error between the two values depending on the empirical equation used. She then outlined a procedure for determining regression coefficients for pouches made of materials other than the ones used in her study.

Parameter Estimation

Yam suggests that there is a linear relationship between seal peel strength and burst pressure. The linear relationship is expressed by Equation (22).

$$S = \frac{D}{2}P \tag{22}$$

S = Seal Strength (lbf/in)

D = Restrained Burst Test Plate Gap (in)

P = Burst Pressure (PSI)

The validity of this model can be evaluated by using experimental data and regression analysis to estimate the parameters for the linear relationship.

The regression model for the regression analysis is expressed as Equation (23) below. The y-intercept(β_0) and slope (β_1) are unknown parameters that must be estimated. Assumptions associated with the model are that X_i is a known value and that ε_i has a mean of 0 with a variance of σ^2. It is also assumed that is εi uncorrelated with x_i (Buonaccorsi, 2010). If these assumptions are met, the least squares estimators for the y-intercept and slope are consistent estimators for the unknown parameters β_0 and β_1. Consistent estimators are estimators that converge to the true value of the parameter as the sample size (n) increases(Buonaccorsi, 2010).

$$Y_i = \beta_0 + \beta_1 x_i + \varepsilon_i \quad (23)$$

For all i=1,….,n

Where:

Y_i = Dependent Variable

X_i = Independent Variable

ε_i = Equation Error

β_0 = y-intercept

β_1 = slope

In an ideal situation, it would be possible to obtain both a peel strength measurement and a burst pressure measurement from the exact same pouch. However, the destructive nature of both the peel test and the burst test prevents one from measuring both values on the same pouch. To address the inability to obtain both burst and peel measurements from the same pouch, a "proxy variable" must be used. A proxy variable is a variable that is assumed to have a linear relationship with the immeasurable variable. In this case, the immeasurable burst pressures for the pouches that were tested by the peel test are replaced with the burst pressures for another group of pouches that were manufactured under the same pouch sealing conditions.

However, since the proxy burst pressures are used, the usual least squares estimate of the slope underestimates the true relationship between seal peel strength and burst pressure. This is because the proxy variable is correlated with the equation error

(Ashenfelter, Levine, & Zimmerman, 2002). One method to compensate for the inconsistency caused by using the proxy variable is to incorporate the use of an "instrumental variable." An instrumental variable is a variable that is correlated with the independent variable but is also uncorrelated with the equation error (Ashenfelter et al., 2002). Since an instrumental variable is not correlated with the equation error, it can be used to develop consistent estimators for both β_0 and β_1. In this study, dwell time is used as the instrumental variable because it is varied to obtain different seal peel strengths and burst pressures. The same use of the same dwell times for peel testing and burst testing allows it to be used as an instrumental variable in the study of the relationship between burst pressure and seal peel strength.

To incorporate the instrumental variable properly, a process known as "two-stage least squares" must be performed. During the first stage of two-stage least squares, the independent variable is regressed onto the instrumental variable (dwell time) using ordinary least squares. The second stage of two-stage least squares requires that the predicted values of the independent variable (burst pressure) be regressed onto the dependent variable (seal peel strength). The estimates resulting from the second stage are the instrumental variable estimates (Ashenfelter et al., 2002).

Scope of This Research Study

It is evident that there is disagreement among the packaging community about the relationship between peel strength and burst pressure for pouches. One problem is that there has not been much research done in this area. Baéz only peel tested 32 specimens and burst tested 9 specimens in the study from 2001, which may not be enough for statistical significance, yet she concluded that the equation is not applicable to all situations. Franks and Barcan also caution that the correlation they found may "not necessarily represent a universal algorithm"(Franks & Barcan, 1999). However, they took a different approach in that their correlation is not an analytical correlation based on force diagrams like Yam and Wachala. Wachala's analysis differs from Yam's in that he determined a correction factor. Wachala's study also relies on seal strength being completely uniform at every point, which is something that probably does not happen in practice. With a carefully designed experiment, a significant sample size, and the use of the correct statistical methods it may be possible to determine the relationship between burst pressure and seal peel strength.

In this research, the experiment was designed to vary dwell time to vary the seal strength. The various dwell times were used to seal pouches, and these pouches were burst tested at using inflation burst testing with several restraining plate gaps and compression burst testing. Corresponding peel tests were also performed using the burst peeling time and restraining plate gaps to determine the crosshead speed and gauge length required. The data was then compiled and statistically analyzed using the two-stage least squares method presented in the "Parameter Estimation" section to estimate the slope. The estimated slopes were then compared to the theoretical slopes to determine whether the theoretical relationship suggested by Yam is valid for the pouches used in this study.

CHAPTER THREE

MATERIALS AND METHODS

The materials and methods section covers the material, equipment and procedures used in this study.

Materials and Equipment

The materials used in this study are detailed below in Table 1 below. Equipment is detailed in Table 2.

Table 1. Materials.

	Product Name	**Supplier**
Substrates	7000 series uncoated 92 ga. PET Film	Mitsubishi Polyester Film
	3 mil LLDPE Supplied	Dow Chemical
Adhesive	Adcote 577A/B	Dow Chemical
Septa	1mm Grey Septa	MOCON
Bottle Cap	Small Water Bottle Cap w/ Hole Drilled in Center (1/4in hole)	Deer Park Bottled Water

Table 2. Equipment.

Equipment	Description
Heat Sealer	Sentinel 1-Side Constant Heat Sealer Model 12AS
Laminator	Custom Solvent-Based Coater/Laminator
Pouch Maker	Shanghai Gaoqin Sunshine Pouch Maker (Similar to Totani Pouch Maker)
Closed Pouch Inflation Burst Tester	Mocon Lippke Model 4500
Open Pouch Inflation Burst Tester	Origin Unknown
Vacuum Burst Tester	Visual Check Vacuum Chamber
Compression Tester	Lansmont Squeezer
Stopwatch	Armitron C311 Digital Wrist Watch Accurate to 0.01s
Graduated Cylinder	1000 mL Nalgene Graduated Cylinder
Digital Thermometer	Digi-Sense Digital Thermocouple Thermometer Accurate to 0.1 ˚F

Selection of Materials

Both Polyethylene Terephthalate (PET) and linear low density polyethylene (LLDPE) are common materials in pouches, and are especially common in drink pouches. LLDPE is a commonly used sealant material, whereas PET is used as for its oxygen barrier and strength.

To prevent stretching of the material during both burst testing and peel testing, a strong substrate was needed. This led to the selection of 92 gauge PET, as recommended by Mitsubishi Polyester Film. It was suggested that 92 gauge PET would allow for more consistent results with less stretching.

It was also necessary to select a sealant material that would allow for a wide range of sealing conditions that would result in peelable seals. LLDPE was already available in thicknesses of 2 mil and 3 mil. Preliminary testing of both materials showed that 3 mil LLDPE had a wider range of sealing conditions that resulted in peelable seals.

Adcote 577 A/B adhesive was selected based on the recommendation of Dow Chemical. The recommendation was made based on the compatibility with the two substrate materials and the laminating equipment available.

Lamination

First, the two substrates were placed on the laminator in preparation for running the lamination. The adhesive was mixed according to the adhesive data sheets. The final adhesive mixture contained 3560g of Adcote 577A and 227g of Adcote 577B along with 3800g of Ethyl Acetate.

The lamination was performed at a line speed of 55 feet per minute with the dryer set at 180˚F and the laminating nip set at 190˚F. Two rolls of 92 ga. PET laminated to 3 mil LLDPE were produced.

Bond Testing

Bond testing was performed on the lamination after allowing it to cure for 1 week. The bond testing was performed on the Satec Instron T10000 according to ASTM F904 with a crosshead speed of 10 in/min and a gauge length of 2 in.

Pouch Making

The two laminated webs were then placed on the pouch maker. The seal bars were adjusted to obtain pouches with dimensions detailed in Figure 25 below. The pouches were oriented so that seals B and D run perpendicular to the machine direction. Seal A was not made by the pouch maker. The pouch maker was run at 25 cycles per minute with all seal bars set at 130 ˚C. Two pouches were produced per cycle equaling 50 pouches per batch. 31 batches were produced in the same run to equal a total of 1550 pouches. The seals produced on the pouch maker were tested according to ASTM F88 on the Satec Instron T10000 to determine the seal strength.

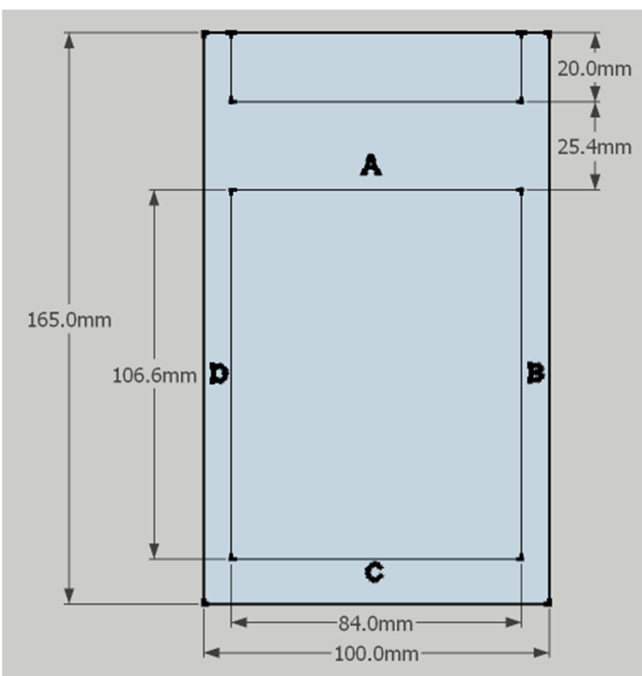

Figure 25. Pouch Dimensions

Pouch Sealing

Sealing of pouch seal A (Figure 25) occurred on a Sentinel 1-side constant heat sealer. In order to maintain consistent sealing temperatures, it was necessary to use the stopwatch to measure the time elapsed between each sealing repetition. During sealing, the top jaw loses heat and the bottom jaw gains heat. When the sealing repetition is complete, the thermostat starts the heating cycle to bring the top jaw back up to temperature. The bottom jaw loses heat as soon as the top jaw releases pressure and moves to the starting position. The time elapsed between each sealing repetition was held constant at 1 minute and 15 seconds. This allowed for a consistent amount of time for the bottom seal bar to cool to approximately 110 +/-1°F. Keeping the time consistent between each sealing repetition also allowed for the top (heated) seal bar to returne to the temperature of 270 +/- 1°F.

The temperature was also monitored using a DigiSense Digital Thermocouple Thermometer. One thermocouple was placed in the center set screw hole on the top seal bar. The other thermocouple was placed on the left side of the rubber seal bar so that it was between the glass fabric that comes in contact with the top seal bar and the rubber, bottom seal bar (Figure 26).

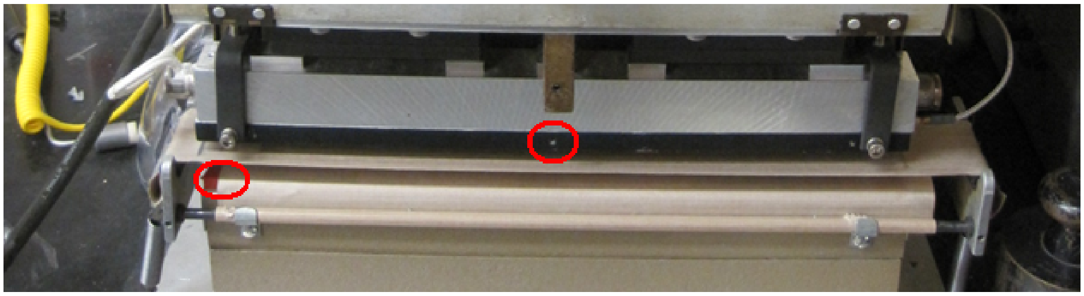

Figure 26. Thermocouple Placement.

The Sentinel sealer was also allowed to warm up to consistent temperatures by performing at least 10 sealing repetitions before any pouches were sealed. To seal a pouch, the open end of the pouch was placed between the seal bars. It was positioned so that the seal A would be perpendicular to seals D and B. The pouch was placed in the middle of the sealer, and measures were taken to ensure that there were no wrinkles in the material. After a pouch was sealed, it was placed in a box to allow for a 24 hour conditioning time.

Prior to the sealing of any pouches, a 0.25in diameter hole was drilled in the center of small polypropylene bottle caps (Figure 27). A flat pouch does not provide enough depth to properly puncture the pouch with the inflation needle. The bottle caps were provided depth and were used to assist in the puncturing of pouches when performing inflation burst tests. One bottle cap was placed inside each pouch assigned to burst testing.

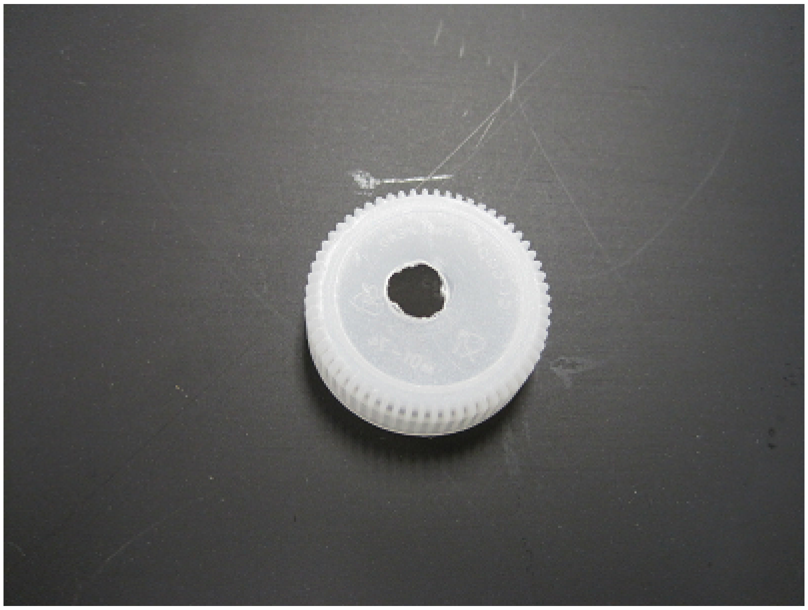

Figure 27. Small Bottle Cap with 0.25in Diameter Hole

For pouches assigned to compression testing, 50 mL of water was measured using a graduated cylinder. The 50 mL of water were poured into the pouch and the sealing area was dried of any water prior to sealing. Effort was made to ensure that a minimal amount of air was left in the pouch when sealed. Minimizing air content was accomplished by flattening the sealing area by hand and forcing the water as close to the seal as possible.

Sealing Matrix

After making pouches, the first step necessary was to determine at least 5 sealing conditions that resulted in peelable seals. The 5 conditions selected needed to be significantly different from one another. A sealing matrix (Table 23. Sealing Matrix) was developed to assist in the determination of appropriate sealing conditions. The sealing matrix was first populated with temperature, pressure, and dwell time. The dwell times were varied by 0.25s.The temperature and pressure were held constant. Seals were produced according to the sealing matrix and conditioned at ambient lab temperature and humidity for 24 hours prior to testing. Peel testing was performed on the seals. The remainder of the sealing matrix was populated with the data obtained from the peel tests.

Preliminary Peel Testing

Peel testing was performed on the seals created according to the sealing matrix after they were allowed to be conditioned for 24 hours. The peel testing was performed on the Satec Instron T10000 tensile tester using Instron Bluehill software. The gauge length was set to 1 inch. The crosshead speed was set to 10 in/min. A one-inch wide sample was cut perpendicular to the sealing area of each sealed pouch. The specimens were testing using the unsupported method according to ASTM F88. The unsupported method was chosen to more closely simulate the peeling that occurs during a burst test.

When performing the peel testing it was noted that there was no distinct peak distinguishing the initiation point of the peel (Figure 28). Therefore, the Instron Bluehill software was set up to calculate average burst across 2.5 mm to 20 mm. The software was also set up to determine the maximum peel strength between 2.5 mm to 20 mm.

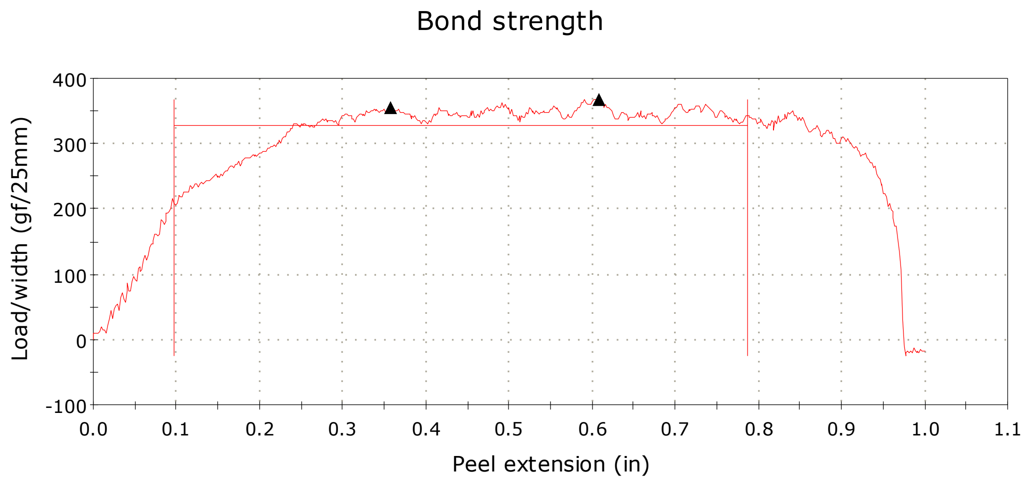

Figure 28. Example Peel Test.

The mode of failure was an important aspect since the goal was to establish at least 5 different conditions that resulted in peelable seals. Accordingly, the seals were designated either "peelable" or "fusion." If a seal began tearing before the peel test was completed, it was designated as a fusion seal. If a seal peeled throughout the entire test, it was designated as a peelable seal. The data collected from the preliminary peel testing was the average load per width, maximum load per width, and the mechanism (peel vs. fusion). The data were used to populate the rest of the sealing matrix.

Statistical Analysis of the Preliminary Peel Testing Data

The data obtained from the preliminary peel testing were analyzed by first determining which dwell times resulted in peelable seels. For the dwell times that resulted in peelable seals with seal strength of at least 200gf/in, analysis of variance was used to determine if the means for the log of load were different for any of the dwell times. Dwell times selected for further study were based on the results from the analysis of variance.

The preliminary peel data were also used to determine the number of replicates to use for each dwell time for the regression analysis. The number of replicates for each dwell time was chosen so that the margin of error for a 95% confidence interval of the mean for the log of load was approximately 10%.

Preliminary Burst Testing

The dwell times selected from the preliminary peel testing analysis were used to seal pouches for burst testing. The same sealing process detailed in "pouch sealing" was used.

A 1mm grey septum was placed on the outside of the center of the sealed area of the pouch. The bottle cap was positioned so that the hole lined up with the hole on the septa (Figure 29). The inflation needle was used to puncture the pouch at the location of the septa. The pouch was positioned with seal A on the left side of the testing apparatus (Figure 30).

Figure 29. Alignment of Septa with Bottle Cap

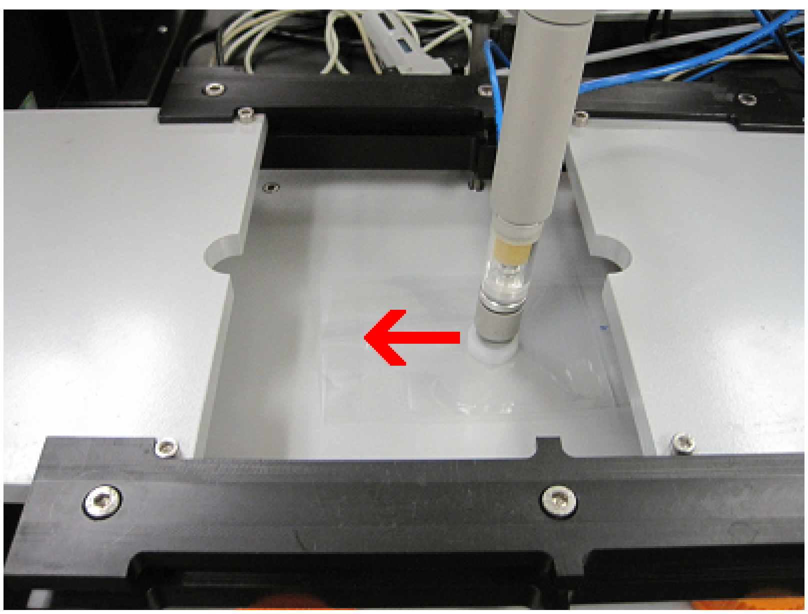

Figure 30. Pouch Orientation for Burst Testing

For each dwell time selected 10 samples were tested at 1 inch plate gap on the Lippke 4500 burst tester. The inflation rate was set to 0.300 PSI/s. The pretest inflation pressure was set to 0.500 PSI. The maximum pressure limit was set to 30 PSI to ensure that the pouches were burst before the test was completed. The data collected was the burst pressure and burst peeling time.

The burst pressure was measured by the Mocon Lippke 4500 software. The burst peeling time was recorded using the stopwatch. The time was started when seal A began to peel. The time was stopped when seal A burst.

Statistical Analysis of Preliminary Burst Testing

The preliminary burst test data were also used to determine the number of replicates to use for each dwell time for the regression analysis. The number of replicates for each dwell time was chosen so that the margin of error for a 95% confidence interval of the mean for the log of pressure was approximately 10%.

Randomization Procedure

The five dwell times selected after the statistical analysis of the preliminary peel testing were assigned numbers 1 through 5. A random permutation was used to determine the testing order for the pouches sealed at the different dwell times.

Based upon the results of the preliminary peel testing and preliminary burst testing, it was decided that 15 samples per iteration of dwell time and restraining plate gap combination would be sufficient for both the burst test and the corresponding peel test. Therefore, 30 pouches were selected and numbered 1 through 30. A random permutation of these numbers was used to assign the pouches to the different tests. The first 15 pouch numbers of the generated permutation were assigned to burst testing, and the last 15 pouch numbers of the permutation were assigned to corresponding peel testing. A different random permutation was used for each dwell time and restraining plate combination.

Inflation Burst Testing

The burst testing was carried out according to the testing schedule (Table 25). The data collected were the burst pressures, burst peeling times, and locations of the burst. The burst location was denoted as either “center” or “corner.” If the pouch burst at a corner of Seal A and either Seal B or Seal D, it was considered a corner burst. If the pouch burst away from the corner, it was considered a center burst. The burst peeling time was measured using the stopwatch. The time was started at the point at which the seal began to peel. The time was stopped at the point of burst. The burst pressure was recorded by the Mocon Lippke 4500 software.

Compression Burst Testing

For compression testing, pouches were sealed according to the “Pouch Sealing” section. Compression testing was performed on the first 15 pouches in the permutations generated. Compression testing was performed on the Lansmont Squeezer with Lansmont Squeezer Reader software. In order to make the peeling of the seal more visible during compression testing, a solid steel platform was used as the bottom platen. The use of the solid steal platform kept the pouch raised off the true bottom platen of the machine to allow for easier visual observation of the pouch burst.

The Lansmont Squeezer is a fixed platen compression tester. It was set to compress at a constant rate of 0.5 in/min. The preload was set to zero. In addition, the machine was set to stop at 5000 lbf. However, the test was manually stopped when failure of the pouch occurred. Compression force was recorded in pounds force. The final distance between the top platen and the steal platform was calculated by subtracting the height of the platform from the position of the top platen at burst. This value will be referred to as the compression plate gap and was recorded in inches. Data recorded were the plate gap and the force at burst.

Determination of Crosshead Speed and Gauge Length Used in Peel Testing

The next step was to analyze the burst pressure data to determine the mean burst peeling time. The mean burst peeling time for each plate gap and dwell time combination was used to determine what the crosshead speed needed to be for the peel test. The crosshead speed was calculated according to Equation (18).

The gauge length was calculated based on Equation (19). For each plate gap used for burst testing, there was a corresponding gauge length used for peel testing.

Peel Testing

After the necessary crosshead speeds and gauge lengths were calculated, the remaining pouches from each permutation were prepared for peel testing. To prepare the pouches for peel testing, a 1 inch wide sample was cut perpendicular to seal A using the 1 inch sample cutter.

Prior to peel testing a group of pouches, the gauge length was adjusted to a length corresponding to the restraining plate gap from burst testing. As described in "Determination of Crosshead Speed and Gauge Length for Peel Testing," the crosshead speed was calculated using Equation (18), and the gauge length was calculated using Equation (19).

The data collected from the peel test was the maximum load per width, the average load per width, and the failure mode. Similar to preliminary peel testing, the maximum load/width and the average load/width was measured across 2.5 mm to 20 mm peel extension. The failure mode of the sample was designated as either peelable or fusion. A seal was considered peelable if the test was completed without any tearing of the sample. A seal was considered fusion if a tear occurred before the test was completed.

Preparation of Data

The compression burst force values needed to be converted to the same units as inflation burst values. To accomplish this, the burst pressure was calculated by dividing the compression force by the area of the pouch in contact with the platen and the compression force. The area of the pouch in contact with the top platen was approximated to the inside area of the pouch according to Equation (24). The compression burst pressure was calculated according to Equation (25). The dimensions used can be seen in Figure 25.

$$Contact\ Area\ of\ Pouch = A = L\ x\ W = 13.879\ in^2 \tag{24}$$

Where:

$$L = \frac{106.6mm}{25.4\frac{mm}{in}} = 4.197\ in$$

$$W = \frac{84.0mm}{25.4\frac{mm}{in}} = 3.307\ in$$

$$Compression\ Burst\ Pressure = \frac{F_c}{A} \tag{25}$$

Where:

$A = Contact\ Area\ of\ Pouch\ (in^2)$

$F_c = Compressive\ Force\ at\ Burst\ (lbf)$

The average load per width from the peel tests was used to correlate burst pressure to peel strength. The values were transformed into the logarithm using SAS and are denoted as “logload.” The mean of the logarithm of the average load per width values was calculated for each dwell time and plate gap combination. The mean burst pressure was also calculated for each dwell time and plate gap combination.

Statistical Analysis of Data

For the regression analysis, the peel strength was the dependent variable and the load was the independent variable. The reason the peel strength was considered the dependent variable is because the crosshead speed and gauge length used for each peel test were dependent upon the results of the corresponding burst tests. The Two-Stage Least Squares method described in the "Parameter Estimation" section of the literature review was used to estimate the slope of the regression for each plate gap tested. According to Equation (17), the slope of the regression for each plate gap tested should have been the plate gap divided by two. A two-tailed t-test with a level of significance of 0.05 was used to determine if the slopes were different from the theoretical slopes in Table 3.

Table 3. Perfect Slope Estimates.

Plate Gap (in)	Theoretical Slope
0.17	0.085
0.25	0.125
0.5	0.25
1	0.5
Unrestrained	0.75

CHAPTER FOUR

RESULTS AND DISCUSSION

This section presents the data collected from each part of testing. This section will also discuss the statistical analysis of the data.

Bond Testing Results

Bond testing was performed to ensure that an adequate bond between the LLDPE and the PET occurred. The bond tests resulted in an average bond strength of 2140 gf/25mm. Due to the high bond strength, it was determined that the adhesive bond had set and the material could be used to make pouches.

Pouch Making Results

Peel testing was performed on the pouches according to ASTM F88 within an hour after they were made on the pouch maker to ensure that all seals created were very strong fusion seals. As can be seen in Table 4 below, all seals tested exhibited a material break failure mode and had seal strengths greater than 8000 gf/25mm. This was desired to ensure that only seal A would fail during burst testing. Batch number 1 denoted in Table 4 is the beginning of the run after the machine warmed up, and batch number 31 is the end of the run.

Table 4. Pouch Side and Bottom Seal Testing.

Batch	Seal Location	Failure Mode	Maximum Load/Width (gf/25mm)
1	C	Material Break	10982
1	C	Material Break	10112
1	B	Material Break	9757
1	B	Material Break	10069
1	D	Material Break	9538
1	D	Material Break	9529
31	C	Material Break	8969
31	C	Material Break	10438
31	B	Material Break	9186
31	B	Material Break	9709
31	C	Material Break	9351
31	C	Material Break	9720
Mean			9780
Std. Dev.			556

Preliminary Peel Testing Results

Table 5. Preliminary Peel Testing - Mean Seal Strengths and Sample Numbers.

Dwell Time (s)	Failure Mode	Mean of Seal Strength (gf/25mm)	Std. Dev.	Sample Number Required
2	Peelable	82	10	6
2.25	Peelable	129	23	13
2.5	Peelable	206	31	9
2.75	Peelable	295	53	13
3	Peelable	431	52	6
3.25	Peelable	568	53	4
3.5	Peelable	689	79	6
3.75	Peelable	821	94	5
4	Peelable	1011	78	3
4.25	Fusion	2115	871	66
4.5	Fusion	2493	1141	81
4.75	Fusion	3940	877	20
5	Fusion	5852	574	4

Table 5 displays a summary of the data obtained from preliminary peel testing. The failure mode was designated either peelable or fusion for each individual sample from a particular dwell time. If more than one sample from a grouping resulted in a fusion failure, the dwell time was considered to produce fusion seals. If all samples obtained from the dwell time tested resulted in peelable seals, the dwell time was considered to produce peelable seals. Since the focus of this study was peelable seals, any dwell time that was considered to produce fusion seals was eliminated from further testing.

The mean seal strength was calculated by taking the average of the load per width values for each sample. If a particular sample resulted in a fusion seal, the maximum load per width value was used. If a particular dwell time was considered to produce peelable seals, any fusion seals were left out of the mean seal strength calculation, and the average load/width was used from the peelable samples. Also, an industry standard of minimum seal strength of 200 gf/25mm was used to eliminate any dwell times that produced seals that were too weak. The rows of the table that are shaded red indicate the dwell times that were eliminated from further testing

The sample number required was calculated to determine the sample size required to ensure that the margin of error for a 95% confidence interval would be within 10% of the mean seal strength. The calculation was performed using the mean seal strength values. It was determined that a sample size of 15 would be adequate for peel testing.

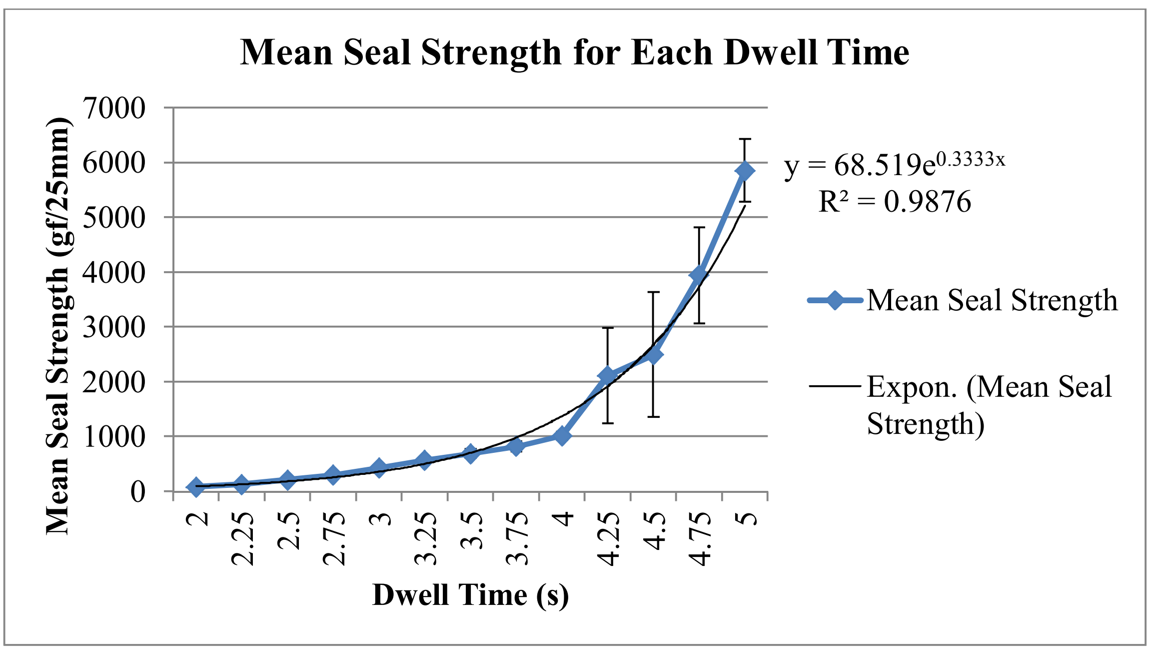

Figure 31. Preliminary Peel Testing - Mean Seal Strength for Each Dwell Time.

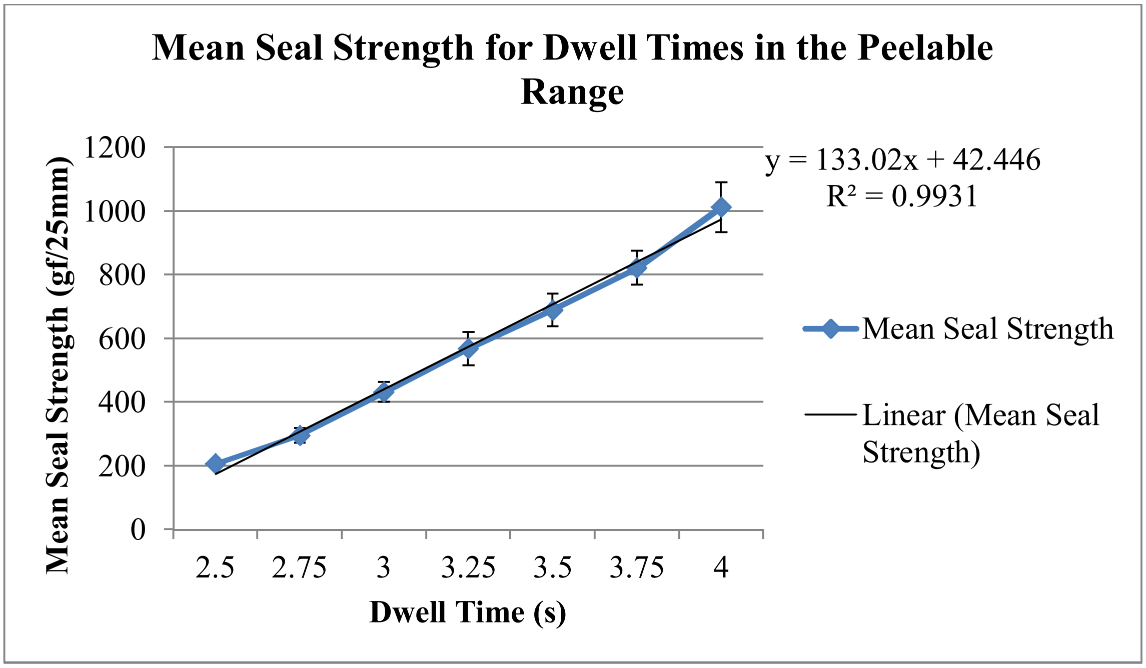

Figure 32. Preliminary Peel Testing - Mean Seal Strength for Dwell Times in the Peelable Range.

The chart in Figure 31 shows a plot of the mean seal strength values calculated for each dwell time plotted against the dwell time. The chart shows a general upward trend in seal strength as dwell time increases. Another point to consider is the large standard deviations associated with dwell times considered to be in the fusion range. This can be attributed to the fact that a tensile test of a fusion seal is affected by tear resistance, tensile strength of the material and the strength of the seal itself. This is because the sealed area of the sample is stronger than the unsealed area, and the failure of the sample occurs because the material begins to tear or stretch. The chart also shows that an exponential trend line is a good fit for the data with a coefficient of determination of 0.9876.

After dwell times were narrowed down to the peelable range of 2.5s to 4.0s, analysis of variance (ANOVA) was used to compare the means for the logarithm of average load per width (logload) for the selected dwell times. The ANOVA procedure tested the following hypotheses:

H_o: Means are equal for the selected dwell times.

H_A: At least one mean is different for the selected dwell times.

The ANOVA output from SAS is shown in Table 6 below. Since the p-value is less than α, the null hypothesis was rejected. That is, the ANOVA test showed that at least one dwell time produced seals with a mean seal strength that was significantly different from the mean seal strength values calculated for the other dwell times.

Table 6. ANOVA Results.

The GLM Procedure

Dependent Variable: logload

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	6	3.56509369	0.59418228	204.33	<.0001
Error	61	0.17738900	0.00290802		
Corrected Total	67	3.74248269			

R-Square	Coeff Var	Root MSE	logload Mean
0.952601	2.001815	0.053926	2.693857

Due to the outcome of the ANOVA procedure, it was deemed appropriate to determine which dwell times produced significantly different mean seal strengths. Thus, SAS was used to perform a least significant differences (LSD) comparison of the means for each dwell time with a 95% confidence level. The results of this test can be seen in Table 7 below. The check mark is used to indicate that the comparison is significantly different. The results of the SAS program showed that all means were significantly different from each other.

Table 7. Comparison of Means - Significantly Different Means.

	2.5s	2.75s	3s	3.25s	3.5s	3.75s	4s
2.5s	-	✓	✓	✓	✓	✓	✓
2.75s	✓	-	✓	✓	✓	✓	✓
3s	✓	✓	-	✓	✓	✓	✓
3.25s	✓	✓	✓	-	✓	✓	✓
3.5s	✓	✓	✓	✓	-	✓	✓
3.75s	✓	✓	✓	✓	✓	-	✓
4s	✓	✓	✓	✓	✓	✓	-

Since all dwell times produced mean seal strengths that were significantly different, all dwell times were acceptable for use in further testing. However, in order to limit the total number of samples required for testing, only 6 dwell times were selected for burst testing. The dwell times selected were 2.5s, 2.75s, 3s, 3.25s, 3.75s, and 4s.

Preliminary Burst Testing Results

The 6 dwell times selected in the previous section were used to seal pouches for burst testing. The pouches produced were tested on the Lippke inflation burst tester at 1.0 inch plate gap. If a pouch leaked before seal failure occurred, the burst pressure value and burst peeling time were thrown out. All seals had a peeling failure mechanism. All leaks occurred at the location of the septa.

Table 8. Preliminary Burst Tests - Mean Burst Peeling Time and Equivalent Crosshead Speed.

Dwell Time (s)	Mean Burst Peeling Time (s)	Std. Dev.	Equivalent Crosshead Speed (in/min)
2.5	4.91	0.95	24.43
2.75	6.10	2.68	19.69
3	6.72	1.33	17.85
3.25	10.41	2.62	11.53
3.75	19.48	4.02	6.16
4	21.79	5.25	5.51

Table 8 shows a summary of the burst peeling times recorded during the preliminary burst testing. One point of interest is the equivalent crosshead speed. This value was calculated to determine the speed at which a corresponding peel test would need to be pulled to match the peeling time of the burst test. The value was calculated using the mean burst peeling time. An aspect that must be considered is the maximum rate at which the SATEC Instron T10000 can pull a sample. The maximum crosshead speed capability of this machine is 20 in/min. Since the 2.5s dwell time resulted in an equivalent crosshead speed of greater than 20 in/min, it was eliminated from further testing leaving only 2.75s, 3s, 3.25s, 3.75s, and 4.0s.

Table 9. Preliminary Burst Tests - Mean Burst Pressure and Sample Number Required.

Dwell Time (s)	Mean Burst Pressure (PSI)	Std. Dev.	Sample Number Required
2.5	1.959	0.382	15
2.75	2.321	0.806	47
3	3.128	0.368	6
3.25	4.141	0.827	16
3.75	7.310	1.334	13
4	8.849	1.394	10

Table 9 shows the mean burst pressures and the sample numbers required to ensure that the margin of error for a 95% confidence interval would be within 10% of the mean burst pressure. It is clear that the 2.75s dwell time had a much higher sample number required, which suggests that there must be an anomaly in the data obtained. In Table 10, it can be seen that sample 5 at the 2.75s dwell time had a much higher burst peeling time. This may indicate that there was a leak occurring. Accordingly, the resulting burst pressure was higher. This value may be causing a much higher variability and sample number required. It was decided that sample size of 15 would be adequate for burst testing.

Table 10. Preliminary Burst Testing - 2.75s Raw Data.

Sample	Burst Peeling Time (s)	Burst Pressure (PSI)
1	7.06	3.017
2	7.06	2.611
3	5.06	2.466
4	4.43	2.234
5	12.72	4.105
6	6.16	1.769
7	2.47	1.508
8	5.44	2.147
9	5.25	2.016
10	5.31	1.334
Mean	**6.10**	**2.321**
Std. Dev.	2.68	0.806
Margin of Error		0.1
Sample Number Required		47

The chart in Figure 33 shows an overall upward trend in mean burst pressure as dwell time increases. This means that pouches sealed using longer dwell times resulted in higher burst pressures when burst tested using the Lippke 4500 inflation tester. It is also evident that the standard deviation associated with the burst tests generally increased as dwell time increased.

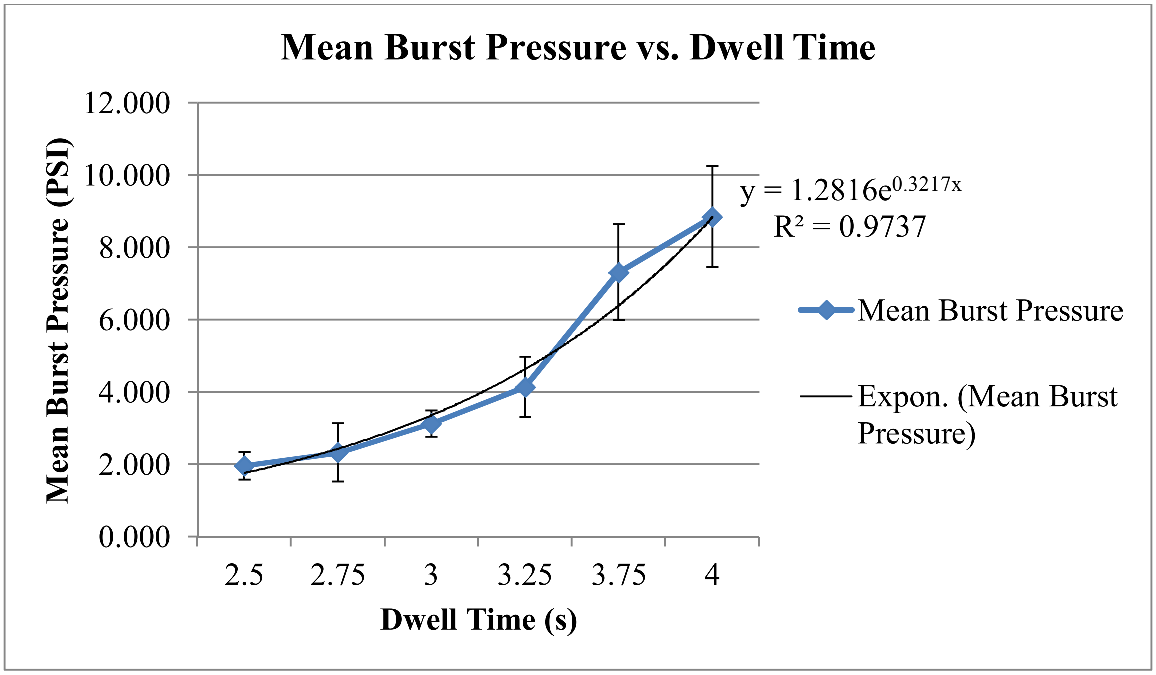

Figure 33. Preliminary Burst Testing - Mean Inflation Burst Pressure vs. Dwell Time.

The preliminary testing was used to shape the design of the experiment. First, the dwell times were narrowed down to the peelable range of seals with at least 200gf/in seal strength. The preliminary peel data was also used to determine if there were any dwell times that resulted in peeling speeds that exceed the limitations of the tensile tester. The results of these tests helped narrow the dwell times down to a range of 2.5s to 4.0s. Then, the sample size required to ensure that the margin of error for a 95% confidence interval would be within 10% of the mean seal strength was calculated. The results show that a sample size of 15 was required for both peel testing and burst testing for the experiment.

Burst Testing Results

Since the crosshead speed and gauge length were controlled to match with corresponding burst testing, the burst testing was performed first. The chart in Figure 34 shows the mean burst peeling time at each plate gap for each dwell time. It is clear that, with most testing configurations, the burst peeling time increased with the dwell time, except for the case of the compression burst tests.

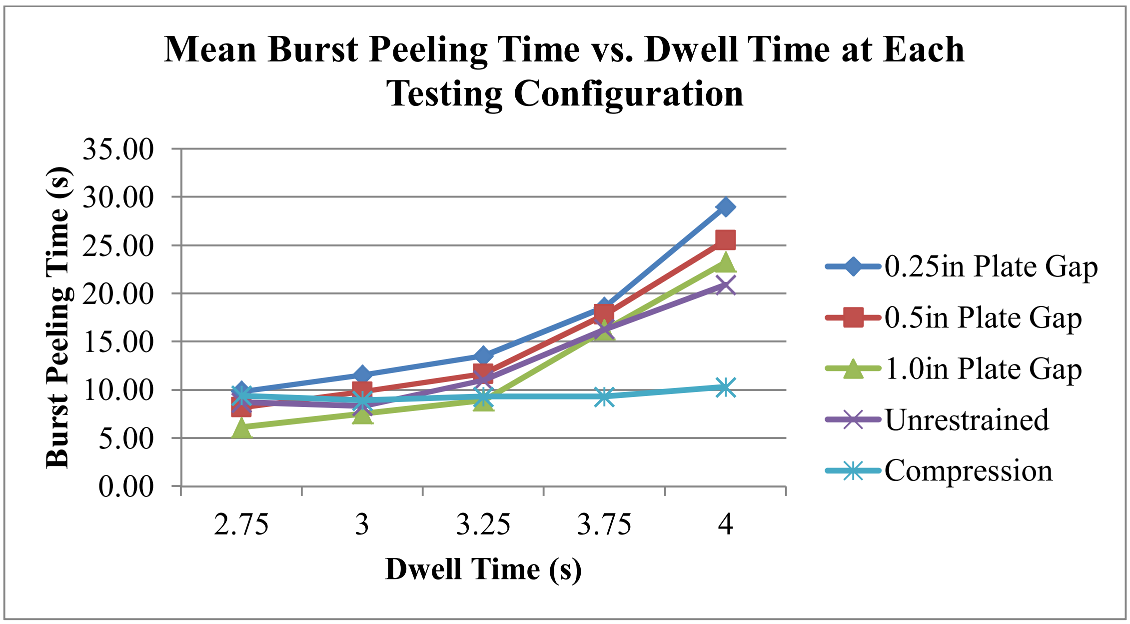

Figure 34. Burst Testing - Mean Burst Peeling Time vs. Dwell Time.

In the compression burst test, the peeling times were roughly the same across the dwell times. To further analyze the data, an ANOVA test was performed to determine if there was a significant difference between the mean peeling times for the compression test at each dwell time. The ANOVA test was performed with a 95% confidence level. The procedure tested the following hypotheses:

H_0: Mean peeling times are equal for the selected dwell times.

H_A: At least one mean peeling time is different for the selected dwell times.

As can be seen from Table 11, the p-value was greater than the level of significance (0.05), so the decision was to fail to reject the null hypothesis. In other words, there was insufficient evidence at the 95% confidence level to conclude that the mean peeling times were different for the compression test across each of the dwell times. These results may be explained by the fact that the top platen of the compression tester moved downward at a constant rate of 0.5in/min and the pouches were filled with water. Water is a not compressible, which means changes in volume of the pouch caused peeling of the seal. Since the platen came down at a constant rate, the peeling time should have been approximately the same across all dwell times.

Table 11. ANOVA Results - Comparison of Peeling Times for Compression Burst Test.

The GLM Procedure

Dependent Variable: peeltime

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	4	15.7671253	3.9417813	1.00	0.4159
Error	70	277.1934667	3.9599067		
Corrected Total	74	292.9605920			

R-Square	Coeff Var	Root MSE	peeltime Mean
0.053820	21.09966	1.989951	9.431200

When comparing the inflation burst pressure data in Figure 35, it can be seen that the 0.25in plate gap resulted in higher burst pressures than the other configurations. LSD comparisons were performed to determine which mean burst pressures were significantly different from each other at a 95% confidence level. The LSD comparisons were performed for each dwell time to compare the mean burst pressure obtained from each plate gap. The results can be seen in Table 12 to Table 16. The checkmarks denote comparisons that were considered statistically different at a confidence level of 95% (level of significance = 0.05). An interesting note is that the unrestrained burst test and the 1.0in plate gap resulted in no statistically significant differences. On the other hand, the 0.25in plate gap was statistically different from all other configurations. These results indicate that the 0.25in plate gap resulted in significantly higher burst pressures than all other inflation burst tests configurations.

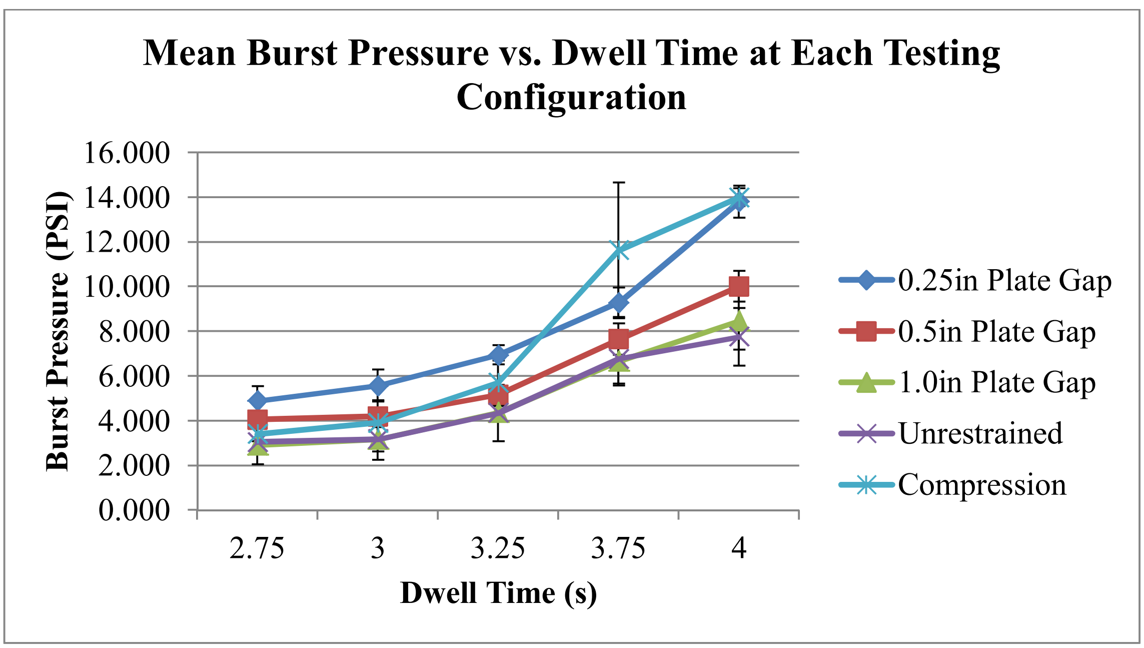

Figure 35. Burst Testing - Mean Burst Pressure vs. Dwell Time at Each Testing Configuration.

Table 12. LSD Comparison of Mean Burst Pressures for Each Testing Configuration for 2.75s Dwell Time.

	Compression	0.25in	0.5in	1in	Unrestrained
Compression		✓	✓		
0.25in	✓		✓	✓	✓
0.5in	✓	✓		✓	✓
1in		✓	✓		
Unrestrained		✓	✓		

Table 13. LSD Comparison of Mean Burst Pressures for Each Testing Configuration for 3.0s Dwell Time.

	Compression	0.25in	0.5in	1in	Unrestrained
Compression		✓		✓	✓
0.25in	✓		✓	✓	✓
0.5in		✓		✓	✓
1in	✓	✓	✓		
Unrestrained	✓	✓	✓		

Table 14. LSD Comparison of Mean Burst Pressures for Each Testing Configuration for 3.25s Dwell Time.

	Compression	0.25in	0.5in	1in	Unrestrained
Compression		✓		✓	✓
0.25in	✓		✓	✓	✓
0.5in		✓		✓	✓
1in	✓	✓	✓		
Unrestrained	✓	✓	✓		

Table 15. LSD Comparison of Mean Burst Pressures for Each Testing Configuration for 3.75s Dwell Time.

	Compression	0.25in	0.5in	1in	Unrestrained
Compression		✓	✓	✓	✓
0.25in	✓		✓	✓	✓
0.5in	✓	✓			
1in	✓	✓			
Unrestrained	✓	✓			

Table 16. LSD Comparison of Mean Burst Pressures for Each Testing Configuration for 4.0s Dwell Time.

	Compression	0.25in	0.5in	1in	Unrestrained
Compression			✓	✓	✓
0.25in			✓	✓	✓
0.5in	✓	✓		✓	✓
1in	✓	✓	✓		
Unrestrained	✓	✓	✓		

Another result that is of interest is the number of occurrences of each type of burst location. Figure 36 indicates that there were many more center bursts than corner bursts. This was contrary to expectations. With seal geometry that creates 90 degree angles at the corners, it was expected that the corner would become what is known as a "stress concentrator." This concentration applies to these areas, and it is generally expected that these areas are where most of the failures will occur. The data can also be analyzed in regards to dwell time (Figure 37). The data presented in Figure 37, also show that there were more center bursts than corner bursts for each dwell time. It may be that the dimensions of these pouches had more of an effect on the location of the burst than the 90 degree corners. This could be of interest for future research.

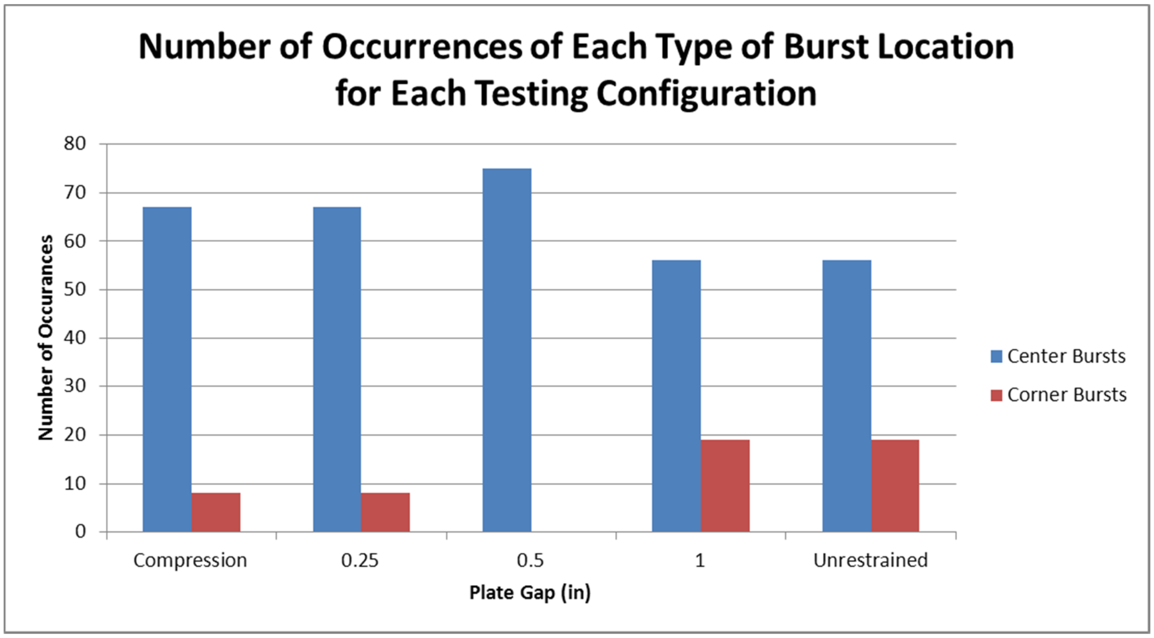

Figure 36. Number of Occurrences of Each Type of Burst Location for Each Testing Configuration.

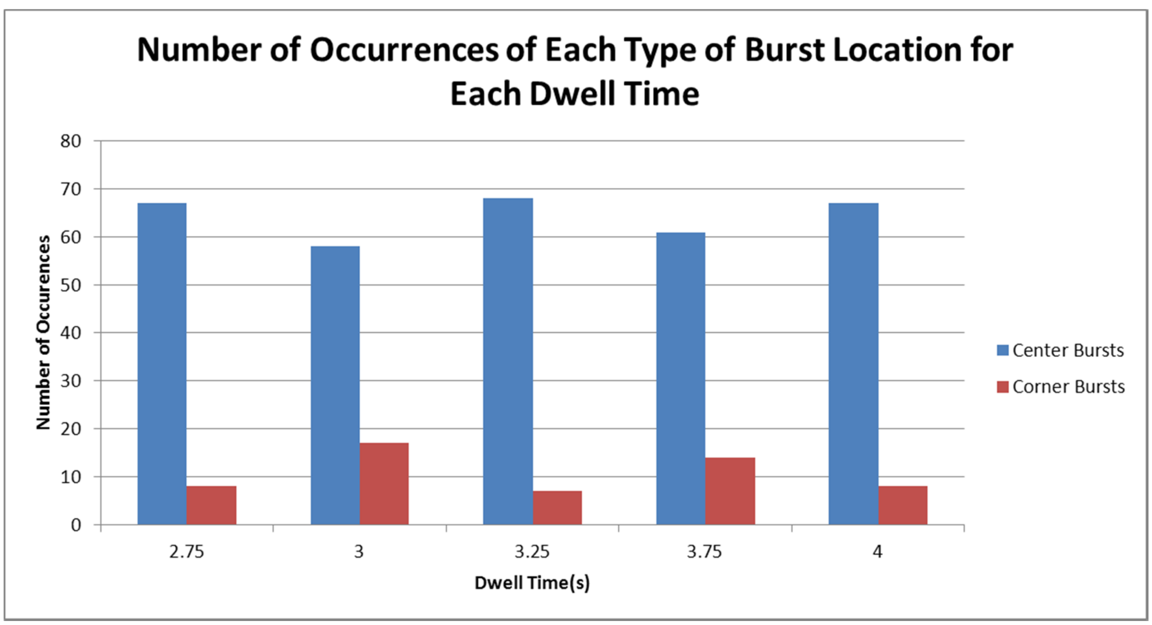

Figure 37. Number of Occurrences of Each Type of Burst Location for Each Dwell Time.

Peel Testing Results

Once the crosshead speeds and gauge lengths required for peel testing were determined, peel tests were performed. As can be seen from Figure 38, the data followed what appeared to be similar trends as the data from the burst tests. This indicated that there was a relationship between the data, but the data needed to be analyzed using the methods described in the "Parameter Estimation" section of Chapter Two. The following section describes the results of the statistical analysis and the empirical equations developed.

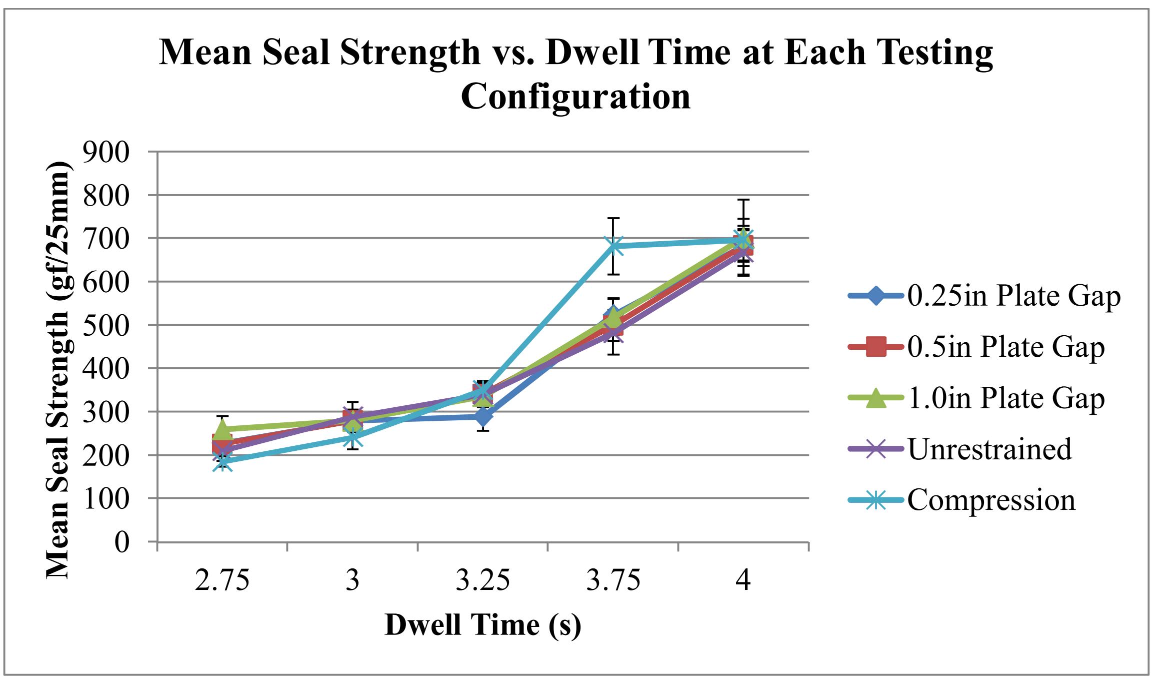

Figure 38. Peel Testing - Mean Seal Strength vs. Dwell Time.

Evaluation of Yam's Equation

Yam derived the relationship between seal strength and burst pressure and indicated that this relationship could be expressed as,

S= (D/2)P

where S is the seal strength (lbf/in), P is the burst pressure (PSI), and D is the plate gap (in). Therefore, if Yam's conjectured relationship is correct, the slope for the regression line should be equal to D/2. The two-stage least squares (2SLS) method described in the "Parameter Estimation" section of the literature review was used to estimate the slope for the experimental data. The hypotheses tested were:

$$H_0: \beta_1 = \left(\frac{D}{2}\right)$$

$$H_A: \beta_1 \neq \left(\frac{D}{2}\right)$$

A two-tailed t-test was performed to determine whether the estimated slope was significantly different from the theoretical slope.

The estimate of the slope parameter (β1) can be seen in

Table 17 for each testing configuration. The test statistic can also be seen in

Table 17. For a level of significance of 5%, if the test statistic was less than -2.776 or greater than 2.776, the null hypothesis was rejected. There is significant evidence at $\alpha = 0.05$ to conclude that the estimated slopes for compression burst testing,

0.50in plate gap, 1.0in plate gap, and unrestrained burst testing were not equal to the theoretical slopes calculated for each one. This indicates that the theoretical model studied does not work for any testing configuration except the inflation burst tester with a 0.25in plate gap. Even the test statistic for the 0.25in plate gap data was also very close to the rejection region.

Table 17. Test Statistics for Each Testing Configuration.

	Compression	**0.25in Plate Gap**	**0.50in Plate Gap**	**1.0in Plate Gap**	**Unrestrained**
Slope Estimate (B1)	0.120437	0.110637	0.146006	0.178845	0.175864
Theoretical Slope (D/2)	0.085	0.125	0.25	0.5	0.75
Approx. Std. Error	0.00699	0.00526	0.00282	0.00386	0.00973
Test Statistic	5.06967	-2.73061	-36.8773	-83.20078	-59.00678
Coefficient of Determination	0.9623	0.9498	0.9833	0.9849	0.9359
Decision	Reject Null Hypothesis	Fail To Reject Null Hypothesis	Reject Null Hypothesis	Reject Null Hypothesis	Reject Null Hypothesis

The theoretical relationship derived by Yam may not work with the pouches in this study due to the geometry of the pouch. Yam's equation is based on hoop stress and a force balance that assumes an inflated pouch forms a cylindrical shape. However, an inflated pouch does not form a cylindrical shape due to end seals. With flat end seals, an inflated pouch forms more of a pillow-like shape. Additionally, as Wachala noted, the tangent to the sealing surface at burst does not form a 90 degree angle (Figure 22) as is required for hoop stress (Equation (2)) to apply. Other reasons the theoretical equation does not work may be because of wrinkles that form and stretching that occurs during inflation. Materials were selected to minimize stretching, but in some cases there may have been unpreventable stretching that occurred. An assumption associated with the hoop stress equation is that the wall of the container stretches minimally. There was nothing that could have been done to prevent wrinkles, but it was noted that less wrinkling occurred when burst testing at smaller plate gaps. It was also noted that wrinkling severely affected the burst peeling time. In some cases, the seal began to peel, and then a wrinkle began to form. When the wrinkle formed, it diverted the force to another area of a seal, and peeling started again in that area.

The following empirical equations were developed from the slope estimates in

Table 17. A chart comparing the predicted values to the experimental values and a table showing the percent difference is shown after each equation.

Compression Burst Empirical Equation

$$Seal\ Strength\ \left(\frac{lbf}{in}\right) = 0.120437(Burst\ Pressure\ (PSI)) \tag{26}$$

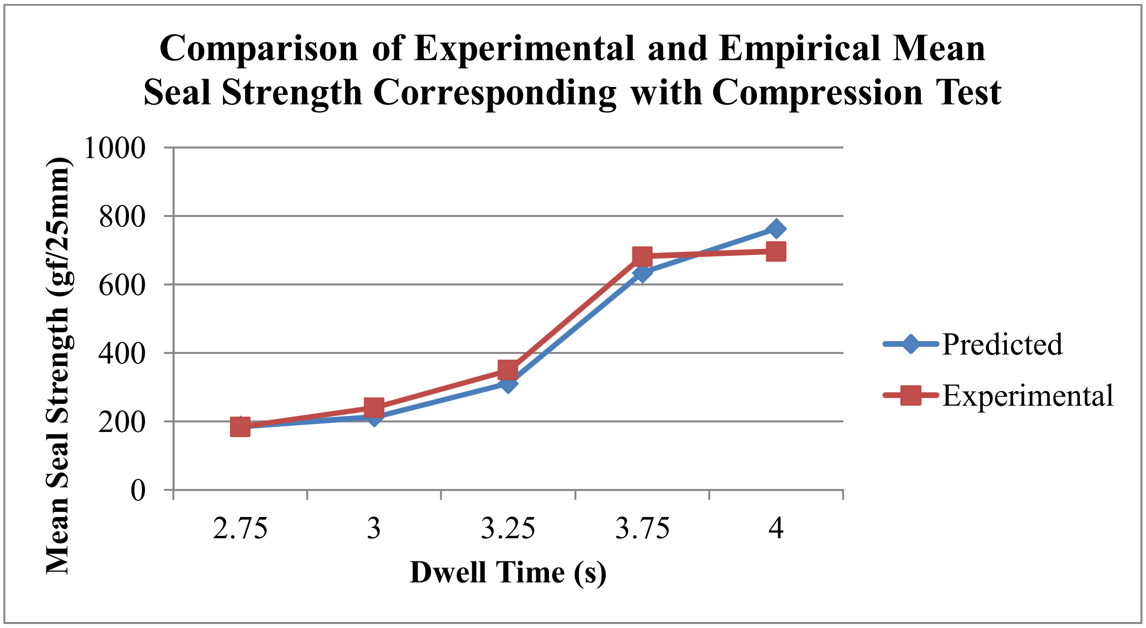

Figure 39. Comparison of Experimental and Empirical Mean Seal Strength Corresponding with Compression Test.

Table 18. Percent Difference Between Experimental and Empirical Mean Seal Strengths Corresponding with Compression Test.

Dwell Time (s)	Experimental Seal Strength (gf/25mm)	Predicted Seal Strength (lbf/in)	% Difference
2.75	184	186	1%
3	241	213	18%
3.25	350	311	17%
3.75	682	635	11%
4	697	764	14%

Using Equation (26), the predicted seal strength was calculated for each dwell time. The predicted seal strength was calculated using the mean burst pressure from the compression tests. The predicted data calculated from the empirical equation appear to be close to the experimental data obtained from testing (Figure 39). However, it can be seen from the data presented in numerical form in Table 18 that the percent difference between the two values ranged from 1% to 18%. At some dwell times, the empirical equation underestimates the seal strength. At other dwell times, the empirical equation overestimates the seal strength.

0.25in Plate Gap Inflation Burst Empirical Equation

$$Seal\ Strength\ \left(\frac{lbf}{in}\right) = 0.110637(Burst\ Pressure\ (PSI)) \tag{27}$$

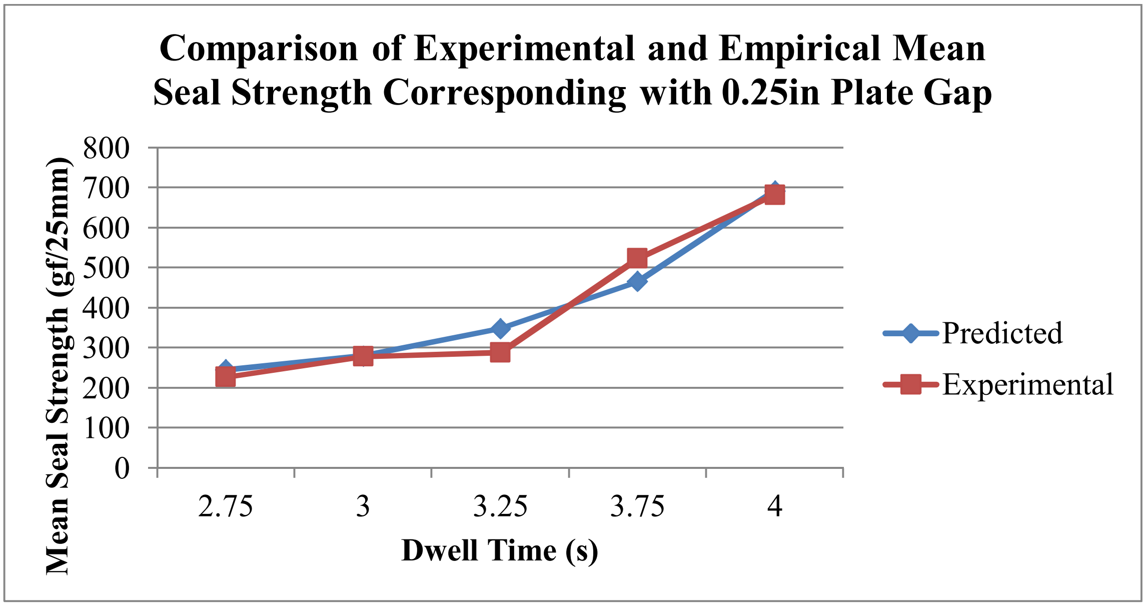

Figure 40. Comparison of Experimental and Empirical Mean Seal Strength Corresponding with 0.25in Plate Gap.

Table 19. Percent Difference Between Experimental and Empirical Mean Seal Strengths Corresponding with 0.25in Plate Gap.

Dwell Time (s)	Experimental Seal Strength (gf/25mm)	Predicted Seal Strength (lbf/in)	% Difference
2.75	228	245	11%
3	279	279	0%
3.25	288	349	28%
3.75	524	466	17%
4	682	693	2%

Figure 40 also shows that the predicted data appear to be close to the experimental data. However, there is a large percent difference at the 3.25s dwell time (Table 19). One interesting note is that the percent difference between the predicted value and the experimental value was 0% at the 3s dwell time.

0.5in Plate Gap Inflation Burst Empirical Equation

$$Seal\ Strength\ \left(\frac{lbf}{in}\right) = 0.146006(Burst\ Pressure\ (PSI)) \qquad (28)$$

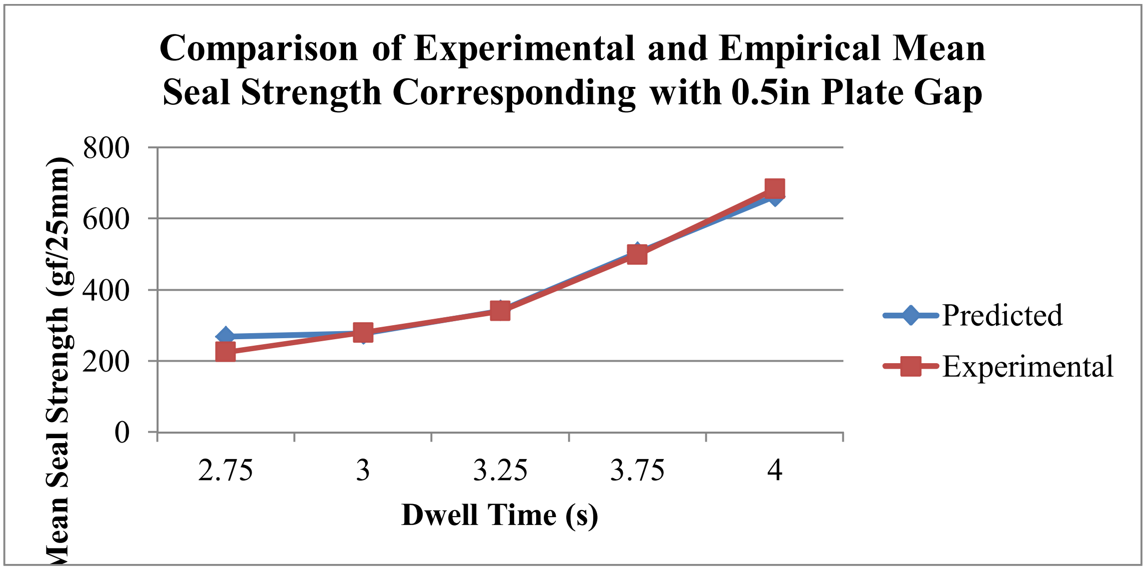

Figure 41. Comparison of Experimental and Empirical Mean Seal Strength Corresponding with 0.5in Plate Gap.

Table 20. Percent Difference Between Experimental and Empirical Mean Seal Strengths Corresponding with 0.5in Plate Gap.

Dwell Time (s)	Experimental Seal Strength (gf/25mm)	Predicted Seal Strength (lbf/in)	% Difference
2.75	226	268	26%
3	280	277	2%
3.25	341	342	1%
3.75	499	506	2%
4	684	663	5%

Other than the 2.75s dwell time, the predicted data in Figure 41 appear to be very close to the experimental data. Further verification of this can be seen in Table 20 by comparing the percent difference for each dwell time. At the 2.75s dwell time, the percent difference between the predicted value and the experimental value was much higher.

1.0in Plate Gap Inflation Burst Empirical Equation

$$Seal\ Strength\ \left(\frac{lbf}{in}\right) = 0.178845(Burst\ Pressure\ (PSI)) \tag{29}$$

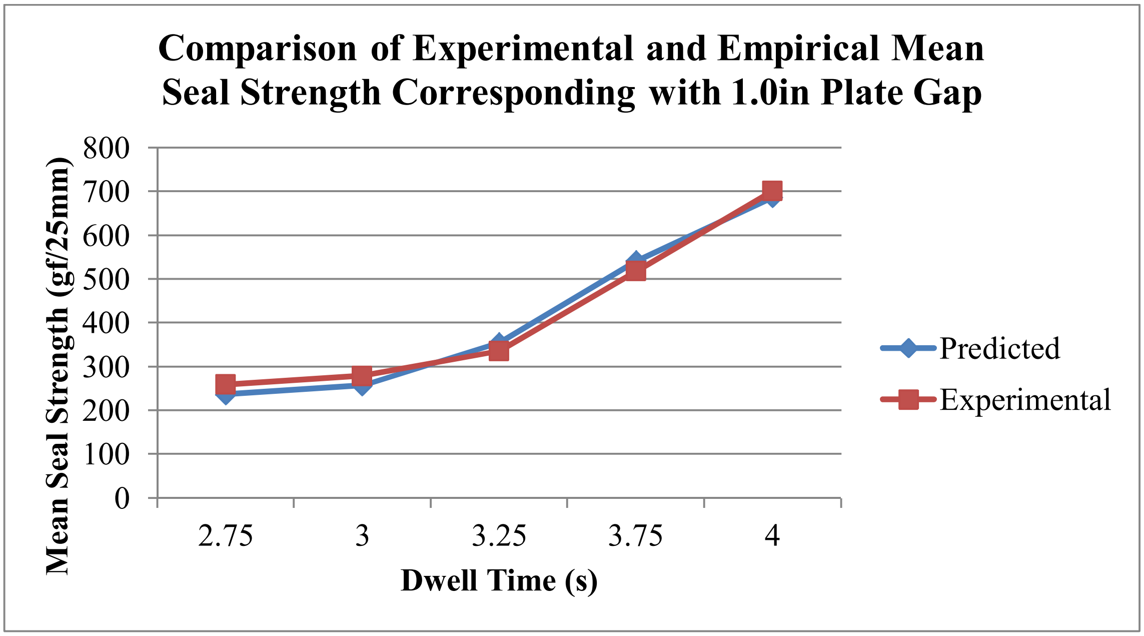

Figure 42. Comparison of Experimental and Empirical Mean Seal Strength Corresponding with 1.0in Plate Gap.

Table 21. Percent Difference Between Experimental and Empirical Mean Seal Strengths Corresponding with 1.0in Plate Gap.

Dwell Time (s)	Experimental Seal Strength (gf/25mm)	Predicted Seal Strength (lbf/in)	% Difference
2.75	259	236	14%
3	279	257	12%
3.25	336	355	8%
3.75	518	541	7%
4	702	687	3%

The data for the 1.0in plate gap shown in Figure 42 shows that the predicted seal strengths are close to the experimental seal strengths. The percent difference between the two values can be seen in Table 21. The maximum percent difference for the 1.0in plate gap was only 14%, which indicates that the empirical equation does a good job of predicting the seal strength.

Unrestrained Inflation Burst Empirical Test

$$Seal\ Strength\ \left(\frac{lbf}{in}\right) = 0.175864(Burst\ Pressure\ (PSI)) \tag{30}$$

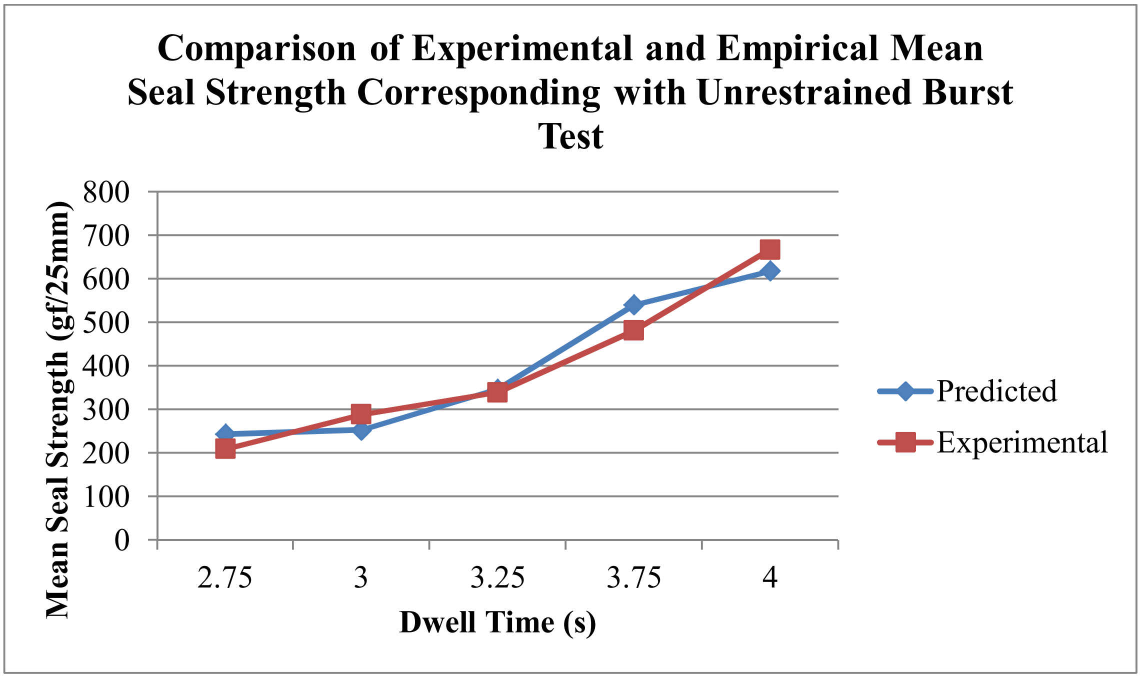

Figure 43. Comparison of Experimental and Empirical Mean Seal Strength Corresponding with Unrestrained Burst Test.

Table 22. Percent Difference Between Experimental and Empirical Mean Seal Strengths Corresponding with Unrestrained Burst Test.

Dwell Time (s)	Experimental Seal Strength (gf/25mm)	Predicted Seal Strength (lbf/in)	% Difference
2.75	209	243	22%
3	288	253	20%
3.25	339	347	3%
3.75	481	540	17%
4	667	618	11%

It can be seen from Figure 43 that the predicted data do not appear to be as close of a fit as the data from the other plate gaps. This is further verified by comparing the percent difference for each dwell time in Table 22. The data show that at most dwell times there is percent error of at least 11%, excluding the 3s dwell time.

Additionally, a visual representation of the percent differences was generated and can be seen in Figure 44. Figure 44 shows a comparison of the percent differences between predicted seal strengths and experimentally obtained seal strengths for each testing configuration. The data show that the percent difference can range from 0 to almost 30 percent.

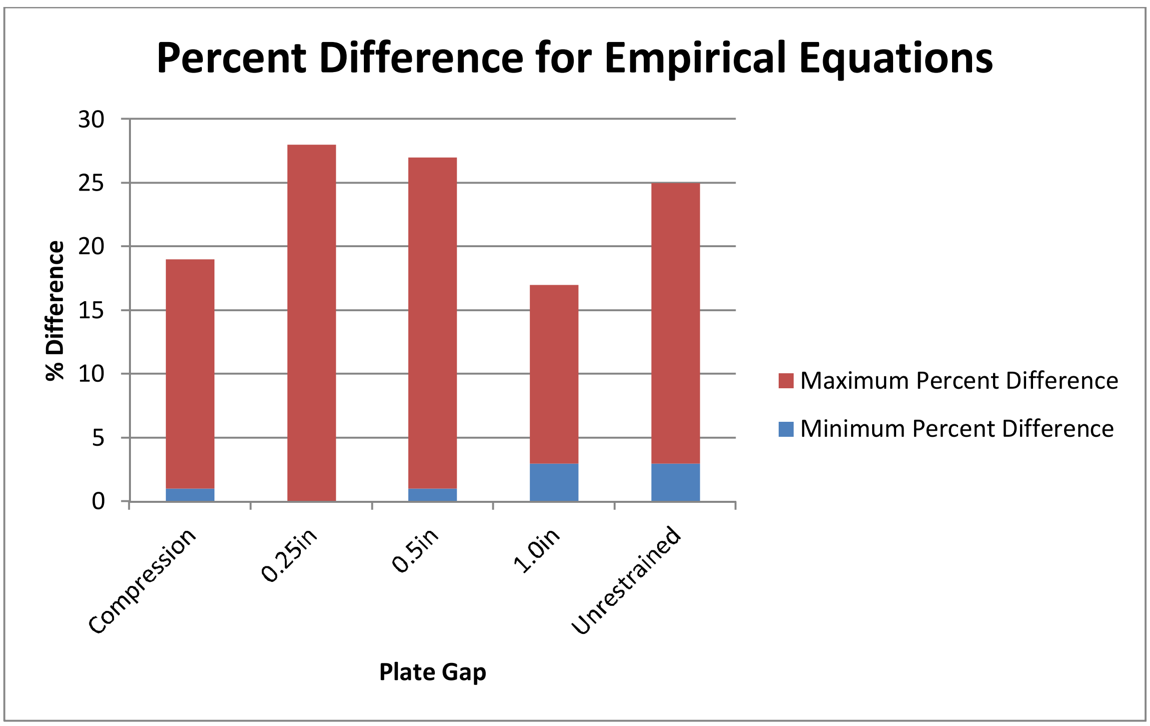

Figure 44. Percent Difference for Empirical Equations.

The results of the statistical analysis in this study show that the only empirical equation that did not have a slope that was significantly different from the theoretical slope was the 0.25in plate gap. However, since the slope for the 0.25in plate gap was close to being significantly different from the theoretical slope, it can be concluded that Yam's theoretical model is not universally applicable to all pouches.

One caveat that should be mentioned is that Equation (26) – Equation (30) are only useful for this particular set of materials and size of pouches. This is true, in general of empirical equations. It is also necessary to match the crosshead speed to pull a seal within the same amount of time as the burst peeling time. Additionally, the predicted seal strength values would only be valid for peelable seals that are peel tested with controlled crosshead speeds and gauge lengths.

In addition, the following graphs were developed to show a comparison between the empirical formulas and Yam's equation. As the graphs show in **Error! Reference source not found.** through **Error! Reference source not found.**, the empirical formulas appear to be a good fit for the experimentally obtained data. The line representing Yam's equation does not appear to be a good fit for the experimental data. Baéz found that Yam's equation had a tendency to overestimate the burst pressure when using seal strength as the known parameter. Using this assumption, it would be expected that Yam's equation would underestimate seal strength when using burst pressure as the known parameter. However, the comparisons shown in **Error! Reference source not found.** through **Error! Reference source not found.** clearly show the opposite. When using Yam's equation for inflation burst testing, the predicted values for seal strength are overestimations. In contrast, **Error! Reference source not found.** shows that Yam's equation underestimates the seal strength when using data obtained from compression testing. Additionally, overestimation appears to increase as the restraining plate gap increases for inflation burst testing.

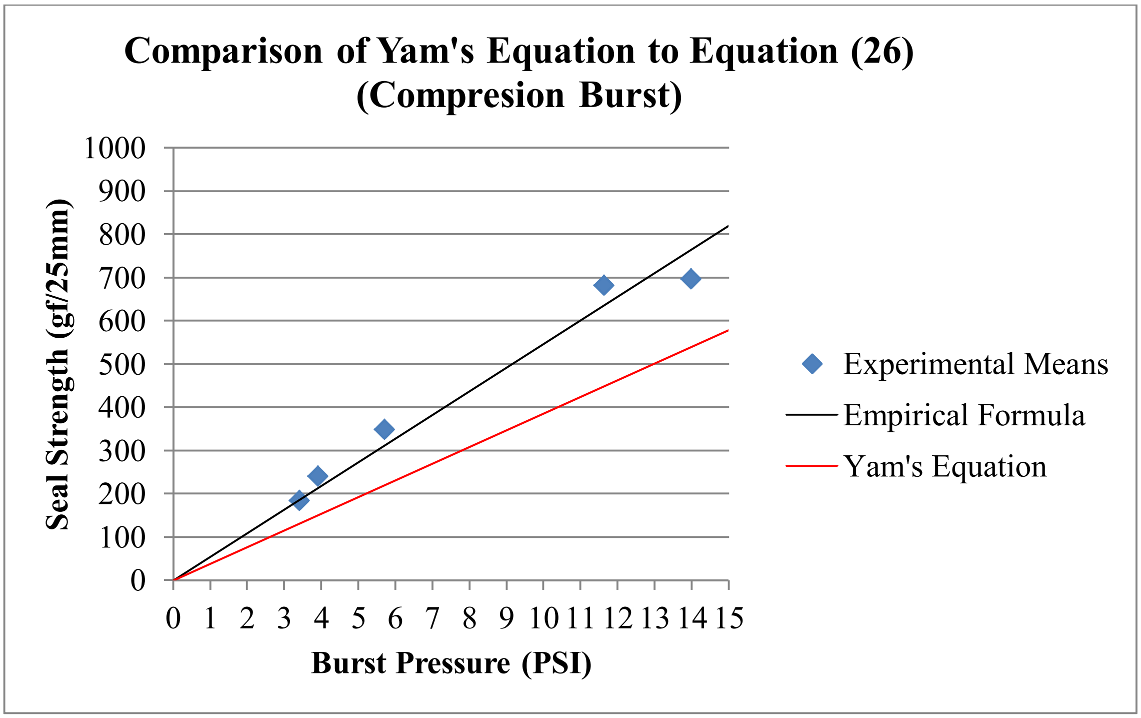

Figure 45. Comparison of Yam's Equation to Empirical Formula for Compression Burst Testing.

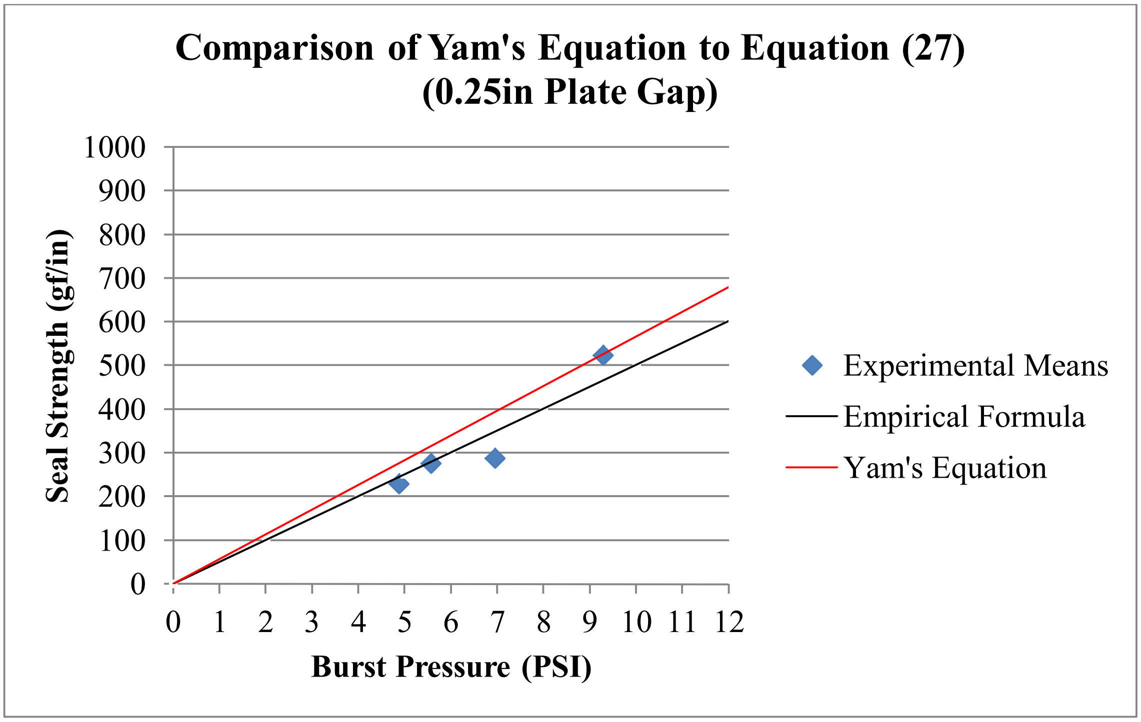

Figure 46. Comparison of Yam's Equation to Empirical Formula for 0.25in Plate Gap.

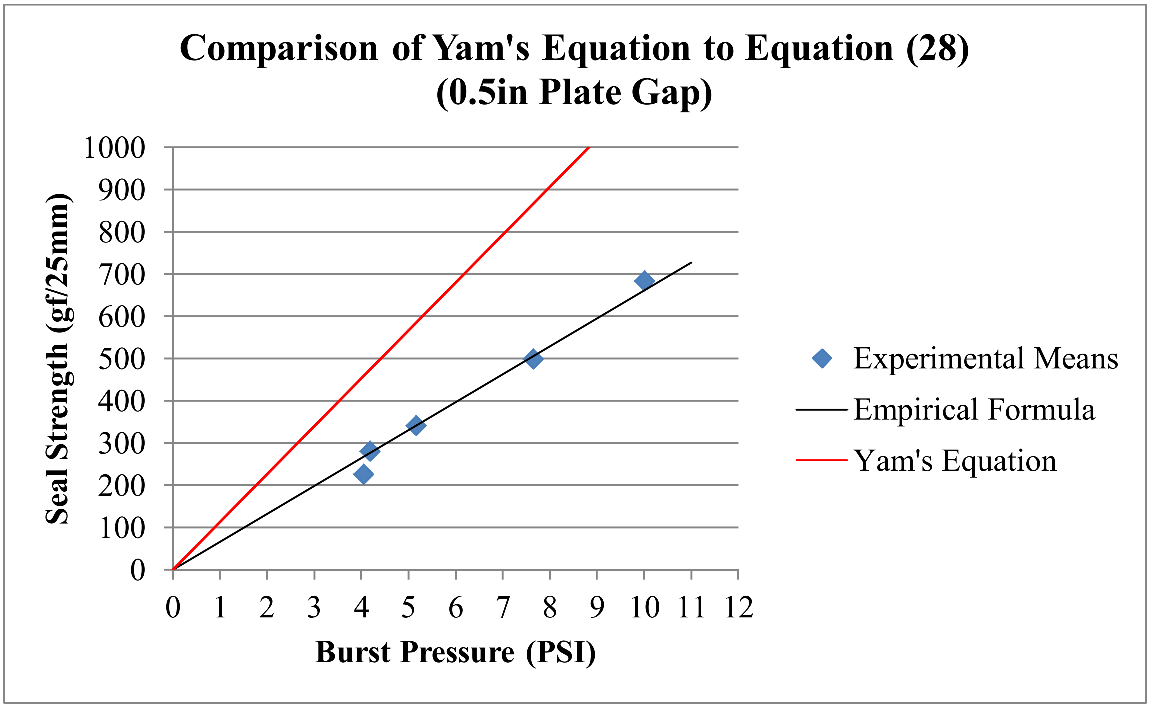

Figure 47. Comparison of Yam's Equation to Empirical Formula for 0.5in Plate Gap.

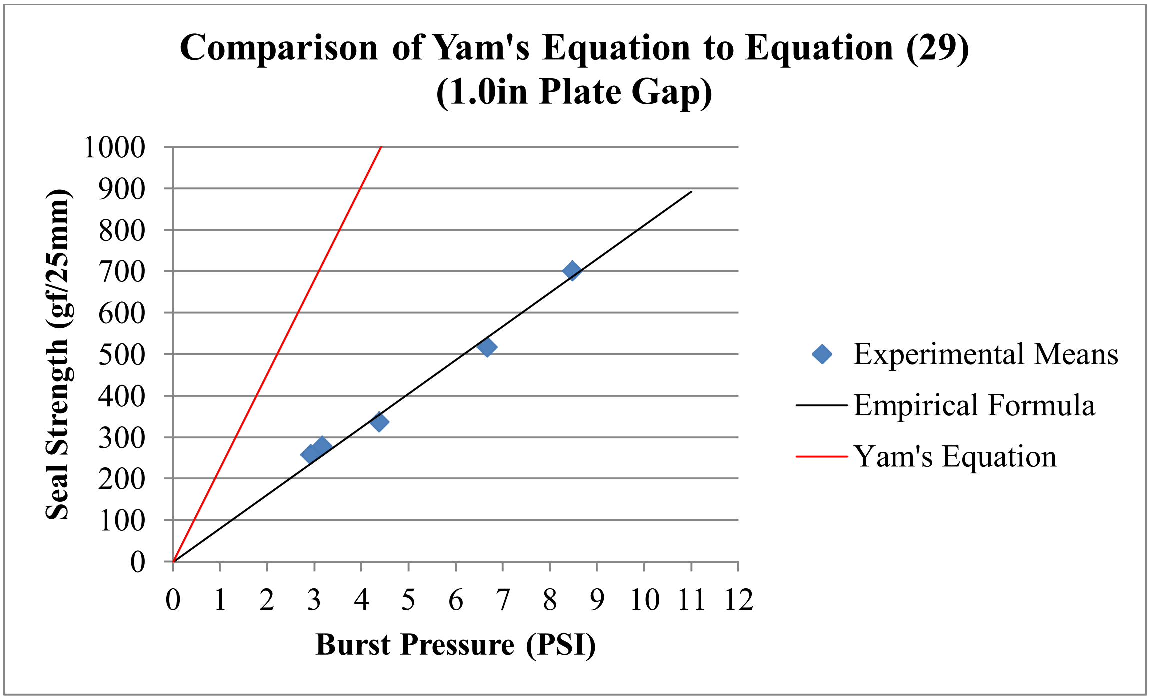

Figure 48. Comparison of Yam's Equation to Empirical Formula for 1.0in Plate Gap.

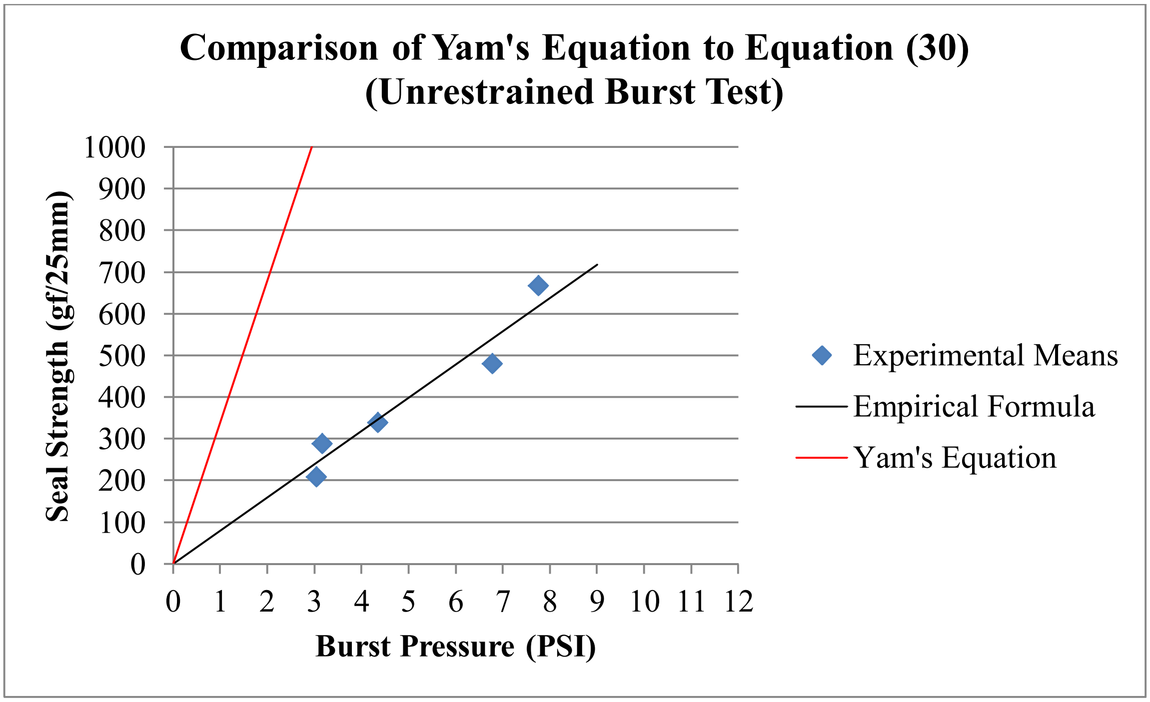

Figure 49. Comparison of Yam's Equation to Empirical Formula for Unrestrained Burst Testing.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

The validity of these results can be attributed to the fact that more samples were tested than in any other previous study. In contrast to other studies, which used ordinary least squares estimation to develop empirical equations, this study used a number of other statistical concepts to handle the data properly. The work presented here also studied pouches with peelable, flat seals instead of fusion, flat seals or peelable, chevron seals.

This study treated the burst pressures obtained from testing as proxy variables for the true burst pressures of pouches that were destroyed during peel testing. To account for the inconsistency caused by using the proxy burst pressures, the dwell time was used as an instrumental variable. The instrumental dwell time variable was incorporated using the two-stage least squares method to estimate the slope of the equation. Past studies are flawed because they assume the burst pressures obtained from separate pouches are the exact values that would have been obtained from the pouches destroyed during peel testing and do not properly account for the error involved by using the two-stage least squares method of parameter estimation.

The results of this study lead to the conclusion that the theoretical equation does not work for any of the plate gaps used for burst testing except for the 0.25in plate gap. However, the test statistic for the 0.25in plate gap was very close to the rejection region. It should also be noted that the empirical formulas generated are only valid for the materials and pouch dimensions used in this study, which is the nature of using an empirical approach.

Another interesting result of this study is that the burst locations mostly occurred in the center of the seal. This was an unexpected result and there could be a number of different reasons why this occurred. With 90 degree corners formed by the seals, it was expected that bursts would occur mostly at the corners because of the stress concentration in these areas. It would be interesting to study pouches with curved corners. It may also be interesting to study whether pouch size, seal width, or material thickness has an effect on the location of the burst.

In comparison to this study, Baéz's first study showed that using Equation (17) resulted in gross overestimation of the burst pressures (Feliú-Baéz, 2001). Using the same logic, it would be expected that rearranging the equation to use the seal strength as the dependent variable would result in gross underestimation of the seal strength. The comparison of Yam's theoretical equation to the empirical equations developed in this study shows that Yam's equation over estimates seal strength and underestimates burst pressure when using inflation burst testing. When using compression testing, Yam's equation underestimates seal strength and overestimates burst pressure. Using the empirical equations developed in this study to predict seal strengths resulted in overestimation at some dwell times and plate gaps and underestimation at others.

Baéz's latest study shows that her empirical formulas gave a percent error of 1% to 7% when comparing the predicted burst pressure to the actual burst pressure obtained from testing. This is lower than the percent differences obtained in this study which resulted in 0% to 28%. However, Baéz studied pouches with a chevron seal. The chevron seal may create a stress concentration at the center of the sealed area which may lead to more consistent burst pressures, peeling times, and burst locations. The pouches in this study were made with flat seals. For future study, it may be interesting to study pouches of similar length and width but with varying angles of chevron seals. It would be interesting to determine if the angle of the chevron seal has any effect on the burst pressure, peeling time, and burst location

Yam found his theoretical model to be in good agreement with the data obtained from experimentation. One point to consider is that Yam was working with seals that were in the fusion range which resulted in much higher burst pressures. Since fusion seals have a completely different failure mechanism than peelable seals, it may be that the theoretical equation is better suited for these types of seals. In addition, it may be that the pouch more closely forms a cylindrical shape with stronger seals. Baéz's study did not agree with Yam's study, and it may be due to the use of peelable chevron pouches versus the fusion sealed MRE pouches used in Yam's study. For future study, it may be of interest to study pouches of same material and dimensions as used in this study but with fusion seals instead.

For future research, it may also be of interest to test plate gaps less than 0.25in with the same pouches as were used in this study. It appears that the compression test resulted in mostly lower percent differences between the predicted burst pressure and the experimental burst pressure. The distance between the platen and the bottom plate was only 0.17in for the compression test at burst. It is possible that smaller plate gaps would be more accurate for inflation burst tests. According to Equation (1) and ASTM 1140, the proper plate gap for the pouches used in this study is 0.25in. Further research in the relationship between seal strength and burst pressure using compression testing may be of interest to the packaging community. Additionally, it may be of interest to study the relationship between peel strength and burst strength for cup and lid systems.

APPENDICES

APPENDIX A

Sealing Matrix

Table 23. Sealing Matrix – Appendix.

Sample Number	**Temperature (F)**	**Pressure (PSI)**	**Dwell Time (S)**	**Maximum Load/Width (gf/25mm)**	**Average Load/Width (gf/25mm)**	**Type of Seal**
1	270	50	2			
2	270	50	2			
3	270	50	2			
4	270	50	2			
5	270	50	2			
6	270	50	2			
7	270	50	2			
8	270	50	2			
9	270	50	2			
10	270	50	2			
1	270	50	2.25			
2	270	50	2.25			
3	270	50	2.25			
4	270	50	2.25			
5	270	50	2.25			
6	270	50	2.25			
7	270	50	2.25			
8	270	50	2.25			
9	270	50	2.25			
10	270	50	2.25			
1	270	50	2.5			
2	270	50	2.5			
3	270	50	2.5			
4	270	50	2.5			
5	270	50	2.5			
6	270	50	2.5			
7	270	50	2.5			
8	270	50	2.5			
9	270	50	2.5			

Sample Number	Temperature (F)	Pressure (PSI)	Dwell Time (S)	Maximum Load/Width (gf/25mm)	Average Load/Width (gf/25mm)	Type of Seal
10	270	50	2.5			
1	270	50	2.75			
2	270	50	2.75			
3	270	50	2.75			
4	270	50	2.75			
5	270	50	2.75			
6	270	50	2.75			
7	270	50	2.75			
8	270	50	2.75			
9	270	50	2.75			
10	270	50	2.75			
1	270	50	3			
2	270	50	3			
3	270	50	3			
4	270	50	3			
5	270	50	3			
6	270	50	3			
7	270	50	3			
8	270	50	3			
9	270	50	3			
10	270	50	3			
1	270	50	3.25			
2	270	50	3.25			
3	270	50	3.25			
4	270	50	3.25			
5	270	50	3.25			
6	270	50	3.25			
7	270	50	3.25			
8	270	50	3.25			
9	270	50	3.25			
10	270	50	3.25			
1	270	50	3.5			
2	270	50	3.5			
3	270	50	3.5			
4	270	50	3.5			
5	270	50	3.5			
6	270	50	3.5			

Sample Number	Temperature (F)	Pressure (PSI)	Dwell Time (S)	Maximum Load/Width (gf/25mm)	Average Load/Width (gf/25mm)	Type of Seal
7	270	50	3.5			
8	270	50	3.5			
9	270	50	3.5			
10	270	50	3.5			
1	270	50	3.75			
2	270	50	3.75			
3	270	50	3.75			
4	270	50	3.75			
5	270	50	3.75			
6	270	50	3.75			
7	270	50	3.75			
8	270	50	3.75			
9	270	50	3.75			
10	270	50	3.75			
1	270	50	4			
2	270	50	4			
3	270	50	4			
4	270	50	4			
5	270	50	4			
6	270	50	4			
7	270	50	4			
8	270	50	4			
9	270	50	4			
10	270	50	4			
1	270	50	4.25			
2	270	50	4.25			
3	270	50	4.25			
4	270	50	4.25			
5	270	50	4.25			
6	270	50	4.25			
7	270	50	4.25			
8	270	50	4.25			
9	270	50	4.25			
10	270	50	4.25			
1	270	50	4.5			
2	270	50	4.5			
3	270	50	4.5			

Sample Number	Temperature (F)	Pressure (PSI)	Dwell Time (S)	Maximum Load/Width (gf/25mm)	Average Load/Width (gf/25mm)	Type of Seal
4	270	50	4.5			
5	270	50	4.5			
6	270	50	4.5			
7	270	50	4.5			
8	270	50	4.5			
9	270	50	4.5			
10	270	50	4.5			
1	270	50	4.75			
2	270	50	4.75			
3	270	50	4.75			
4	270	50	4.75			
5	270	50	4.75			
6	270	50	4.75			
7	270	50	4.75			
8	270	50	4.75			
9	270	50	4.75			
10	270	50	4.75			
1	270	50	5			
2	270	50	5			
3	270	50	5			
4	270	50	5			
5	270	50	5			
6	270	50	5			
7	270	50	5			
8	270	50	5			
9	270	50	5			
10	270	50	5			

APPENDIX B

SAS Seeds

Table 24. SAS Seeds Used for Each Testing Configuration.

Dwell Time (s)	Plate Gap (in)	Seed
2.75	0.25	62349
2.75	0.5	16379
2.75	1	19713
2.75	Unrestrained	39153
2.75	Compression	69459
3.25	0.25	24537
3.25	0.5	14595
3.25	1	40469
3.25	Unrestrained	67331
3.25	Compression	93365
3	0.25	71571
3	0.5	25775
3	1	31131
3	Unrestrained	3091
3	Compression	39411
4	0.25	06089
4	0.5	42831
4	1	95113
4	Unrestrained	43511
4	Compression	34733
3.75	0.25	70361
3.75	0.5	41047
3.75	1	03395
3.75	Unrestrained	17635
3.75	Compression	09697

APPENDIX C

Testing Schedule

Table 25. Testing Schedule.

Day	Seal	Burst Test	Peel Test
1	2.75s - 0.25in	-	-
1	2.75s - 0.5in	-	-
1	2.75s - 1in	-	-
1	2.75s - Unrestrained	-	-
2	-	2.75s - 0.25in	2.75s - 0.25in
2	-	2.75s - 0.5in	2.75s - 0.5in
2	-	2.75s - 1in	2.75s - 1in
2	-	2.75s - Unrestrained	2.75s - Unrestrained
3	3.25s - 0.25in		
3	3.25s - 0.5in		
3	3.25s - 1in		
3	3.25s - Unrestrained		
4		3.25s - 0.25in	3.25s - 0.25in
4		3.25s - 0.5in	3.25s - 0.5in
4		3.25s - 1in	3.25s - 1in
4		3.25s - Unrestrained	3.25s - Unrestrained
5	3s - 0.25in		
5	3s - 0.5in		
5	3s - 1in		
5	3s - Unrestrained		
6		3s - 0.25in	3s - 0.25in
6		3s - 0.5in	3s - 0.5in
6		3s - 1in	3s - 1in
6		3s - Unrestrained	3s - Unrestrained
7	4s - 0.25in		
7	4s - 0.5in		
7	4s - 1in		

Day	Seal	Burst Test	Peel Test
7	4s - Unrestrained		
8		4s - 0.25in	4s - 0.25in
8		4s - 0.5in	4s - 0.5in
8		4s - 1in	4s - 1in
8		4s - Unrestrained	4s - Unrestrained
9	3.75s - 0.25in		
9	3.75s - 0.5in		
9	3.75s - 1in		
9	3.75s - Unrestrained		
10		3.75s - 0.25in	3.75s - 0.25in
10		3.75s - 0.5in	3.75s - 0.5in
10		3.75s - 1in	3.75s - 1in
10		3.75s - Unrestrained	3.75s - Unrestrained
11	2.75s - Compression		
11	3.25s - Compression		
11	3s - Compression		
11	4s - Compression		
11	3.75s Compression		
12		2.75s - Compression	2.75s - Compression
12		3.25s - Compression	3.25s - Compression
12		3s - Compression	3s - Compression
12		4s - Compression	4s - Compression
12		3.75s Compression	3.75s Compression

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