# DESIGN OF POLYETHYLENE BASED MULTILAYER EXTRUSION BLOWN FILM FOR MANUFACTURE OF LEAK FREE PACKAGING

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Degree of Master of Science

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Thesis submitted in partial fulfillment of the requirements for the degree Master of Science

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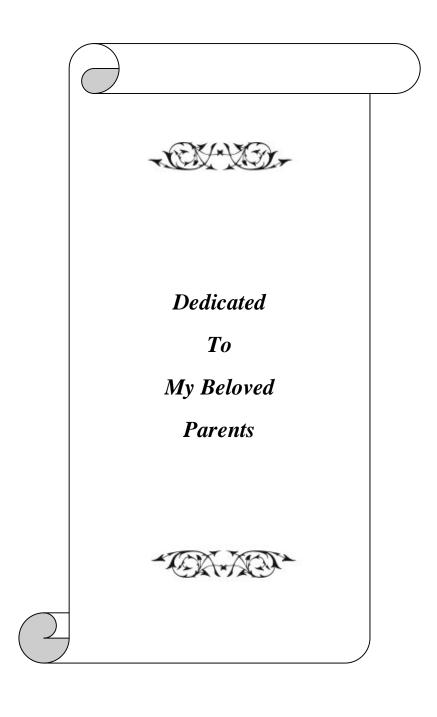
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#### **ABSTRACT**

Key words: LLDPE sealant, leak free, seal through contamination

Flexible packaging is a growing market and the majority of flexible package applications are for the food industry. The demand for process optimization and reduced production costs has led to an increase in flexible packaging. And reducing wastage in production line, during storing and transportation is a critical aspect which food product manufacturers are highly concerning. These wastages are higher for liquid products packing flexible materials. That is due to contamination in the seal area. Most of the time liquid products are packing in Vertical-Form-Fill-Seal (VFFS) machines. Therefore seal through contamination is highly occurring while packing of liquid products. The study uses three types of liquid and semi liquid products such as tomato sauce, spicy oil and water based perfume. Since the aggressiveness of these products leak percentage is higher with current material structures. Hence target of this study was to develop a PE based blown film extrusion material which can be used for the laminate structures for these selected products. Newly developed Linear Low Density Polyethylene (LLDPE) was replaced the sealant material of existing structure of those products. Also all the tests were carried out for both existing material structure and new material structure with developed LLDPE. The existing sealant material was blown using 80% LLDPE and 20% Low Density Polyethylene (LDPE) in all 3 layers. But newly developed sealant material was blown incorporating Polyolefin Plastomer (POP) and metallocene LLDPE materials to the inner and middle layers. The study tests a combination of different sealing temperatures and dwell time to determine the optimal sealing condition. Then optimal sealing condition was applied to the production line in order to trial the material structures. Leakages tests were conducted to the packed sachets and final results were determined. Developed blown film extrusion LLDPE film was shown good results compared to the existing material. From new sealant material leak percentage of tomato sauce, spicy oil and water based perfume could be reduced by 20%.

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#### LIST OF ABBREVIATIONS

LLDPE Linear Low Density Polyethylene

PE Polyethylene

POP Polyolefin Plastomer

FFS Form-Fill-Seal

CT PET Chemically Treated Polyethylene terepthalate

ALU Aluminium foil

BOPP Bi-axially Oriented Polypropylene

LDPE Low Density Polyethylene

EVA Ethylene Vinyl Acetate

HDPE High Density Polyethylene

PVC Polyvinyl Chloride

PVDC Polyvinylidene Chloride

BUR Blow up ratio

MD Machine Direction

TD Transverse Direction

HFFS Horizontal-Form-Fill-Seal

VFFS Vertical-Form-Fill-Seal

PLA Polylactic Acid

OPP Oriented Polypropylene

COF Coefficient of Friction

FDA Food and Drug Administration

GSM Grams per square meter

BAI Backscattered amplitude integral

PPA Polymer Processing Agent

DSC Differential Scanning Calorimeter

RPM Rounds per meter

ASTM American Standard Test Method

TDS Technical Data Sheet

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#### CHAPTER 01

#### INTRODUCTION

Packaging of any product is the link between the manufacturer and the consumer for the safe delivery of the product through the various stages starting from manufacturing, storing, transporting, distribution to marketing. Also Packaging is the technique of storing or placing a product in liquid, solid or powdered form into a protective container or wrapper to protect, carry, identify and merchandise the product. Customer and consumer requirements are tough to get high quality packaging with low cost. Demand for plastic packaging is increasing day by day. Flexible packaging has taken market share from rigid packaging as a result of number of factors such as Advances in technology, Lower cost and lower material consumption and Advantages of Paper, film and Aluminum foil. Flexible package is a material like foil or paper or plastic films sheeting which, when filled and sealed, acquires pliable shape and is used to form products like bags, envelopes, pouch, sachets and wraps.

Flexible thermoplastic films are often used in packages for liquid food products, such as milk, sauces ,oil and perfumes. The formation of these packages involves different sealing technologies, including heat sealing, hot air welding, ultrasonic welding and application of adhesives. Heat sealing is the most widely used sealing technique in form-fill-seal packaging, where two layers of film are heat, melted and bonded together with the application of pressure. A typical process involves compressing the films between two jaws that are both heated or one is heated. As the polymer melts under the compression force, the molecular segments diffuse across the film-film interface, thereby forming an intact seal with desirable strength.

The main process parameters of heat sealing are temperature, dwell time and the applied pressure. The seal strength is primarily controlled by the film-film interface temperature which is dependent on the jaw temperature and dwell time. Heat sealing is widely used in form-fill-seal packaging of liquid products. Packaging industries set tough requirements for quality. One of the problems encountered by them is the

contamination of seal area by the liquid product during heat sealing. Seal through contamination weaken the seal and product tends to leak from the seal. Relate to this matter researchers have done several type of researches. Heat sealing of Linear Low Density Polyethylene (LLDPE) films and developing a heat transfer model with liquid presence at film-film interface, Finding correlation between thermal behavior of a sealant and heat sealing of Polyolefin films and Develop high performance sealable films based on biodegradable/compostable blends are some of those researches. From this research I'm also going to contribute to the industry by developing a Polyethylene (PE) based compound for blown film extrusion packaging with free of leak seal. If we could develop a PE blend to eliminate seal through contamination of seal area by liquid product during heat sealing the wastage percentage while packing a liquid product would be minimized. Hence the total rejection of liquid product and packing material would be reduced drastically. Therefore from this research several parties would be beneficial such as liquid product manufacturer, packing material supplier to the manufacturer and finally the consumer of the product by getting a safe and quality packed product.

### CHAPTER 02 OBJECTIVE

The objective of the study is;

- Determining the effect of the components of multilayer laminated film structures on seal strength,
- Analyzing the effect of sealing machine process parameters on formation of leak free joint,
- Analyzing the effect of PE blend composition on formation of leak free sealing joint,

in packing applications of Sauce, Spicy oil and Water base perfume.

#### CHAPTER 03

#### LITERATURE REVIEW

#### 3.1 Importance of Plastic Packaging

Fluid products have been packaged in several types of containers across the globe. The general requirements of a fluid packaging container are as follows;

- It must be tamper proof, so that it is protected against fraudulent practices between the time of packaging and consumption
- It must be of such a size that the capacity always relates to the keeping quality of the product and that its contents correspond to the consumer's daily requirements.
- It must be clean and attractive

A container of good quality must prevent post-processing contamination from bacterial sources. It should be prevented all indirect chemical change in the packed fluid. The package should be of such materials that their constituents do not get transmitted into packed fluid.

#### 3.2 Flexible Packaging

These packaging materials can be derived into three main categories based on consumer usage. That is Single-use packaging materials, Multiple-use packaging materials and Materials for bulk supply.

From these categories the main targeted category for this research work is Single-use packaging which also classified as Flexible packaging. They are used to package product once and are discarded after removing the product.

Flexible plastic material can be made into pouch packs (Figure. 3.1(a)) or deliver to fluid product manufacturer in reels (Figure. 3.1(b)) to use in form-fill-and-seal (FFS) packing machines. So that flexible packing material should have the property of heat sealing. Normally flexible packages can be mono layer or double or triple or quadruple layer laminates depend on the fluid product to be packed (Figure. 3.2).

The advantages offered by the flexible packages are as follows;

- o Good barrier properties
- o Sometimes visibility of the contents
- o Light weight thus reducing the cost of transportation
- o Single-service thus eliminating the need for return, washing and sanitation
- o Easy to carry
- o Economical
- o Can be made more attractive

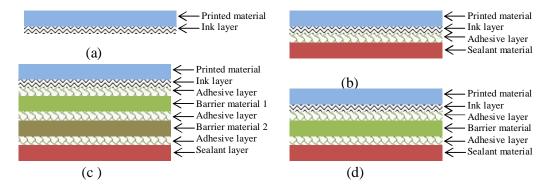
Figure 3.1(a) Flexible packaging pouch packs



Figure 3.1(b) Flexible packaging products in reel form



Figure 3.2 (a) Mono layer (b) Double layer (c) Quadruple layer (d) Triple layer



#### 3.3 Material Used in Flexible Packaging

The most commonly used material in flexible packaging is Polyethylene. There are several types of Polyethylene available and are characterized by their density. These are Low Density Polyethylene (LDPE), Linear Low Density Polyethylene (LLDPE), Medium Density Polyethylene (MDPE) and High Density Polyethylene (HDPE). Further more common materials used for flexible packaging are Polyethylene terephthalate (Polyester - PET), Metalized Polyester (METPET), Bi-axially Oriented Polypropylene (BOPP), Cast Polypropylene (CPP), Aluminium foil (ALU) and Nylon.

#### 3.4 Properties of Packaging Materials

Laminate packages require the mechanical properties such as tensile strength, stiffness, coefficient of friction, elongation and formability, etc. Barrier properties require from the laminated packaging materials are oxygen barrier, essential oil barrier, water vapour barrier and light barrier.

#### Polyethylene terepthalate

Polyesters are made by condensation–polymerization of carbonyl groups forming carbon–oxygen–carbon links. Probably the most important polyester use in food packaging is Polyethylene Terephthalate (PET) produced by reacting ethylene glycol with terephthalic acid (TPA). Commonly, a dimethyl ester of TPA is usually used to ensure a more controllable reaction. Linear-saturated PET is hard, semicrystalline, and transparent. It is stable over a wide temperature range (–60 to 220 °C), with a

high crystalline melting temperature ( $T_{\rm m}$ ) of 267 °C and  $T_{\rm g}$  ranging from 67 to 80 °C. PET has excellent tensile strength, chemical resistance, and is light weight. In its glassy state, PET is stiff, ductile, and tough, making it a polymer of choice for rigid packaging. Its elastic properties can be further modified through molding and extrusion.

PET is highly impermeable to aromas and gases, but has little resistance to water vapor. PET films can reduce the WVTR and oxygen permeability by the order of 40 and 300, respectively, compared to some other low-barrier polymers. PET is used to make bottles, films, and ovenable trays for frozen foods. PET can also be used as a component of foil trays, since it can withstand high electromagnetic fields during microwave (MW) heating. Two-layer polymeric trays comprised of crystalline polyethylene terephthalate (CPET) to provide rigidness and amorphous polyethylene terephthalate (APET) for low-temperature impact strength are popular for refrigerated food storage.

Polyethylene terepthalate (PET) has a density of 1.40 g/cm<sup>3</sup> and a melting point of 260°C. PET films are almost exclusively used in the bi-oriented, heat stabilized form. PET posses outstanding tear resistance, high transparency and gloss as well as remarkable resistance to scratching and abrasion. In addition its excellent printability combined with an advantageous machinability and dimensional stability makes it one of the converters preferred materials. PET can be used a temperature range of -50°C to 150°C. it exhibits good barrier properties with respect to water vapour, gases and fats.

#### **Bi-axially Oriented Polypropylene**

Bi-axially Oriented Polypropylene (BOPP) has a density of 0.9 g/cm<sup>3</sup> which is the lowest density of the common packaging materials. Its melting point lies with 169<sup>0</sup>C high enough to allow for sterilization applications. BOPP prominent qualities are: excellent moisture barrier, toughness, clarity and low cost. Low tear resistance make it suitable for easy open applications. Some of the disadvantages are low gas and aroma barrier and poor sealability.

#### **Aluminium foils**

Aluminium foil (ALU) is essentially impermeable to gasses and water vapour above a thickness of approx. 20 microns. For thinner gauges one can detect a small but non-zero permeability due to pinholes.

#### **Polyamides (Nylon)**

Polyamides are formed by condensation of amine monomers and carboxylic acid. As a result, polymers with amide linkages with high-mechanical strength and barrier properties have been developed. Several polyamides are used in food-packaging applications, including nylon 6, 10, and 11, as well as nylon 6, 6, and nylon 6, 10. In the United States, packaging films are usually produced from nylon 6, while European films are usually produced from nylon 11 due to the lower raw material costs in these markets until recently (Robertson 2006). Nylon 6 has better high temperature, grease and oil properties than nylon 11. Nylons have a  $T_{\rm g}$  below room temperature, but very high  $T_{\rm m}$  values (nylon 6 = 215 °C; nylon 6, 6 = 264 °C; nylon 6, 10 = 215 °C; nylon 11 = 185 °C). Due to the presence of the polar amine group in the polymeric structure, PAs are highly permeable to water vapor, and also absorb water. Absorbed water provides plasticizing effects to the material, increasing impact strength but reducing tensile properties. Their oxygen and other gas barrier properties are excellent compared to other films, but only when the films are dry.

Biaxially oriented PAs (BOPA) have received a great deal of attention for laminate films due to higher flex-crack resistance, mechanical strength and barrier properties than that of other PAs. Combining BOPA with PE improves water vapor barrier properties. Applications of PAs in food packaging include vacuum packaging of cheese, bacon, fresh and processed meats, and frozen foods.

#### **Polyethylene**

Polyethylene (PE) is the largest volume single polymer used in food packaging. The way in which the gaseous monomer ethylene is polymerized determines to large extend the density and the properties of the final product. The crystallinity of LDPE usually varies between 50-70 % and its density between 0.915-0.935 g/cm<sup>3</sup>. The

softening point of LDPE is just below 100<sup>o</sup>C thus precluding its utilization in sterilization applications. LDPE is tough, with good tensile strength, impact and tear resistance and posses an exceptional barrier to water. The permeability for oxygen and other gases is however very high. It has very good chemical resistance to acids, alkalis but is sensitive to hydrocarbons, oils and greases. Oils and many other organic compounds including many aromas are absorbed by LDPE leading to swelling of the polymer. The most outstanding property of LDPE is its ability to fusion welded to itself to yield good, tough, liquid tight seals.

LLDPEs can be produced in wide range of densities ranging from 0.90-0.935 g/cm<sup>3</sup>. A mayor feature of LLDPE is that its molecular weight distribution is narrower than that of LDPE leading to an improved chemical resistance, tear resistance and impact strength, higher surface gloss and higher melting point.

Some properties and functions of materials used in flexible packaging are briefly mentioned in Table 3.1.

Table 3.1 Properties and functions of flexible packaging materials

Material	Properties / Functions
Paper	Stiffness and Printability
ALU foil	Barrier properties ans Aesthetic Appeal
Cellophane	Clarity and Printability
LDPE	Heat sealability and Barrier properties
EVA	Strength and Heat sealability
HDPE	Stiffness and Low WVTR
ВОРР	Clarity and Barrier properties
PET	Impact and Barrier properties
PVC	Economy and Versatility in uses
Nylon	Strength, Grease and Oil resistance
PVDC	Grease resistance and Barrier properties

#### 3.5 Manufacture of Flexible Packaging

#### 3.5.1 Polyethylene blown film extrusion

Extruders consist of barrel, screw, drive mechanisms and control. The solid polymer is fed into the extruder as pellets and then melted and mixed by being passed through a screw. The shear friction of the pellets between each other and to the machine causes heating of the resins which finally leads to melting. The extruder is heated additionally in various zones to be able to control the temperature distribution, but this heating itself does not affect the melting of the solid resins. The resins melt temperature depends mainly on the rotation speed of the screw, the demanded output and the pressure of the polymer melt at the outlet filter. Blown film extrusion can also be used with polymer blends, the extruder then requires different feeder stations. Because food packaging can require a wide range of properties, two or more polymers are often coextruded through a single die to form a multilayer film structure. The resulting thin film has either the form of a sheet or a tube, the later often referred to as a bubble. In either configuration as the film comes out of the circular die, it is cooled and then rolled up on a core.

#### Blow-Up-Ratio

To produce the best physical properties in an extruded film the proper balance of film orientation in the machine direction (MD) and transverse direction (TD) of a film must be achieved. This relationship is achieved by adjusting the Blow-up-ratio (BUR) of the film. The BUR is the ratio of bubble diameter to the die diameter. It indicates the amount of stretching the polymer is undergoing during the shaping of the film.

$$BUR = (0.637 \text{ x Lay-Flat width}) / Die diameter$$

For PE standard BURs are between 2:1 and 3.5:1. Bubble stability problems are typically observed if the BUR is too high. Excessively high MD/TD oriented film is produced when the BUR is too low.

#### 3.5.2 Printing

Printing is one important step in producing a package material. The choice of the printing technique and inks will to a large extend influence on the visual and sensory quality of the final material. Two different methods are commonly employed in flexible packaging.

Rotogravure printing technique is commonly used in packaging. Printing can be performed on foil, film and paper/carton. Rotogravure machines usually range is between 6 to 12 colours. Printing speed is 100-300 m/min. In rotogravure an engraved cylinder is rotating partially within the printing ink, where its indentions are filled with the respective ink. Excess ink is removed by a doctor blade. The ink from the indentions is then transferred onto the substrate, which is pressed to the rotogravure cylinder by a second press cylinder. The quality of the print is very high.

Flexo printing is the second standard printing method in packaging. Printing plates are mounted to a cylinder, which then transfers the ink from a cylinder dipping into the ink reservoir to the substrate with nearly no pressure applied.

#### 3.5.3 Lamination

A lamination is created when two or more individual films are bonded together with special adhesives and run through rolling, heated cylinders to produce a composite film structures. Solvent-based lamination adhesives are very often two component Polyurethane-isocyanate systems with Ethyl acetate (EA) as the principal solvent. Curing occurs after lamination during the following few days. Lamination is preferred when a specific film composition cannot be effectively run on coextrusion systems due to equipment limitations and also when the high temperatures required in coextrusion would be harmful to films. Lamination is also recommended when it is desirable to produce a composite film with properties superior to those afforded by a single film layer of the same gauge.

#### 3.6 Fluid Filling and Sealing

Packing machines can be derived into two main categories. That is Horizontal-Form-Fill-Seal (HFFS) machine (Figure. 3.3) and Vertical-Form-Fill-Seal (VFFS) machine (Figure. 3.4). Packing machine which used for fluid/liquid products is VFFS machine.

Figure 3.3 Horizontal-Form-Fill-Seal machine



Figure 3.4 Vertical-Form-Fill-Seal machine



The term form-fill-seal means producing a bag/pouch from a flexible packaging material, inserting a measured amount of product and closing the bag top. The configuration of a VFFS machine is shown in Figure. 3.5. Coming from the roll and passing through a series of rollers to a bag-forming collar, the film is wrapped around

a metallic tube that gives it a cylindrical form. The two edges of the film are sealed as they pass along the metal tube. A first transverse seal forms the bottom of the bag that is then able to fill through the hollow tube. The packaging film then advances a predetermined distance that equals the desired bag length. The cylindrical film gets another transverse seal to close the filled bag. As the product comes in direct contact with the bottom seal when it is still hot, the hot-tack capability of the film is extremely important. The hot-tack force of the seal determines the quality of the closed bag and the machine speed.

Figure 3.5 Configuration of a VFFS machine

Piece #	DESCRIPTION
1	Filling tube
2	Forming shoulder
3	Packaging structure
4	Vertical seal tool
5	Transfer belts
6&7	Horizontal seal tool

#### 3.6.1 Sealing

Sealing or closure process for packaging is a key process. It ensures the protective function of packaging and assures the packaged goods a long life. In this context, efficient and reliable sealing processes are essential for the production of sustainable packages.

The formation of packages involves different sealing technologies, including heat sealing, hot air welding, ultrasonic welding, and impulse sealing.

Heat sealing is melting the plastic on either side of the bag opening, fusing the two films together. In hot air welding a metal wire, heated to red heat, is used to form a bead seal while cutting the film at the same time.

With Ultrasonic sealing, the heat required for melting is only generated inside the thermoplastic sealing layer [1].

Impulse sealing is the process of joining thermoplastic sheets by pressing them between elements equipped to provide a pulse of intense thermal energy to the sealing area for a very short time, followed immediately by cooling.

#### 3.7 Heat Sealing

Heat sealing is the most widely used technique in Form-Fill-Seal (FFS) packaging. As we discuss earlier typically thermoplastic material can be thermally sealed to itself or another polymeric material. Specially, heat and pressure are applied to the thermoplastic material for a period of time, referred to as the 'dwell time', to melt the polymers. The liquefied polymer molecules at the joint are thus allowed to intersperse, forming a strong bond once the polymeric materials cool and resolidify [2].

The sealing process involves melting, interdiffusion and crystallization of macromolecules at the interface of two materials to be sealed. In the case of semicrystalline polymers, the sealing temperature must be near the melting temperature of the polymer to allow macromolecule mobility at the interface. The sealing strength, toughness, failure mode and appearance of these seals after cooling to room temperature are important seal variables.

Among the different properties required in a material to be applied in flexible packaging, sealing performance is one of the most important, being essential for the integrity of the package. LLDPE is used mainly in film application due to its toughness, flexibility and relative transparency. LLDPE with good sealing performance is expected to present low sealing temperature, broad sealing window and high packaging performance, contributing to cost reduction by speeding up the automatic packaging processes [3].

#### 3.8 Factors Affecting on Heat Sealing

Dwell time allows for the polymer chains to reach molten or partially molten stage to entangle and create a hermetic seal. If dwell time is too short for the polymer chains to reach molten or partial molten stage the corners and the T-joint(Figure 3.6) will have a weak seal and are more likely to show leaks during hermeticity testing. The T-joint refers to the point on the seal where the fin seal meets the end seal. The pressure applied to seal both sides of the film together will remain the same throughout the study. Pressure is needed to seal two film surfaces together but increasing the pressure has no effect on seal strength [4,5].

Corner T-Joint Corner

Figure 3.6 Most important points in sealing area

The seal jaw temperature is a primary factor for seal properties but the interface temperature is the actual temperature of the sealed surface during the sealing process. Interfaced temperatures are important to reaching desirable sealing properties. Interfacial temperature is a lower than the platen temperature. In addition the effect of dwell time has less effect on interface temperature as the sealing temperature increases. Moreover sealing temperature has more effect on seal strength than dwell time [4].

Furthermore seal jaw styles can differ between machines and different sealing technologies. Matthews et. al [6], studied seal strength and effect of crimp angle and pitch of the seal jaw for heat sealing processes. The study compared Cellulose (38 $\mu$ m) and Polylactic Acid (PLA) (35 $\mu$ m) to Oriented Polypropylene (OPP) (25 $\mu$ m, 35 $\mu$ m and 50 $\mu$ m) and found that crimp angle is a secondary factor to seal strength. The crimp styles with more than 80° angles provide greater seal strength for films

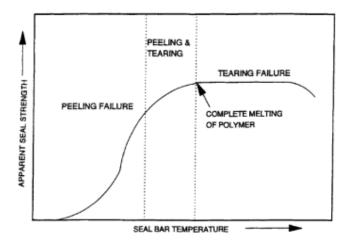
outside 25-45  $\mu$ m. This study did not record the interface temperature or crimp angles of the seal jaws.

Although sealing temperature is one of the two primary factors to reaching hermeticity. It is important to consider the peel force required to open the package. The temperature and dwell time combination may provide the strongest seal strength but it may make it impossible for the consumer to open the package. Companies can increase sealing temperatures with shorter dwell times to expedite the filling process. However the change in temperature and dwell time to reach the desired interface temperature more quickly can change the seal properties [7].

#### 3.9 Seal Strength

Testing the seal strength determines the amount of force or stress on the seal with respect to the elongation or strain to reach the material or peel failure. Testing the seal strength of a flexible package determines the type of seal failure for the given sealing conditions.

Figure. 3.7 Relationship between seal bar temperature and apparent seal strength for semicrystalline polymer films [4]



The Figure 3.7 shows that if the seal bar temperature is above the melting point  $T_m$  of the sealant then the seal strength test will show a tearing mode failure. On the other hand the seal strength test will show a peel failure if the seal bar temperature is below the  $T_m$  of the sealant. However if the seal bar temperature is within close range

of the  $T_m$  but below the melting point the seal strength test will more likely result in a peeling and tearing mode failure [4].

There are several types of results from a seal strength test: peel failure, tear failure, peel and tear failure and elongation failure. A seal will result in a tear failure, which shows that the strength of the seal is stronger than the strength of the film [8]. In addition there is also delamination failure mode that can occur in combination with the other failure types. Delamination occurs when one of the layers separates from the film during seal strength while either the outer layer or sealant layer remains attached during tensile testing.

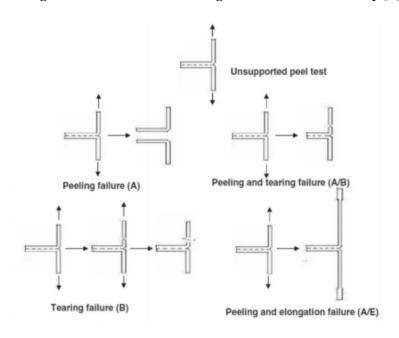


Figure 3.8 Previous seal strength results found in study [7]

A sealing temperature of a few degrees before the melting temperature  $T_m$ , seal strength will significantly increase and result in a peel, delaminating a tear mode or combination of failure modes. If the sealing temperature is more than a few degrees below the melting point than a peel failure will occur and the result will be a lower strength than the other failures.

#### 3.10 Liquid Contaminants in Seal Area

The liquid contaminant will act as a heat sink by absorbing the thermal energy that passes from the seal jaws through both plies of film. The thermal diffusivity of vegetable oil is  $0.09 \times 10^{-4} \text{ m}^2/\text{s}$  at  $20^{0}\text{C}$  and it is lower than thermal diffusivity of water which value as  $1.4 \times 10^{-4} \text{ m}^2/\text{s}$  at  $20^{0}\text{C}$  [8].

Different oil-based and salt-based liquid contaminants have different surface tension with the film which refers to the contact area between the contaminant and film. The contact of the area of the liquid contaminant is due to the surface tension between the liquid and film. Young determined the equation for the relationship between liquid, solid and vapour between a liquid droplet and a solid surface:

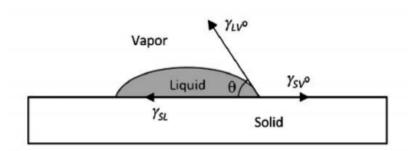
$$\gamma_{SV}^0 - \gamma_{SL} = \gamma_{LV}^{0} \cos\theta$$

Where  ${\gamma_{\rm SV}}^0-\text{Surface}$  tension of the solid and vapour boundary

 $\gamma_{\text{SL}}$  -Surface tension of the solid and liquid boundary

 $\gamma_{\rm LV^0}$  – Surface tension of the liquid and vapour boundary [8,9]

Figure 3.9 Liquid contact angle with solid surface



The contact angle indicates the amount of contamination that comes in direct contact with the film over an area of the film. However the movement of the seal jaws will cause the contaminant to display over the seal area. Furthermore the surface tension and contact angle influence the displacement of the contaminant that occurs during

the sealing. Both contaminants apply the same volume of contamination but the contaminant to film contact area will be different [8].

In addition to contact angle the liquid's density is an important property for determining liquid displacement during the heat sealing process. The density varies slightly for different vegetable oils [10]. Density for vegetable oil decreases by 0.00064 g/cm<sup>3</sup> for every 1°C increase in temperature [11]. Water has a density of 0.988 g/cm<sup>3</sup> at 21°C which is greater than vegetable oil and will displace more compared to vegetable oil.

Different densities mean the contaminants will displace differently during the sealing process. Once the two seal jaws bring the two film surfaces together then the liquid contaminant with high density should be expected to spread over a greater area. And liquid contaminant with greater contact angle expected to come in less contact with film due to the surface tension.

#### 3.11 Sealant Layer and Characteristics

The sealant layer is the inner most layer of the flexible packaging laminates that comes in direct contact with the opposing sealant layer during the sealing process. A high quality sealant has a broad sealing window and high hot tack strength [3]. A wide range of sealing temperature also allows for lower sealing temperatures without compromising the integrity of the seal. In addition the hot-tack strength refers to the film's ability to refrain from strains during its molten state [12]. A sealant with a low seal initiation temperature allows for lower process sealing temperatures and a lower sealing temperature will use less energy than a higher sealing temperature.

Package performance depends on the laminate structure chosen for a product. Laminate structure is more important to provide high quality seal for the packaged pouch, it must support the product and its expected shelf life from the time the product is packaged followed by transportation and lastly consumed by the consumer. A failed seal can shorten the shelf life of a product.

Most commonly using sealant layer material is PE. PE with good sealing performance is expected to present low sealing temperature, broad sealing window and high packaging performance.

#### Lamination sealant layer must:

- Be corona treated to the proper dyne level to accept either solvent or lamination adhesive.
- Heat seal through contaminants both liquid and dry in an often hostile environment. The sealant layer must prevent channel leaks when a fold over wrinkle occurs.
- Have a low Coefficient of Friction (COF) for machinability
- Be Food and Drug Administration (FDA) approved for prolonged and direct food contact in food grade packaging.

When designing multilayer films for flexible packaging applications the sealant resin choice is generally considered the most critical material decision. The sealant must be strong enough to maintain package integrity throughout the converting process and distribution chain yet to be soft and pliable enough to weld at low temperatures. A common seal problem in flexible packaging is channel leakers caused by product contamination in the seal area. Properly designed seal jaws can push low-viscosity liquids out of the seal area insuring good sealant contact. Particulate contaminants however are less mobile and may stay trapped within the seal area creating an incomplete seal around the particle. If the incomplete sealed area extends beyond the length of the seal a channel leak may result causing barrier and/or package integrity loss. Small leaks may not be caught at the packaging line and only become evident later in the distribution cycle.

#### **Hot-tack**

The sealant should have sufficient hot-tack strength to make and hold a secure seal before it cool. Hot-tack strength is important for VFFS packages. In a VFFS package the sealant's hot-tack strength must resist being pulled apart when the product drops into the package or air is introduced (Figure 3.10).

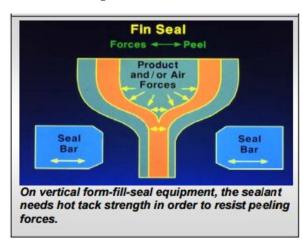


Figure 3.10 Fin seal

Each sealant has a particular temperature range within which it has sufficient hottack strength to avoid seal separation. The wider the hot-tack range, the greater the chance is for a leak proof seal.

#### Seal through/around contaminant

Sealing bar contamination is a major challenge to hermetic sealing of packages for both wet and dry products. In even the best controlled operations product spills onto the sealing bars. Sealants vary in their ability to cope with contamination of the sealing area. (Figure 3.11)

Figure 3.11 Hermetic seal vs. Leak path channels

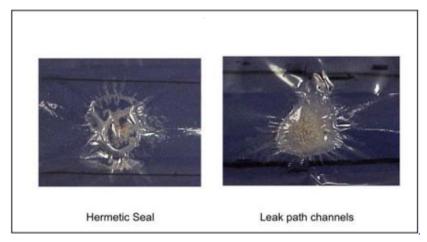
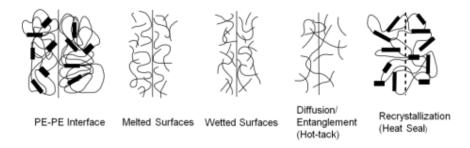


Figure 3.12 Hot-tack and seal strength at molecular level [13]



## 3.12 Lamination Factors Affecting on Heat Sealing

There are different methods of lamination and can be classified as;

Adhesive lamination – The process where a solution or emulsion of low molecular weight polymer adhesive material is coated on to the surface of one substrate before joining the second substrate.

Hot melt lamination – The molten wax is roller coated on to the substrate and the other substrate is laminated under nip pressure and passed over a chill roll immediately. Wax impregnation is carried out by entirely dipping the substrate in molten wax tray. Wax coated or laminated papers are widely used in biscuits and confectionery industry.

Extrusion coating lamination – The material in the barrel is heated, molten and extruded through a flat die in the form of thin layer. This thin layer is coated on to

the other substrate and passed immediately through the chilled roller and wound in a reel form.

Lamination process for different product is selected on the basis of characteristics of product packed inside. Solventbase lamination is suitable for all type of lamination. Solventless lamination is suitable for ready to eat products and for light weight products. Extrusion lamination is used for all bulk products packaging which require more strength in lamination [14].

The adhesive bond strength is affected by surface tension, solubility parameter and viscosity. These factors should be assessed in order to match up a particular adhesive to a set of substrates. In order to achieve good wettability the critical surface tension of the substrate should be greater than the surface tension of the adhesive [12]. The surface tension of the substrate can be increased by surface treatment techniques like corona discharge and plasma treatment or by applying a primer. The viscosity of the adhesive also plays a critical role in making good adhesive bonds. A low viscosity aids in spreading out the adhesive on the substrate evenly. Viscosity decreases with temperature and increases with the molecular weight [12]. Furthermore the solubility parameter of both the adherents and the adhesive should be similar for achieving desirable adhesive bond strength [12]. Cohesive bond strength relates to the strength within the adhesive and is attributed to the physical state and chemical nature of the adhesive material. Since performance of the adhesive depends on the adhesive bond strength an adhesive system should preferably have an adhesive bond strength that exceeds or equal to its cohesive bond strength. A higher molecular weight increases cohesive bond strength while decreasing the wettability. Therefore a balance between these factors is necessary in obtaining a desired level of overall bond strength [12].

In packaging, a pouch made of a laminated film can be no stronger than the seal that holds it together [15]. Therefore acceptable heat seal can be defined as a seal which has its strength greater than the strength of the packaging material. If this requirement is not fulfilled heat seal of a package will break open before the laminated film. This would be a waste of using strong laminated film which is usually more expensive as the package can easily failed at its seal when placed under

stress. Based on this an acceptable heat seal is one that failed in delamination or tearing mode failure during peeling test. Hence qualitative study on the failure modes of heat seals is equally important as quantitative measurement on the heat seal strength.

## Adhesive coating weight

Baral et. al [14], have analyzed that coating weight of adhesive used is more in solvent base lamination as compare to solventless lamination. This is because some amount of solvent in adhesive evaporates in the environment.

# **Curing time**

Also Baral et. al [14], have identified that curing time in solvent base lamination is more because there is solid content in solvent base adhesive when compared with solventless adhesive.

In this research study dry lamination machine is used. (Figure 3.13)

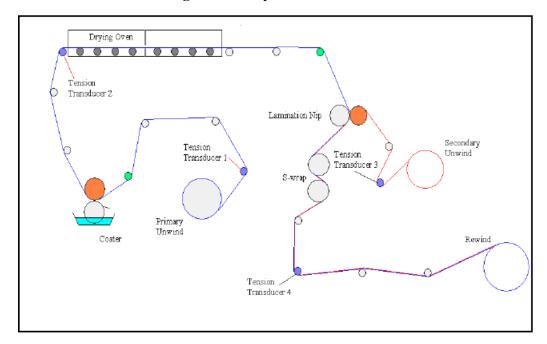


Figure 3.13 Dry bond lamination

#### 3.13 Testing Methods for Leaks with Seal Contamination

To test seal integrity there is either destructive or non-destructive methods. Some destructive methods include tensile testing used for seal strength, water vacuum chamber used for hermeticity tesing and dye penetration to show leaks in seal. Dye penetration is a visual inspection to check for leaks shown by a path through the seal from the inside to the outside of the bag. Matthews et. al [6], determined dye penetration is a poor method to test the presence of seal leaks because only samples exposed to excessive sealing conditions pass the test.

Non-destructive tests include ultrasonic pulse-echo or ultrasonic transmission testing for defects in the seal such as contamination. Transmission uses transmitting and receiving transducers on opposite sides of the seal. A contaminant in the seal will decrease the amplitude of the ultrasonic beam passing through the seal [16]. On the other hand pulse-echo used a reflective pulse to test for cracks, folds, voids, shrinks, porosity and flaking in metals [16]. Prior to Ozguler's [16] study on ultrasonic pulse-echo technique for flexible packaging it was assumed that the technology was insensitive to test seal integrity for flexible films [16]. Ultrasonic pulse-echo used backscattered amplitude integral (BAI) which is an acoustic compared to optical to record the reflective sound waves to detect seal defects. Furthermore BAI measures sound waves at 17.3 MHz and can detect any defects whether it is water or an bubbles as long as the test is done within 10 µm range of the film [16].

#### 3.14 Literature Review Summary

Flexible packaging industry is a growing and successful market mainly for the food industry. Developments in film and polymerization process technologies have made film packaging more desirable and applicable to many food industries. Food safety and consumer confidence in quality is very critical in today's packaging market. The failure of packages in most cases due to seal failures or leaks. Therefore improvement in seal integrity is desired. The seal integrity is a critical expectation from a sealant material. The selection of the sealant material can define the performance of the film on packaging equipments. Hot-tack and heat seal strength of a sealant is important. Sealing temperature and dwell time are the two primary

sealing factors to produce a quality seal. As mentioned previously seal jaw pressure has little effect on the quality of the seal. This study is for design of PE based multilayer extrusion blown film for manufacture of leak free packaging.

## **CHAPTER 04**

## **EXPERIMENTAL**

#### 4.1 Materials

Six samples were used in this study, which are commercial laminated films of various constructions, thickness and sealant material use in flexible packaging application. Details on laminated film structures are as shown in Table 4.1.

Table 4.1 Details on laminated film structures used in this study

Sample Name	Material Structure	Product to be packed	Total thickness (µm)
T-	12μm CT PET / 7μm ALU / 60μm	Tomato Sauce	81
normal	LLDPE(With normal material blend)		
T-	12μm CT PET / 7μm ALU / 60μm	Tomato Sauce	81
special	LLDPE(With special material blend)		
O-	15μm Nylon / 60μm LLDPE(With	Spicy oil	76
normal	normal material blend)		
O-	15μm Nylon / 60μm LLDPE(With	Spicy oil	76
special	special material blend)		
P-	12μm CT PET / 15μm Nylon / 60μm	Water based	89
normal	LLDPE(With normal material blend)	perfume	
P-	12μm CT PET / 15μm Nylon / 60μm	Water based	89
special	LLDPE(With special material blend)	perfume	

## Chemically treated Polyester (CT PET)

Thickness of 12 micrometers (12  $\mu$ m) CT PET from SRF Ltd. India was used for lamination structures as the outer layer of the laminate structure (For Sample T-normal, T-special, P-normal and P-special).

## Aluminium foil

Thickness of 7 micrometers (7  $\mu$ m) ALU foil from Shanghai Guanchang China was used as the barrier material or middle layer of the laminate structure (For Sample T-normal and T-special).

## Nylon

Thickness of 15 micrometers (15  $\mu$ m) Nylon film from PT Emblem India was used as the outer layer and middle layer of laminate structures (For Sample O-normal, O-special, T-normal and T-special).

## Polyethylene films

PE films were produced using blends of different resins such as Equate 7087, Lotrene 274, Lotrene 5026, Affinity 1881G, Elite 5401G and Polymer Processing Agent (PPA).

Equate 7087 is a linear low-density polyethylene (LLDPE) resin for tubular blown film extrusion. Films made from this material have good toughness, high tensile strength and outstanding puncture resistance. This resin contains high level of slip and antiblocking agent. (Figure 4.1)(Table 4.2)



Figure 4.1 Equate 7087 resin pallets

**Table 4.2 Main properties of Equate 7087** 

Property	Value	Test Method
Density	0.918 g/cm <sup>3</sup>	ASTM D1505
Melt Flow Index (MFI)	1.0 g/10 min	ASTM D1238
Melting Temperature	124 <sup>0</sup> C	ASTM E-794

Lotrene 274 is a low density polyethylene (LDPE). It contains both slip and antiblock agents. (Figure 4.2)(Table 4.3)

Figure 4.2 Lotrene 274 resin pallets



**Table 4.3 Main properties of Lotrene 274** 

Property	Value	Test Method
Density	0.923 g/cm <sup>3</sup>	ASTM D1505
Melt Flow Index (MFI)	2.4 g/10 min	ASTM D1238
Melting Temperature	145°C	ASTM E-794

Lotrene 5026 is a low density polyethylene (LDPE) resin contain additive that gives good slip properties. (Figure 4.3)(Table 4.4)

Figure 4.3 Lotrene 5026 resin pallets



**Table 4.4 Main properties of Lotrene 5026** 

Property	Value	Test Method
Density	0.920 g/cm <sup>3</sup>	ASTM D1505
Melt Flow Index (MFI)	0.6 g/10 min	ASTM D1238
Melting Temperature	108°C	ASTM E-794

AFFINITY 1881G is a Polyolefin Plastomer (POP) which includes slip and antiblock additives. AFFINITY polyolefin plastomer is an ethylene alpha-olefin resin designed for use in a variety of demanding applications including high-speed, form-fill seal products, display films and fresh produce bags. AFFINITY polymer offers excellent low temperature seal initiation and ultimate hot tack strength in addition to excellent abuse resistance, outstanding optics and high oxygen transmission rates. AFFINITY polymer has excellent compatibility with other polyolefins, allowing efficient blending and coextrusion. AFFINITY polymer complies with FDA regulation which allows for its use in direct food contact applications. (Figure 4.4)(Table 4.5)

Figure 4.4 Affinity 1881G resin pallets



**Table 4.5 Main properties of Affinity 1881** 

Property	Value	Test Method
Density	0.904 g/cm <sup>3</sup>	ASTM D792
Melt Flow Index (MFI)	1.0 g/10 min	ASTM D1238
Melting Temperature	100°C	ASTM E-794

Elite 5401G resin is a copolymer. It offers a unique combination of low seal initiation, moderate stiffness and low blocking for excellent performance on automated packaging equipment. This resin contains high level of slip and antiblocking agent. (Figure 4.5) (Table 4.6)

Figure 4.5 Elite 5401G resin pallets

**Table 4.6 Main properties of Elite 5401** 

Property	Value	Test Method
Density	0.918 g/cm <sup>3</sup>	ASTM D792
Melt Flow Index (MFI)	1.0 g/10 min	ASTM D1238
Melting Temperature	123°C	Internal method

PPA is a Polymer Processing Agent which used to ease the production process and increase productivity.

In dry lamination solvent based two component polyurethane Adcote 545-80 adhesive was used. And two components were diluted in Ethyl Acetate solvent.

# 4.2 Equipments

- LLDPE materials were produced from 3-layer blown film extrusion machine
- Samples were laminated in a dry lamination machine

- Differential Scanning Calorimeter (DSC) was used to determine the melting point  $T_m$  of the sealant materials (DSC Q20 V24.4 Build 116).
- Total thickness of sample materials were measured using a digital thickness meter (Model 49-70) which could measure the thickness range of 0.001 mm to 1 mm (1  $\mu$ m to 1000  $\mu$ m).
- Hot-tack tester was used to test both hot-tack strength and heat seal strength (HTT-L1 i-Thermotek 2500 Hot-tack tester).
- Manual heat sealer machine was used to seal the materials for heat seal strength test (Direct heat pedal sealer model DPS-300)
- Vacuum leakage tester was used to test leak in sachet samples (Model VLTC 2511)
- In production line VFFS machines were used for trials (For Tomato sauce Model YX SP60, For Spicy oil Model DXD 50Y, For Water based perfume Model DS 200Y)
- Weighing scale was used to measure the weight of packed sachets. (Model AY220)

#### 4.3 Procedure

## 4.3.1 Preparation of materials

All samples are manufactured using dry lamination process. In this technique a liquid adhesive (urethanes) is applied to one substrate. It is then dried with hot air. The dried surface is then adhered to a second substrate using heat and pressure. Lamination bond strength is achieved to 6.8 – 8.4 (N/25 mm) by setting adhesive viscosity to 21 seconds. In Sample T-normal,2,5 and 6, second lamination is done after curing for 12 hours of first lamination. All samples were kept in a hot cupboard of temperature 45°C for 12 hours and 6 hours in room temperature for curing.

Sealant material LLDPE is produced through 3-layer blown film extrusion machine and online corona treatment was applied in order to achieve a desired treatment level of 38 dyne.

LLDPE is formulated in different blends. LLDPE materials blend which use for Sample T-normal, O-normal and P-normal is mentioned in Table 4.7.

Table 4.7 Resin blend of LLDPE used for Sample T-normal, O-normal and P-normal

Material	Inner Layer	Middle Layer	Outer Layer
Equate 7087	80 %	80 %	80 %
Lotrene 274	20 %	20 %	20 %
PPA	100 g	100 g	100 g

When blowing the LLDPE with the blend mentioned in Table 4.7 machine parameters were set as in Table 4.8.

Table 4.8 Machine parameters while producing Sample T-normal, O-normal and P-normal sealant material

Temperature profile ( <sup>0</sup> C)				
Barrel No.	Inner Layer	Middle Layer	Outer Layer	
1	145	145	145	
2	150	150	150	
3	155	155	155	
4	165	165	165	
5	175	175	175	
Die Temperature ( <sup>0</sup> C)		1	185	
Screw RPM			78	
Line Speed			18	

LLDPE material blend used for Sample T-special, O-special and P-special are mentioned in Table 4.9.

Table 4.9 Resin blend of LLDPE used for Sample T-special, O-special and P-special

Material	Inner Layer	Middle Layer	Outer Layer
Equate 7087	-	-	80 %
Lotrene 5026	20 %	20 %	20 %
Affinity 1881G	50 %	50 %	-
Elite 5401G	30 %	30 %	-
PPA	100 g	100 g	100 g

When blowing the LLDPE with the blend mentioned in Table 4.9 machine parameters were set as in Table 4.10.

Table 4.10 Machine parameters while producing Sample T-special, O-special and P-special, sealant material

Temperature profile ( ${}^{0}$ C)				
Barrel No.	Inner Layer	Middle Layer	Outer Layer	
1	145	145	145	
2	150	150	150	
3	155	155	155	
4	165	165	165	
5	175	175	175	
Die Temperature ( <sup>0</sup> C)		:	185	
Screw RPM			64	
Line Speed			20	

Before producing LLDPE using the material blend which mentioned in Table 4.9 several blends were tried out (See Appendix – J). But from those material blends it was difficult to maintain bubble stability and other material properties such as film thickness, film width.

#### **4.3.2** Tests

In this study Differential Scanning Calorimeter (DSC) was used to determine the melting point  $T_m$  of the sealant materials. The melting point of a polymer is generally taken as the point at which the maximum energy is absorbed. In DSC experiment this corresponds to the peak maximum of the melting endotherm [17].

However the sealing temperature was selected based on total thickness of the laminate structure, material composition of the material structure and finally on the speed of VFFS packing machine. Since customer is highly concerning on their productivity need to run the filling machine with minimum dwell time. Hence to achieve the desire level of quality sealing temperature should be increased. Before trial in VFFS packing machine most feasible sealing temperature and dwell time was tested in hot-tack tester machine to achieve the desired hot-tack strength.

Total thickness of samples are measured from a digital thickness meter (Model 49-70). Referring to the standard ASTM F1921 for Standard test methods for hot seal temperature range and dwell times were decided for the testing of this study.

All VFFS packing machines for Tomato sauce, spicy oil and water base perfume had fixed parameters for heat seal pressure, dwell time and sealing area in production line. Maximum possible operating temperature for tomato sauce packing machine, spicy oil packing machine and water base perfume packing machine was 137 °C, 150 °C and 150 °C respectively. And customer of tomato sauce and spicy oil requires hottack strength higher than 2 N while customer of water based perfume requires hottack strength higher than 1.5 N.

Under the above conditions dwell time should be reduced as much as possible. As well energy consumption should be taken into consideration.

Therefore tested sealing temperatures and dwell times for the samples are mentioned in Table 4.11.

Table 4.11 Tested sealing temperatures and dwell times for hot-tack strength

Sample No. Seal Temperature (°C) T-normal 119 122	
122	
122	
125	
128	0.7
131	
134	
137	
119	
122	
125	
128	0.9
131	
134	
137	
119	
122	
125	
128	1.1
131	_
134	
137	
T-special 119	
122	
125	
128	0.7
131	
134	
137	
119	
122	
125	
128	0.9
131	
134	7
137	7
119	
122	
125	1.1
128	]

	101	
	131	-
	134	_
	137	
O-normal	132	_
	135	
	138	
	141	0.7
	144	_
	147	4
	150	
	132	4
	135	-
	138	
	141	0.9
	144	_
	147	_
	150	
	132	
	135	
	138	_
	141	1.1
	144	_
	147	_
	150	
O-special	132	_
	135	4
	138	
	141	0.7
	144	_
	147	_
	150	
	132	_
	135	
	138	
	141	0.9
	144	
	147	
	150	
	132	_
	135	_
	138	_
	141	1.1
	144	
	147	_
	150	

	100	T
P-normal	132	-
	135	-
	138	
	141	0.7
	144	=
	147	_
	150	
	132	_
	135	_
	138	
	141	0.9
	144	
	147	
	150	
	132	
	135	-
	138	-
	141	1.1
	144	1
	147	-
	150	<u>-</u>
P-special	132	
i special	135	<u>-</u>
	138	_
	141	0.7
	144	-
	147	1
	150	-
	132	
	135	
		-
	138	0.0
	141	0.9
	144	-
	147	-
	150	
	132	-
	135	-
	138	=
	141	1.1
	144	
	147	
	150	

Based on above parameters mentioned in Table 4.11 Hot-tack strength was measured and most feasible dwell time and temperature range was selected. Those selected parameters were then applied to test heat seal strength. By comparing both results most suitable parameters were chosen to meet customer's requirements for final trials in VFFS packing machine.

Hot-tack strength refers to the peel strength of the sealing area when it is not completed cooled down. For hot-tack strength tester machine air pressure was set to 6 bar and sealing pressure was set to 3 bar. Material strip of 350 mm length and 25 mm width was used for testing (Figure 4.6).



Figure 4.6 Sample strip used for hot-tack strength test

Formula for hot-tack strength is as follows.

Hot-tack strength (force on width per mm),  $B = F \over b$ 

Where B: hot-tack strength (N/25 mm)

F: force (N)

b: width (mm)

After completing and observing results of hot-tack strength, parameters were selected as in Table 4.12 in order to conduct the heat seal strength test.

Table 4.12 Tested parameters for heat seal strength

Sample No.	Seal Temperature (°C)
T-normal	130
	134
	137
T-special	122
	131
	137
O-normal	142
	147
	150
O-special	135
	144
	150
P-normal	143
	147
	150
P-special	133
	141
	150

Heat seal strength refers to the peel strength when seal area is completely cooled down. Manual heat sealing was done using a pedal heat sealer and sealed sample strip was allowed to condition at room temperature for at least 24 hours to achieve chemical stabilization. Aging of heat seal was necessary as the strength of seal may change in time, which may due to the memory of polymer or thermophysical properties of polymer as the heat seal samples undergo melting and cooling processes [18]. In practice packaging materials are aged through storage before being used by the customer. Therefore heat seals made in this study were aged at least 24

hours before tested. After 24 hours heat sealed strip was clamped to the heat seal strength tester machine. Width and length of strip was 25 mm and 350 mm respectively. Speed was set to 300 mm/min in order to conduct the heat seal strength test.

Heat seal strength (force on width per mm),  $\sigma = \underline{F}_{b}$ 

Where  $\sigma$ : heat seal strength (N/25 mm)

F: force (N)

b: width (mm)

To trial the materials in VFFS packing machine parameters were selected observing the results of heat seal strength test which achieved the highest heat seal strength. Parameters which selected for trials in VFFS packing machines are mentioned in Table 4.13.

Table 4.13 Parameters used in production line

Sample No.	Seal Temperature ( <sup>0</sup> C)	Dwell time (s)	Packed product
T-normal	134	0.9	Tomato Sauce
T-special	131	0.9	Tomato Sauce
O-normal	147	0.9	Spicy oil
O-special	144	0.9	Spicy oil
P-normal	147	0.9	Water based perfume
P-special	141	0.9	Water based perfume

After packing the product in VFFS packing machine, leak tests were done for packed sachets in vacuum chamber that places sachets into a contained tub of water. The vacuum pressure causes the bag to expand and allows bubbles to form at the leaking

points at the seal. The bubbles indicate a failed seal and no bubbles indicate a pass seal.

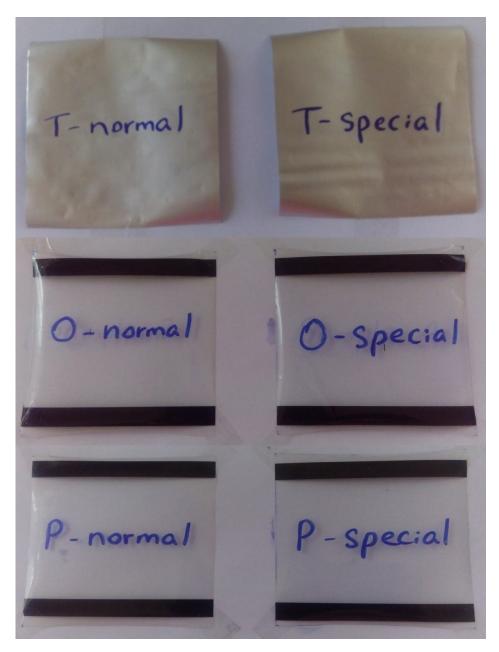
Also 10 packed sachets from each sample set were store in usual storage conditions in order to monitor the weight loss of sachets for 1 month. Weight were measured once a week and visually checked for leaks through seals.

# CHAPTER 05 RESULTS AND DISCUSSION

# 5.1 Samples

Following samples are used for this study (Figure 5.1) (As mentioned in Table 4.1)

Figure 5.1 Samples used in this study



# 5.2 DSC Experiment

In DSC sealant materials were tested to identify the melting temperature,  $T_m$  of the sealant material. Results of DSC test are mentioned in Table 5.1.

Table 5.1 Results of DSC experiment (See Appendix - J)

Sealant Material	$T_m(^0\mathbf{C})$
LLDPE with blend mentioned in Table 4.10	122.78
LLDPE with blend mentioned in Table 4.12	121.92

Based on the above melting point results of sealant materials and to meet the customer's requirements for filling efficiency in production line various temperatures and dwell times were selected in order to find out the optimum sealing conditions.

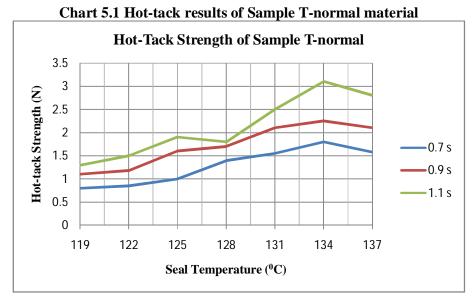
## 5.3 Hot-tack strength

# 5.3.1 Results of tomato sauce packing material structure

Results of the hot-tack strength for Sample T-normal material are mentioned in Table 5.2. And graphical presentation of results is included in Chart 5.1.

Table 5.2 Results of hot-tack strength for Sample T-normal material

Hot-tack Strength (N)			
<b>T</b> 400	Dwell time (s)		
Temperature $({}^{\theta}C)$	0.7	0.9	1.1
119	0.80	1.10	1.30
122	0.85	1.18	1.50
125	1.00	1.60	1.90
128	1.40	1.70	1.80
131	1.55	2.10	2.50
134	1.80	2.25	3.10
137	1.58	2.10	2.80



Results of the hot-tack strength for Sample T-special material are mentioned in Table 5.3. And graphical presentation of results is included in Chart 5.2.

Table 5.3 Results of hot-tack strength for Sample T-special material

	Hot-tack Strength (N)			
Temperature ( <sup>0</sup> C)	Dwell time (s)			
	0.7	0.9	1.1	
119	0.60	2.00	2.05	
122	2.10	3.10	4.70	
125	4.10	5.10	5.80	
128	5.00	5.78	6.10	
131	6.14	7.00	7.80	
134	4.90	6.00	6.20	
137	4.15	5.17	5.75	

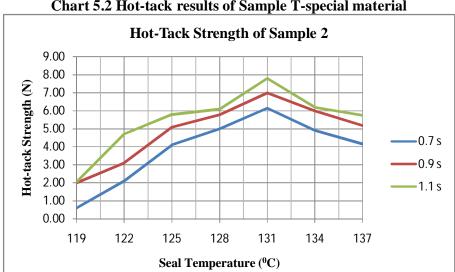


Chart 5.2 Hot-tack results of Sample T-special material

Today form-fill-seal machine has been widely applied in food, cosmetic and other fields. In the filling production line flexible package making and product filling are carried out in the intervening time. The filling method is basically to fill the product into the package from a certain height which would cause strong impact to the bottom of the package. If the bottom of package cannot withstand the impact it would break, so there is a package breakage. Package breakage will contaminate the environment as well the filling efficiency would be influenced. In actual production the interval between the heat seal process and product filling is quite short. So it is impossible for the sealing area cools down completely. Therefore heat seal strength is not suitable for evaluation of the heat seal property of the materials in production line. Hence before optimizing heat seal strength the most suitable method is optimizing hot-tack strength. Based on the results of hot-tack strength same parameters of production line could be used to optimize heat seal strength. Through hot-tack test the operator could decide the optimum heat seal parameters for the packaging materials which have been considered as an effective way to shorten the packaging cycling process. To be concluded proper hot-tack strength of the packaging material could guarantee the normal running of the filling production line. Hot-tack strength would directly influence the filling efficiency and the package breakage rate. Heat seal strength is significant to the evaluation of the performance

of the packaging material in storage in order to avoid the leak problems which may occur in transportation, storage and in display showcases in supermarkets.

It could be seen from the above test curves there is an optimum sealing temperature at which the hot-tack strength is highest. When the temperature exceeds the optimum value the hot-tack strength begins to decrease. As shown in Chart 5.1 and Chart 5.2 the optimum sealing temperature for Sample T-normal and Sample T-special is 134  $^{0}$ C and 131  $^{0}$ C respectively. Based on data for Sample T-normal when the dwell time is 0.7 s the hot-tack strength is lower than 2 N which does not meet the customer's requirement. When the dwell time is 0.9 s sealing temperature at a range between 130  $^{0}$ C and 137  $^{0}$ C the material could just reach the value required by the customer. While when the dwell time is 1.1 s and the sealing temperature lies in between ~128.5  $^{0}$ C and 137  $^{0}$ C the hot-tack strength of the material could well satisfy the requirement of customer. Thus in the case of Sample T-normal dwell time of more than 0.9 s and sealing temperature of 129  $^{0}$ C ~ 137  $^{0}$ C can satisfy the customer's requirements.

For Sample T-special when dwell time is no less than 0.7 s and the sealing temperature is in the range  $122~^{0}$ C  $\sim 137~^{0}$ C it can satisfy customer's need for hottack strength. With the dwell time is increased the sealing temperature could be decreased greatly. When dwell time reaches 1.1 s the sealing temperature in the range of  $119~^{0}$ C  $\sim 137~^{0}$ C could meet the customer's requirement.

Based on the above analysis on test data dwell time of 0.9 s selected for Sample T-normal with sealing temperature in between 129  $^{0}$ C ~ 137  $^{0}$ C in order to conduct heat seal strength test. For Sample T-special dwell time was selected to 0.9 s with sealing temperature in the range of 122  $^{0}$ C ~ 137  $^{0}$ C.

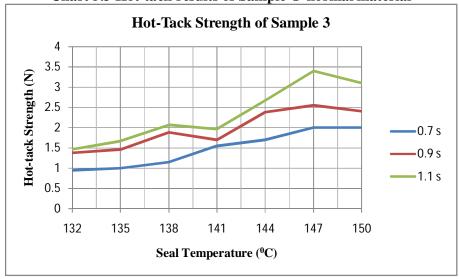
## 5.3.2 Results of spicy oil packing material structure

Results of the hot-tack strength for Sample O-normal material are mentioned in Table 5.4. And graphical presentation of results is included in Chart 5.3.

Table 5.4 Results of hot-tack strength for Sample O-normal material

Hot-tack Strength (N)				
- 0 c		Dwell time (s)		
Temperature $({}^{0}C)$	0.7	0.9	1.1	
132	0.95	1.38	1.47	
135	1.00	1.46	1.67	
138	1.15	1.88	2.07	
141	1.55	1.70	1.97	
144	1.70	2.38	2.67	
147	2.00	2.55	3.4	
150	2.00	2.40	3.10	

Chart 5.3 Hot-tack results of Sample O-normal material

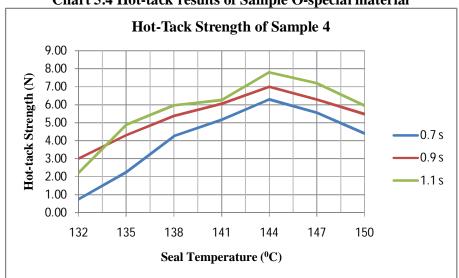


Results of the hot-tack strength for Sample O-special are mentioned in Table 5.5. And graphical presentation of results is included in Chart 5.4.

Table 5.5 Results of hot-tack strength for Sample O-special material

Hot-tack Strength (N)			
7 00	Dwell time (s)		
Temperature $({}^{0}C)$	0.7	0.9	1.1
132	0.75	3.00	2.22
135	2.25	4.30	4.87
138	4.25	5.38	5.97
141	5.15	6.06	6.27
144	6.29	7.00	7.80
147	5.55	6.28	7.20
150	4.40	5.47	5.95

Chart 5.4 Hot-tack results of Sample O-special material



As discussed in Sample T-normal and 2 hot-tack strength result data analysis in these curves also there is an optimum sealing temperature for both samples. For Sample O-normal and Sample O-special optimum sealing temperature is 147 °C and 144 °C respectively. Customer requirement on hot-tack strength on spicy oil packaging material is also 2 N. For Sample O-normal, when dwell time is 0.7 s after the optimum sealing temperature it has reached the required hot-tack strength. When

dwell time is 0.9 s desired strength have achieved in sealing temperature range of  $\sim 141.8 \, ^{0}\text{C} - 150 \, ^{0}\text{C}$ . When dwell time is 1.1 s and sealing temperature in the range of  $141 \, ^{0}\text{C} \sim 150 \, ^{0}\text{C}$  could satisfy the customer's requirement.

For Sample O-special when dwell time is 0.7 s and when sealing temperature is in the range of 134  $^{0}$ C  $\sim 150$   $^{0}$ C, the desired hot-tack strength have achieved. When dwell is 0.9 s and 1.1 s in whole sealing temperature range required hot-tack strength could be achieved.

Based on the above observations for Sample O-normal dwell time of 0.9 s was selected to seal the material in order to conduct the heat seal strength test. Also the sealing temperature range was  $142~^{0}\text{C} \sim 150~^{0}\text{C}$ . For Sample O-special the selected values for dwell time and sealing temperature range is 0.9 s and  $132~^{0}\text{C} \sim 150~^{0}\text{C}$  respectively.

# 5.3.3 Results of water based perfume packing material structure

Results of the hot-tack strength for Sample P-normal material are mentioned in Table 5.6. And graphical presentation of results is included in Chart 5.5.

Table 5.6 Results of hot-tack strength for Sample P-normal material

Hot-tack Strength (N)			
Temperature (°C)	Dwell time (s)		
	0.7	0.9	1.1
132	0.60	0.60	1.00
135	0.65	0.68	1.2
138	0.80	1.10	1.60
141	1.20	1.20	1.50
144	1.55	1.60	2.20
147	1.60	1.75	2.80
150	1.38	1.60	2.60

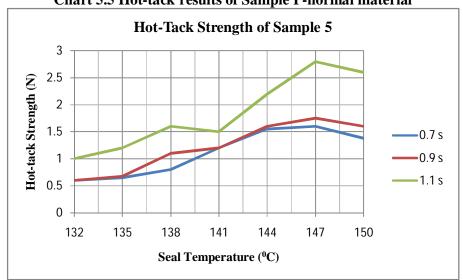


Chart 5.5 Hot-tack results of Sample P-normal material

Results of the hot-tack strength for Sample P-special material are mentioned in Table 5.7. And graphical presentation of results is included in Chart 5.6.

Table 5.7 Results of hot-tack strength for Sample P-special material

	Hot-tack Strength (N)			
Temperature (°C)		Dwell time (s)		
	0.7	0.9	1.1	
132	0.60	1.00	1.99	
135	1.60	2.60	3.00	
138	2.70	3.00	3.50	
141	4.05	4.30	4.50	
144	4.00	4.13	4.26	
147	3.98	4.00	4.10	
150	3.70	3.24	4.01	

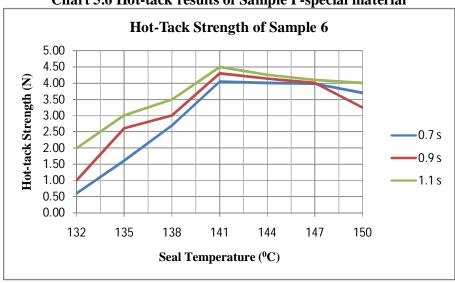


Chart 5.6 Hot-tack results of Sample P-special material

Optimum sealing temperature for Sample P-normal was 147 °C and for Sample Pspecial it was 141 °C. For the water based perfume customer's requirement was to achieve hot-tack strength for 1.5 N. This requirement could be achieved for Sample P-normal when dwell time is 0.7 s and sealing temperature range of 143.5  $^{\circ}$ C ~ 150  $^{0}\mathrm{C}.$  When dwell time is 0.9 s and sealing temperature range of 143  $^{0}\mathrm{C}$  ~ 150  $^{0}\mathrm{C}$ desired results were achieved. When dwell time is 1.1 s the sealing temperature range was  $136.3 \, {}^{0}\text{C} \sim 150 \, {}^{0}\text{C}$ .

For Sample P-special required hot-tack strength was achieved, when dwell time is 0.7 s and sealing temperature range of  $134.7 \, ^{\circ}\text{C} \sim 150 \, ^{\circ}\text{C}$ , when dwell time is  $0.9 \, \text{s}$ and sealing temperature range of 132.8  $^{\circ}$ C ~ 150  $^{\circ}$ C and when dwell time is 1.1 s for whole sealing temperature range.

Hence the selected dwell time was 0.9 s for both Sample P-normal and 6. And sealing temperature range was selected as  $143~^{\circ}\text{C} \sim 150~^{\circ}\text{C}$  for Sample P-normal and 133  $^{0}$ C ~ 150  $^{0}$ C for Sample P-special.

# 5.4 Heat Seal Strength

## 5.4.1 Results of tomato sauce packing material structure

Heat seal strength result data for Sample T-normal material are mentioned in Table 5.8.

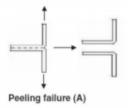
Table 5.8 Results of heat seal strength for Sample T-normal material

Seal Temperature ( <sup>0</sup> C) at 0.9 s Dwell Time	Heat Seal Strength (N)
130	1.92
134	2.68
137	3.51

As discussed in Chapter 3 there are several types of results from a seal strength test: peel failure (A), tear failure (B), peel and tear failure (A/B) and peel and elongation failure (A/E). A seal will result in a tear failure, which shows that the strength of the seal is stronger than the strength of the film. In addition there is also delamination failure mode that can occur in combination with the other failure types. Delamination occurs when one of the layers separates from the film during seal strength while either the outer layer or sealant layer remains attached during tensile testing (Figure 2.8).

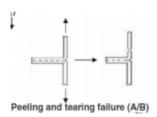
At sealing temperature 130  $^{0}$ C test sample strip was found to fail in peeling mode failure (Figure 5.2). Under this failure mode disentangles and extricates chain ends from the opposite surface occurred in which the heat seal bond was peeled apart. This happened when the strength of the seal is lower than the strength of the laminate structure.

Figure 5.2 Peeling failure of heat seal



In other two tested sample strips at sealing temperature 134  $^{0}$ C and 137  $^{0}$ C showed peel and tear failure mode (Figure 5.3). Acceptable heat seal strength was achieved at this stage.

Figure 5.3 Peeling and tearing failure of heat seal



Hence when dwell time is 0.9 s both sealing temperatures 134  $^{0}$ C and 137  $^{0}$ C are acceptable to use in production line. However sealing temperature was selected as 134  $^{0}$ C with 0.9 s dwell time because energy consumption is more for higher temperatures.

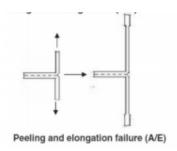
Heat seal strength result data for Sample T-special material are mentioned in Table 5.9.

Table 5.9 Results of heat seal strength for Sample T-special material

Seal Temperature ( <sup>0</sup> C) at 0.9 s Dwell Time	Heat Seal Strength (N)
122	0.83
131	2.80
137	4.01

For the Sample T-special materials peeling failure mode was appear when sealed at the temperature of 122  $^{0}$ C. When sealed at 131  $^{0}$ C temperature material showed peel and tear failure mode while it showed peel and elongation failure mode (Figure 5.4) for sealing temperature of 137  $^{0}$ C. Therefore both 131  $^{0}$ C and 137  $^{0}$ C sealing temperatures are acceptable to use in production line. Since we need to consider on energy consumption 131  $^{0}$ C sealing temperature with 0.9 s dwell time was selected for final trials in production line.

Figure 5.4 Peeling and elongation failure of heat seal



# 5.4.2 Results of spicy oil packing material structure

Heat seal strength result data for Sample O-normal material are mentioned in Table 5.10.

Table 5.10 Results of heat seal strength for Sample O-normal material

Seal Temperature ( <sup>0</sup> C) at 0.9 s Dwell Time	Heat Seal Strength (N)
142	2.67
147	3.41
150	5.26

Heat seal strength result data for Sample O-special are mentioned in Table 5.11.

Table 5.11 Results of heat seal strength for Sample O-special material

Seal Temperature ( <sup>0</sup> C) at 0.9 s Dwell Time	Heat Seal Strength (N)
135	2.68
144	2.89
150	6.03

Both Sample O-normal and Sample O-special showed peel and tear mode failure for all sealing temperatures. Therefore in all sealing temperatures with 0.9 s dwell time could achieve an acceptable seal. Considering energy consumption for this sample set also lowest acceptable sealing temperature was selected in order to use in production line. Sealing temperature for Sample O-normal is 142 °C with 0.9 s dwell time and sealing temperature for Sample O-special is 135 °C with 0.9 s dwell time.

# 5.4.3 Results of water based perfume packing material structure

Heat seal strength result data for Sample P-normal material are mentioned in Table 5.12.

Table 5.12 Results of heat seal strength for Sample P-normal material

Seal Temperature ( <sup>0</sup> C) at 0.9 s Dwell Time	Heat Seal Strength (N)
143	2.59
147	3.12
150	4.35

Here again peel and tear failure mode appear for the samples sealed at 143  $^{0}$ C and 147  $^{0}$ C while peel and elongation failure mode appear for the sample sealed at 150  $^{0}$ C. Therefore all sealing temperatures at 0.9 s dwell time are given acceptable seals. Similarly considering energy consumption as above lowest sealing temperature 143  $^{0}$ C at 0.9 s dwell time was selected for trials in production line.

Heat seal strength result data for Sample P-special material are mentioned in Table 5.13.

Table 5.13 Results of heat seal strength for Sample P-special material

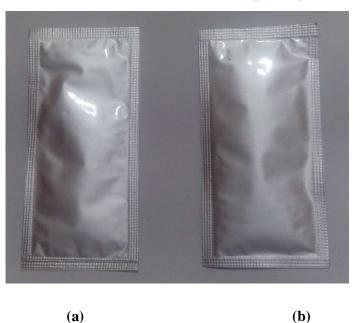
Seal Temperature ( <sup>0</sup> C) at 0.9 s Dwell Time	Heat Seal Strength (N)
133	1.98
141	2.66
150	7.12

For the above test at sealing temperature 133  $^{0}$ C, material was found to be failed in peeling failure mode. However tested samples at 141  $^{0}$ C and 150  $^{0}$ C sealing temperatures found to be accepted in heat seal strength. Therefore 141  $^{0}$ C sealing temperature was selected to use in production line with 0.9 s dwell time.

# 5.5 Trials in Production Line

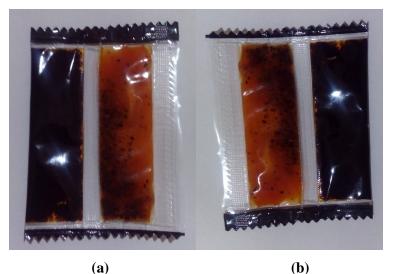
Packing materials for tomato sauce were trialed in a VFFS tomato sauce packing machine at a speed of 50 bags/min. For Sample T-normal, sealing temperature was 134  $^{0}$ C at a dwell time of 0.9 s. For Sample T-special, sealing temperature was 131  $^{0}$ C at dwell time of 0.9 s. From produced tomato sauce sachets 10 sample sachets were selected to conduct leakage test in vacuum chamber and 10 sample sachets were kept in storage conditions in order to check weight loss and to check for leakages.

Figure 5.5 (a) Packed tomato sauce sachet from Sample T-normal material (b) Packed tomato sauce sachet from Sample T-special material



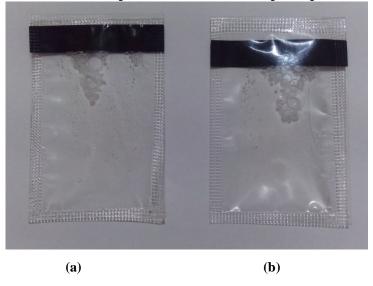
Packing materials for spicy oil were trialed in a VFFS spicy oil packing machine at a speed of 60 bags/min. For Sample O-normal, sealing temperature was 147  $^{0}$ C at a dwell time of 0.9 s. For Sample O-special, sealing temperature was 144  $^{0}$ C at dwell time of 0.9 s. From produced spicy oil sachets 10 sample sachets were selected to conduct leakage test in vacuum chamber and 10 sample sachets were kept in storage condition in order to check weight loss and to check for leakages.

Figure 5.6 (a) Packed spicy oil sachet from Sample O-normal material (b) Packed sachet from Sample O-special material



Packing materials for water based perfume were trialed in a VFFS water based perfume packing machine at a speed of 40 bags/min. For Sample P-normal, sealing temperature was 147  $^{0}$ C at a dwell time of 0.9 s. For Sample P-special, sealing temperature was 141  $^{0}$ C at dwell time of 0.9 s. From produced water based perfume sachets 10 sample sachets were selected to conduct leakage test in vacuum chamber and 10 sample sachets were kept in storage conditions in order to check weight loss and to check for leakages.

Figure 5.7 (a) Packed water based perfume sachet from Sample P-normal material (b) Packed water based perfume sachet from Sample P-special material



# 5.6 Leakage Test Results

# 5.6.1 Test results for tomato sauce packing material

Completely sealed sachet from each product was put inside a vacuum chamber in order to observe leakages through seals. If bubbles were generated while vacuum is increasing it was taken as fail and otherwise it was taken as pass.

However Ozgular et. al [16], have identified that contaminants fall only along the T-joint. Hence leaks through T-joint have identified as a poor seal due to contamination of product and it should consider as a fail product. Corners of the seal are not contaminated. However if leaks appear through corners it also should be taken as a fail product. But considering contamination it is not a true fail [16]. However leakage from a packed pouch is a negative impact to customer. Hence leakages cannot be accepted.

Results for tomato sauce sachets in Sample T-normal material and Sample T-special material are mentioned in Table 5.14.

Table 5.14 Leak test results for tomato sauce sachets

Sochat No.	Sample T-normal Material		Sample T-	special Material
Sachet No.	Result	Remarks	Result	Remarks
1	Pass	No bubble	Pass	No bubble
2	Pass	No bubble	Pass	No bubble
3	Fail	Bubble forms at sealing corners	Pass	No bubble
4	Fail	Bubble forms at sealing corners	Fail	Bubble forms at sealing corners
5	Fail	Bubble forms at sealing corners	Pass	No bubble
6	Pass	No bubble	Pass	No bubble
7	Pass	No bubble	Pass	No bubble
8	Fail	Bubble forms at sealing corners	Fail	Bubble forms at sealing corners
9	Pass	No bubble	Pass	No bubble
10	Pass	No bubble	Pass	No bubble

Summary results of above data for Sample T-normal material are as in Table 5.15.

Table 5.15 Summary results of leak test for Sample T-normal material

	Pass	Fail	Fail Type
Sample T-normal Material	6		None
		4	Corners
Total	6	4	

Based on the above results the fail percentage for the Sample T-normal material was 40% at the sealing temperature of 134  $^{0}$ C when dwell time 0.9 s.

Summary results of above data for Sample T-special material are as in Table 5.16.

Table 5.16 Summary results of leak test for Sample T-special material

	Pass	Fail	Fail Type
Sample T-special Material	8		None
		2	Corners
Total	8	2	

Based on the above results the fail percentage for the Sample T-special material was 20% at the sealing temperature of 131  $^{0}$ C when dwell time 0.9 s.

Though the sealing temperature is low for Sample T-special material it has showed a good result from leakage test when compared to the Sample T-normal material.

# 5.6.2 Test results for spicy oil packing material

Results for spicy oil sachets in Sample O-normal material and Sample O-special material are mentioned in Table 5.17.

Table 5.17 Leak test results for spicy oil sachets

Carlord No.	Sample O	normal Material	Sample O	-special Material
Sachet No.	Result	Remarks	Result	Remarks
1	Pass	No bubble	Pass	No bubble
2	Pass	No bubble	Pass	No bubble
3	Pass	No bubble	Fail	Bubble forms at sealing T-Joint
4	Fail	Bubble forms at sealing T-Joint	Fail	Bubble forms at sealing corners
5	Fail	Bubble forms at sealing T-Joint	Pass	No bubble
6	Fail	Bubble forms at sealing T-Joint	Pass	No bubble
7	Pass	No bubble	Pass	No bubble
8	Fail	Bubble forms at sealing T-Joint	Fail	Bubble forms at sealing T-Joint
9	Fail	Bubble forms at sealing corners	Pass	No bubble
10	Pass	No bubble	Fail	Bubble forms at sealing corners

Summary results of above data for Sample O-normal material are as in Table 5.18.

Table 5.18 Summary results of leak test for Sample O-normal material

	Pass	Fail	Fail Type
Sample O-normal Material	5		None
		1	Corners
		4	T-Joint
Total	5	5	

Based on the above results the fail percentage for the Sample O-normal material was 50% at the sealing temperature of 147  $^{0}$ C when dwell time 0.9 s.

Summary results of above data for Sample O-special material are as in Table 5.19.

Table 5.19 Summary results of leak test for Sample O-special material

	Pass	Fail	Fail Type
Sample O-special Material	6		None
		2	Corners
		2	T-Joint
Total	6	4	

Based on the above results the fail percentage for the Sample O-special material was 40% at the sealing temperature of  $144~^{0}$ C when dwell time 0.9~s.

Though the sealing temperature is low for Sample O-special material it has showed a good result from leakage test when compared to the Sample O-normal material.

# 5.6.3 Test results for water base packing material

Results for water based perfume sachets in Sample P-normal material and Sample P-special material are mentioned in Table 5.20.

Table 5.20 Leak test results for water based perfume sachets

Sachet No.	Sample P-	normal Material	Sample P	-special Material
Sachet No.	Result Remarks		Result	Remarks
1	Fail	Bubble forms at sealing corners	Fail	Bubble forms at sealing corners
2	Pass	No bubble	Fail	Bubble forms at sealing corners
3	Fail	Bubble forms at sealing corners	Fail	Bubble forms at sealing corners
4	Pass	No bubble	Pass	No bubble
5	Fail	Bubble forms at sealing corner	Pass	No bubble
6	Fail	Bubble forms at sealing corners	Fail	Bubble forms at sealing corners
7	Fail	Bubble forms at sealing corners	Fail	Bubble forms at sealing corners
8	Fail	Bubble forms at sealing corners	Fail	Bubble forms at sealing corners
9	Fail	Bubble forms at sealing corners	Pass	No bubble
10	Fail	Bubble forms at sealing corners	Pass	No bubble

Summary results of above data for Sample P-normal material are as in Table 5.21.

Table 5.21 Summary results of leak test for Sample P-normal material

	Pass	Fail	Fail Type
Sample P-normal Material	2		None
		8	Corners
Total	2	8	

Based on the above results the fail percentage for the Sample P-normal material was 80% at the sealing temperature of 147  $^{0}$ C when dwell time 0.9 s.

Summary results of above data for Sample P-special material are as in Table 5.22.

Table 5.22 Summary results of leak test for Sample P-special material

	Pass	Fail	Fail Type
Sample P-special Material	4		None
		6	Corners
Total	4	6	

Based on the above results the fail percentage for the Sample P-special material was 60% at the sealing temperature of 141  $^{0}$ C when dwell time 0.9 s.

Leak percentage is much higher for water based perfume sachet. Even though for the improved material structure it showed a comparatively high leak percentage. However leak percentage has decreased for Sample P-special material when compared to Sample P-normal material.

# 5.7 Storage Test Results

After the production line trials packed sachets were kept in normal storing conditions to monitor leakages and any loss of weights.

Results of tomato sauce sachets in Sample T-normal material is mentioned in Table 5.23.

Table 5.23 Weight readings of tomato sauce sachets in Sample T-normal material

Sachet No.	1st Week	2 <sup>nd</sup> Week	3 <sup>rd</sup> Week	4 <sup>th</sup> Week
1	14.589	14.587	14.588	14.588
2	14.750	14.750	14.749	14.749
3	15.002	15.002	15.001	15.000
4	14.992	14.992	14.992	14.991
5	15.000	15.000	15.000	15.000
6	14.925	14.925	14.923	14.924
7	14.900	14.900	14.900	14.900
8	14.872	14.872	14.871	14.872
9	14.880	14.880	14.880	14.880
10	14.765	14.764	14.765	14.765

Results of tomato sauce sachets in Sample T-special material is mentioned in Table 5.24.

Table 5.24 Weight readings of tomato sauce sachets in Sample T-special material

Sachet No.	1st Week	2 <sup>nd</sup> Week	3 <sup>rd</sup> Week	4 <sup>th</sup> Week
1	14.972	14.972	14.971	14.972
2	14.970	14.970	14.970	14.968
3	14.990	14.990	14.990	14.990
4	15.000	15.000	14.997	14.998
5	14.847	14.840	14.846	14.845
6	14.981	14.979	14.980	14.980
7	14.888	14.880	14.880	14.779
8	14.911	14.910	14.910	14.910
9	14.900	14.900	14.900	14.900
10	15.000	15.000	15.000	15.000

When observed the weight results for one month of tomato sauce sachets packed in Sample T-normal material and Sample T-special material, there was not major weight variation. But some leakages were observed in sachets. And those leakages are minor for Sample T-special material when compared with Sample T-normal material.

Results of spicy oil sachets in Sample O-normal material is mentioned in Table 5.25.

Table 5.25 Weight readings of spicy oil sachets in Sample O-normal material

Sachet No.	1st Week	2 <sup>nd</sup> Week	3 <sup>rd</sup> Week	4 <sup>th</sup> Week
1	5.55	5.54	5.54	5.53
2	5.62	5.61	5.62	5.61
3	5.51	5.50	5.49	5.49
4	5.49	5.49	5.47	5.46
5	5.60	5.57	5.57	5.56
6	5.50	5.50	5.50	5.48
7	5.53	5.52	5.53	5.53
8	5.53	5.54	5.53	5.52
9	5.54	5.53	5.53	5.53
10	5.58	5.58	5.57	5.57

Results of spicy oil sachets in Sample O-special material is mentioned in Table 5.26.

Table 5.26 Weight readings of spicy oil sachets in Sample O-special material

Sachet No.	1st Week	2 <sup>nd</sup> Week	3 <sup>rd</sup> Week	4 <sup>th</sup> Week
1	5.47	5.47	5.46	5.45
2	5.53	5.50	5.51	5.51
3	5.57	5.56	5.55	5.54
4	5.51	5.51	5.51	5.51
5	5.48	5.48	5.48	5.48
6	5.47	5.47	5.47	5.46
7	5.50	5.51	5.50	5.50
8	5.51	5.48	5.49	5.48
9	5.52	5.52	5.51	5.51
10	5.51	5.50	5.50	5.50

In spicy oil sachets also there were no major weight losses. But there were some leakages through T-joint. And leakages were higher for Sample O-normal material compared to Sample O-special material.

Results of water based perfume sachets in Sample P-normal material is mentioned in Table 5.27.

Table 5.27 Weight readings of water based perfume sachets in Sample P-normal material

Sachet No.	1 <sup>st</sup> Week	2 <sup>nd</sup> Week	3 <sup>rd</sup> Week	4 <sup>th</sup> Week
1	4.93	4.93	4.91	4.91
2	4.92	4.91	4.91	4.91
3	4.95	4.95	4.94	4.94
4	4.97	4.98	4.98	4.97
5	4.99	4.99	4.98	4.99
6	4.95	4.95	4.95	4.94
7	4.98	4.97	4.97	4.96
8	5.00	4.98	4.97	4.97
9	4.99	4.98	4.98	4.98
10	5.00	5.00	4.99	4.99

Results of water based perfume sachets in Sample P-special material are mentioned in Table 5.28.

Table 5.28 Weight readings of water based perfume sachets in Sample P-special material

Sachet No.	1 <sup>st</sup> Week	2 <sup>nd</sup> Week	3 <sup>rd</sup> Week	4 <sup>th</sup> Week
1	5.01	5.00	5.00	4.99
2	4.98	4.98	4.98	4.97
3	5.00	4.99	4.98	4.98
4	4.99	4.97	4.98	4.97
5	4.98	4.97	4.97	4.97
6	4.98	4.98	4.98	4.98
7	4.99	4.98	4.98	4.98
8	4.98	4.97	4.97	4.96
9	4.97	4.96	4.95	4.95
10	4.99	4.99	4.97	4.97

In water based perfume sachets, there were no major weight losses. And in both materials there were no visible leakages. In some sachets could observe leakages through seal corners. And the leakage amount was lesser for Sample P-special material structure when compared with Sample P-normal structure.

## CHAPTER 06

# **CONCLUSION**

The results of the study measure the effect of liquid contaminant at the seal area with a linear low density polyethylene sealant. It was noticed that the product contaminant left residuals of product onto the corrugated box during storage with current material structures of the products tomato sauce, spicy oil and water based perfume. Selected products have different laminated structures and it was observed that no effect on the laminated structure for forming a leak free sealing joint.

During production the contamination was a continuous stream from sachet to sachet and may have been the reason for the product found in the box. The effect of the storage temperature and time do not have a significant effect on the performance of the LLDPE sealant when liquid contamination present in the seal area. The results of hot-tack strength were used to determine the optimal sealing conditions and those sealing conditions were applied to test the performance of seal strength. The seal strength performance can be used to determine the integrity of the seal strength. The different sealing temperatures and dwell time were chosen based on acceptable range of sealing conditions. Based on the test results selected sealing conditions were tested in production line. It would be more desirable to have a lower sealing temperature and dwell time for faster production and lower production costs.

When LLDPE sealant was blown using LLDPE 80% and LDPE 20% in all 3-layers, the sealant material was not good enough to withstand liquid contaminants in seal area and tend to weaken seal. Hence the leakages were observed from the seal. Therefore newly blown sealant material was produced incorporating POP material which is named as Affinity 1881G from Dow Chemicals and metallocene material which is named as Elite 5401G from Dow Chemicals. POP resin and metallocene resins are developed for high speed machines which give good seal integrity. Hence LLDPE sealant material incorporating with POP resin and metallocene LLDPE was able to withstand seal through contamination of liquid products such as tomato sauce, spicy oil and water based perfume. And by using newly developed sealant LLDPE

for packing of tomato sauce, spicy oil and water based perfume leakage percentage could be reduced by 20% for tomato sauce and spicy oil, by 10% for water based perfume.

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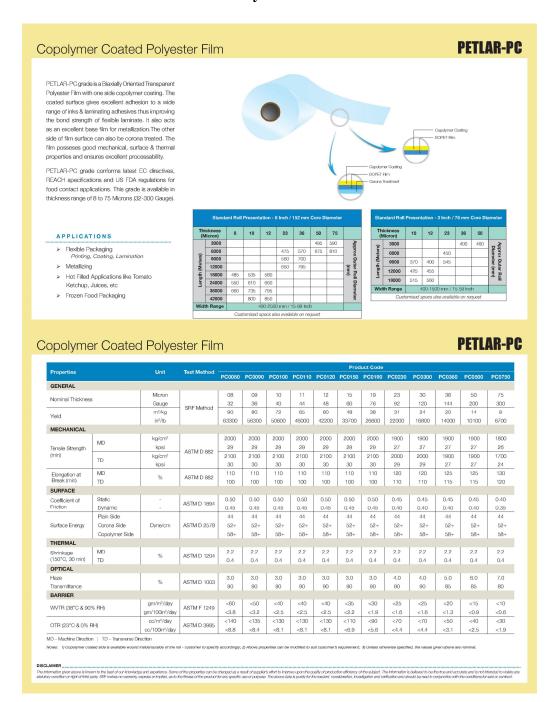
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## **APPENDICES**

# **APPENDIX - A: TDS of Chemically Treated PET**



# APPENDIX - B: TDS of Nylon

# 沧州东鸿包装材料有限公司



www.bopa-dhb.com E-MAIL: bopa.dhb@gmail.com

# TDS for Simultaneous Nylon Film

Typical Properties		Units	Test Method	Value
Thickness		micron		15 ± 3%
Yield		m²/kg		58.0 ± 3%
Tensile Strength	MD	Мра		210
Tensile Siterigiti	TD	IMPA	ASTM D882	230
Elongation	MD	%	ASTIVI DOOZ	115
Liongation	TD	70		95
Dimensional Stability	MD	%	ASTM D1204	1.5
(320°F/160°C - 5 min.)	TD	76	A31W D1204	0.5
Tear Strength		mN	ASTM D1004	105
Coefficient of Friction	Static		ASTM D1894	0.4
Coefficient of Friction	Dynamic		A31W D1094	0.38
Haze		%	ASTM D1003	2.0
Gloss (45°)		%	ASTM D523	105
Surface Tension	inside/outside	Dynes	ASTM D2578	56/50
Oxygen Transmission Rate		cm³/m²/day·0.1MPa	ASTM D3985	28-30
68℉ (20℃) / 0%RH		Cili /ili /day-o. IMPa	70 LINI D0900	20-30

DONGHONG films: complies with FDA regulation 21 CFR 177.1500 (b)(6.2)

Production technology: Three layer co-extrusion flat cast die process, then simultaneous tenter frame biaxially orientation.

# **APPENDIX - C: TDS of Equate 7087**

# **EQUATE PE EFDC-7087**

Linear Low Density Polyethylene **EQUATE Petrochemical Company KSCC** 



EFDC-7087 is a linear low-density polyethylene (LLDPE) resin for tubular blown film extrusion. Films made from EFDA-7087 have good toughness, high tensile strength and outstanding puncture resistance. EFDA-7087 contains high levels of slip and antiblocking agent. The product offers excellent draw down capability for thinner gauge film production.

EFDC-7087 is recommended for the manufacture of thin gauge liner films, garment bags and other industrial and consumer packaging applications

requiring toughness and puncti	ire resistance.		
eneral			
Material Status	<ul> <li>Commercial: Active</li> </ul>		
Availability	<ul> <li>Africa &amp; Middle East</li> </ul>	<ul> <li>Asia Pacific</li> </ul>	<ul> <li>Europe</li> </ul>
Additive	<ul> <li>Antiblock</li> </ul>	Slip	
Features	<ul><li>Antiblocking</li><li>Good Drawdown</li></ul>	<ul><li>Good Toughness</li><li>High Tensile Strength</li></ul>	<ul><li>Puncture Resistant</li><li>Slip</li></ul>
Uses	<ul><li>Film</li><li>Industrial Applications</li></ul>	<ul><li>Laundry Bags</li><li>Packaging</li></ul>	
Forms	<ul> <li>Pellets</li> </ul>		
Processing Method	Blow Molding     Blown Film	<ul> <li>Electrostatic Spray Coating</li> <li>Extrusion Blow Molding</li> </ul>	Film Extrusion

hysical	Nominal Value Unit	Test Method
Density	0.918 g/cm <sup>3</sup>	ASTM D1505
Melt Mass-Flow Rate (MFR) (190°C/2.16 kg)	1.0 g/10 min	ASTM D1238
ilms	Nominal Value Unit	Test Method
Film Thickness - Tested	25 μm	
Film Puncture Energy (25 µm)	65.0 J	Internal Method
Secant Modulus		ASTM D882
1% Secant, MD: 25 μm, Blown Film	193 MPa	
1% Secant, TD: 25 μm, Blown Film	221 MPa	
Tensile Strength		ASTM D882
MD: Break, 25 µm, Blown Film	34.0 MPa	
TD: Break, 25 µm, Blown Film	26.0 MPa	
Tensile Elongation		ASTM D882
MD: Break, 25 µm, Blown Film	800 %	
TD: Break, 25 µm, Blown Film	850 %	
Dart Drop Impact (Blown Film)	100 g	ASTM D1709A
Elmendorf Tear Strength <sup>2</sup>		ASTM D1922
MD: 25.0 µm	35.0 kN/m	
TD: 25.0 µm	135.0 kN/m	
nermal	Nominal Value Unit	Test Method
Melting Temperature	124 °C	Internal Method
ptical	Nominal Value Unit	Test Method
Gloss (45°, 25.0 µm, Blown Film)	50	ASTM D2457

xtrusion	Nominal Value Unit
Hopper Temperature	210 °C
Cylinder Zone 1 Temp.	180 to 210 °C
Cylinder Zone 2 Temp.	180 to 210 °C
Cylinder Zone 3 Temp.	180 to 210 °C
Cylinder Zone 4 Temp.	180 to 210 °C
Cylinder Zone 5 Temp.	180 to 210 °C
Adapter Temperature	210 °C
Melt Temperature	210 °C
Die Temperature	210 °C

14 %

# Die Gap: >1.8 mm

Haze (25.0 µm, Blown Film)

Notes

1 Typical properties: these are not to be construed as specifications.

Revision History

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Revision History

Document Created: Tuesday, March 27, 2012

The information presented on this datasheet was acquired by IDES from the producer of the material IDES makes substantial efforts to assure the accuracy of this data. However, IDES assures no responsibility for the data values and strongly encourages that upon that material supplier.

Last Updated: 6/24/2009

ASTM D1003

<sup>&</sup>lt;sup>2</sup> Blown Film

# APPENDIX - D: TDS of Lotrene 0274

Exclusive Agent for QATAR PETROCHEMICAL COMPANY LTD. (QAPCO)

### Lotrene ® FD0274 LOW DENSITY POLYETHYLENE

#### DESCRIPTION

Lotrene ® FD0274 is mainly recommended for the extrusion of thin film for light and medium duty applications. It contains both slip agent and anti blocking devise.

#### PROPERTIES:

The suitable molecular structure of Lotrene  $\circledast$  FD0274 makes it possible to produce very thin, clear and glossy films.

Lotrene ® FD0274 gives films of especially good dimensional stability with easy sealing no matter what type of machine is used.

POLYMER PROPERTIES	VALUE	UNIT	TEST METHOD
Melt flow index	2.4	g/10min	ASTM D – 1238
Density @ 23 'C	0.923	g/cm3	ASTM D - 1505
Crystalline Melting point	111	'C	ASTM E - 794
Vicat Softening Point	94	<b>'C</b>	ASTM D - 1525
POLYMER PROPERTIES	VALUE	UNIT	TEST METHOD
Tensile strength @ yield MD/TD	14/11	MPa	ASTM D - 882
Tensile strength @ Break MD/TD	22/21	MPa	ASTM D - 882
Elongation @ Break MD/TD	470/570	%	ASTM D - 882
Impact Strength, F50	110	$\mathbf{G}$	ASTM – 1709
Coefficient of Friction	0.10	-	ASTM D – 1894
Haze	6.5	%	ASTM D – 1003
Gloss ( @ 45 ' )	75	GU	ASTM D - 2457
Clarity	85	%	ASTM D – 1746

(The above properties are measured on a blown film of 50  $\mu,\,@$  2.50 BUR)

Note: the values given in this technical data sheet are the results of tests carried out in accordance with standard test procedures. They are given as indication to enable customers to make the best use of our products but must be considered as average values provided without implying any undertaking on our part.

#### PROCESSING

Loyrene® FD 0274 can be easily processed on all types of extruders to make blown or cast films.

The melt temperature is suggested to be in the range of 140-150 –  $^{\prime}$ C.

The best properties of the blown film are achieved at blow up ratios between 2 , 5:1 and 3 , 5:1 .

To avoid blocking and shrinkage on the reel, the temperature at the nip rolls and take – aff should be kept as close as possible to the ambient temperature.

The recommended thickness range is from 25 μm 100 μm.

#### APPLICATIONS:

- . Thin transparent film
- . Food packaging
- . High clarity film
- . Lamination film

#### SAFETY AND STORAGE:

Under normal conditions Loyrene® FD 0274 does not present a toxic hazard through skin contact or inhalation. During processing, contact with molten polymer and inhalation of volatilized fumes should be avoided.

 $Loyrene \& FD 0274 \ complies \ with food \ grade \ regulations \ FDA, 21 \ FR \ chapter \ 177-1520 \ Olefin \ polymers. \ Items \ made \ from \ this \ grade \ do \ not \ transmit \ tests \ Nor \ odor \ to \ the \ material \ in \ contact \ with$ 

Loyrene® FD 0274 is inflammable and combustible ( Category 3 ) according to ISO R 1210.

Loyrene® FD 0274 is supplied in plastic bags of 25 kg ( Net weight ) each. The bags are stscked and shrink wrapped on pallets of 1500 kg each ( Net weight ). The product is forwarded either by trucks or in 20- foot sea containers.

Loyrene® FD 0274 should not be stored for more than three months nor be exposed to direct sunlight and/or heating during storage since this may adversely affect the properties of the product.

# APPENDIX – E: TDS of Lotrene 5026

Exclusive Agent for QATAR PETROCHEMICAL COMPANY LTD. (QAPCO)

#### Lotrene ® FB5026 LOW DENSITY POLYETHYLENE

### DESCRIPTION

 $Lotrene \circledast F5026 is mainly recommended for heavy duty film applications with good optical properties. It contains additive that gives good slip properties.$ 

#### PROPERTIES

The suitable molecular structure of Lotrene  ${\rm \rlap{@}}$  F5026 makes it possible to produce very thin, clear and glossy films.

 $Lotrene \circledast F5026 \ produces \ films \ that \ are \ very \ strong, \ sufficiently \ rigid \ for \ automatic \ packaging \ machines, suitable for shrink \ wrapping \ and \ have \ good \ dimensional \ stability.$ 

POLYMER PROPERTIES	VALUE	UNIT	TEST METHOD
Melt Flow index	0.60	g/10 min	ASTM D-1238
Density @ 23 'C	0.920	g/cm3	ASTM D-1505
Crystalline Melting Point	108	'C	ASTM E-794
Vicat Softening Point	93	' C	ASTM D-1525
FILM PROPERTIES	VALUE	UNIT	TEST METHOD
Tensile Strength @Yield MD/TD	14/11	MPa	ASTM D-882
Tensile Strength @ Break MD/TD	24/24	Mpa	ASTM D-882
Elongation @ Break MD/TD	550/600	%	ASTM D-882
Impact Strength, F50	200	G	ASTM D-1709
Coefficient of Friction	0.20	_	ASTM D-1894
Haze	11	%	ASTM D-1003
Gloss (@45')	45	GU	ASTM D-2457
Clarity	45	%	ASTM D-1746

(The above properties are measured on a blown film of 50  $\mu,\,$  @ 205 BUR)

Note: The values given in this technical data sheet are the results of testes carried out in accordance with standard test procedures. They are given as indication to enable customers to makes the best use of our products but must be considered as average values provided without implying any undertaking on our part.

### **PROCESSING**

 $Lotrene \ {\rm \rlap{@}}\ F5026\ can\ be\ easily\ processed\ on\ all\ types\ of\ extruders\ designed\ for\ polyethylene.$ 

The melt temperature is suggested to be in the range of 160-180'C.

The best properties of the blown film are achieved at blow up ratios between 2.5:1 and 3.5:1.

To avoid blocking and shrinkage on the reel, the temperature at the nip rolls and take-off should be kept as close as possible to the ambient temperature.

The recommended thickness range is from 40  $\mu$ m 200  $\mu$ m.

### APPLICATION

- . Shopping bags
- . Medium duty carrier bags
- . Milk bags (pouches)
- . Industrial liners

#### SAFETY AND STORAGE

Under normal conditions Lotrene ® F5026 does not present a toxic hazard through skin contact or inhalation. During processing, contact with molten polymer and inhalation of volatilized fumes should be avoided.

Lotrene ® F5026 complies with food grade regulations FDA, 21 FR Chapter 177-1520 'Olefin Polymers'. Items made from this grade do not transmit tests Nor odor to the material in contact with.

Lotrene ® F5026 is inflammable and Combustible( category 3) according to ISO R 1210.

Lotrene ® F5026 is supplied in plastic bags of 25 kg ( net weight ) each. The bags are stacked and shrink wrapped on pallets of 1500 kg each (net weight) . The product is forwarded either by trucks or in 20- foot sea containers.

Lotrene ® F5026 should not be stored for more than three months nor be exposed to direct sunlight and/or heating during storage since this may adversely affect the properties of the product.

# APPENDIX - F: TDS of Affinity 1881G

1/25/2017

Dow AFFINITY™ PL 1881G Polyolefin Plastomer (POP)

### Dow AFFINITY™ PL 1881G Polyolefin Plastomer (POP)

Categories: Polymer; Film; Thermoplastic; Polyolefin

AFFINITY  $^{\text{TM}}$  PL 1881G Polyolefin Plastomer (POP) is produced via INSITE  $^{\text{TM}}$  Technologies. It is designed for use in a variety of packaging applications, including high-speed, form-fill-seal products. Material

Notes:

Information provided by Dow

No vendors are listed for this material. Please <u>click here</u> if you are a supplier and would like information on how to add your listing to this material. Vendors:

Physical Properties	Metric	English	Comments
Density	0.9035 g/cc	0.03264 lb/in <sup>3</sup>	ASTM D792
Thickness	50.8 microns	2.00 mil	
Melt Flow	1.0 g/10 min	1.0 g/10 min	ASTM D1238
Antiblock Level	2500 ppm	2500 ppm	
Slip Level	750 ppm	750 ppm	
Mechanical Properties	Metric	English	Comments
Film Tensile Strength at Yield, MD	8.07 MPa	1170 psi	ASTM D882
Film Tensile Strength at Yield, TD	7.17 MPa	1040 psi	ASTM D882
Film Elongation at Break, MD	585 %	585 %	ASTM D882
Film Elongation at Break, TD	630 %	630 %	ASTM D882
Secant Modulus, MD	0.0970 GPa	14.1 ksi	2% Secant; ASTM D882
Secant Modulus, TD	0.0970 GPa	14.1 ksi	2% Secant; ASTM D882
Impact	18.5	18.5	[lb <sub>f</sub> ]; Puncture Resistance Force; Dow Method
	265	265	[ft-lbf/in <sup>3</sup> ]; Puncture Resistance; Dow Method
Puncture Energy	8.09 J	5.97 ft-lb	Dow Method
Coefficient of Friction, Dynamic	0.15	0.15	film/film; ASTM D1894
Elmendorf Tear Strength MD	560 g	560 g	Modified rectangular test specimen; ASTM D1922
Elmendorf Tear Strength TD	730 g	730 g	Modified rectangular test specimen; ASTM D1922
Elmendorf Tear Strength, MD	11.0 g/micron	280 g/mil	ASTM D1922
Elmendorf Tear Strength, TD	14.4 g/micron	365 g/mil	ASTM D1922
Dart Drop Test	>= 830 g	>= 1.83 lb	Method B; ASTM D1709
Film Tensile Strength at Break, MD	45.4 MPa	6580 psi	ASTM D882
Film Tensile Strength at Break, TD	42.5 MPa	6170 psi	ASTM D882
Heat Seal Strength Initiation Temperature	85.0 °C	185 °F	2 lb/in heat seal strength; 0.5 sec dwell, 40 psi bar pressure, pull speed 10 (in./min.); Dow Method
Thermal Properties	Metric	English	Comments
Melting Point	100 °C	212 °F	Dow Method (DSC)
Vicat Softening Point	86.0 °C	187 °F	ASTM D1525
Optical Properties	Metric	English	Comments
Haze	3.2 %	3.2 %	ASTM D1003
Gloss	112 %	112 %	20°; ASTM D2457
Transmission, Visible	83 %	83 %	Clarity; ASTM D1746
Descriptive Properties			
Block Force g		70	ASTM D3354-89

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# APPENDIX - G: TDS of Elite 5401G

1/25/2017

Dow ELITE™ 5401G Enhanced Polyethylene Resin

#### Dow ELITE™ 5401G Enhanced Polyethylene Resin

Categories: Polymer; Film; Thermoplastic; Polyethylene (PE); LLDPE

Material Notes: ELITE™ 5401G is a copolymer produced via INSITE™ Technology from Dow Plastics. It offers a unique combination of low seal initiation, moderate stiffness and low blocking for excellent performance on automated packaging equipment. ELLITE™ 5401G resin complies with U.S. FDA regulation 21 CFR 177.1520 (c) 3.2a.

Information provided by Dow

Vendors:

No vendors are listed for this material. Please <u>click here</u> if you are a supplier and would like information on how to add your listing to this material.

Physical Properties	Metric	English	Comments
Density	0.9175 g/cc	0.03315 lb/in <sup>3</sup>	ASTM D792
Thickness	50.8 microns	2.00 mil	
Melt Flow	1.0 g/10 min	1.0 g/10 min	ASTM D1238
Antiblock Level	2500 ppm	2500 ppm	
Slip Level	1000 ppm	1000 ppm	
Mechanical Properties	Metric	English	Comments
Film Tensile Strength at Yield, MD	8.267 MPa	1199 psi	ASTM D882
Film Tensile Strength at Yield, TD	8.811 MPa	1278 psi	ASTM D882
Film Elongation at Break, MD	572 %	572 %	ASTM D882
Film Elongation at Break, TD	612 %	612 %	ASTM D882
Secant Modulus, MD	0.1809 GPa	26.23 ksi	2% Secant; ASTM D882
Secant Modulus, TD	0.2044 GPa	29.65 ksi	2% Secant; ASTM D882
Impact	18	18	[lbf]; Puncture Resistance Force; Dow Method
	168	168	[ft-lbf/in3]; Puncture Resistance; Dow Method
Puncture Energy	5.76 J	4.25 ft-lb	Dow Method
Elmendorf Tear Strength MD	780 g	780 g	ASTM D1922
Elmendorf Tear Strength TD	975 g	975 g	ASTM D1922
Elmendorf Tear Strength, MD	15.4 g/micron	390 g/mil	ASTM D1922
Elmendorf Tear Strength, TD	19.19 g/micron	487.5 g/mil	ASTM D1922
Dart Drop Test	>= 850 g	>= 1.87 lb	Method B; ASTM D1709
Film Tensile Strength at Break, MD	38.04 MPa	5517 psi	ASTM D882
Film Tensile Strength at Break, TD	36.54 MPa	5299 psi	ASTM D882
Heat Seal Strength Initiation Temperature	95.0 °C	203 °F	2 lb/in heat seal strength; 0.5 sec dwell, 40 psi bar pressure, pull speed 10 (in./min.); Dow Method

Thermal Properties	Metric	English	Comments
Melting Point	123 °C	253 °F	Dow Method (DSC)
Vicat Softening Point	100 °C	212 °F	ASTM D1525
Optical Properties	Metric	English	Comments
Haze	13 %	13 %	ASTM D1003
паге	10 /0	10 /0	ACTIVID 1000

Some of the values displayed above may have been converted from their original units and/or rounded in order to display the information in a consistent format. Users requiring more precise data for scientific or engineering calculations can click on the property value to see the original value as well as raw conversions to equivalent units. We advise that you only use the original value or one of its raw conversions in your calculations to minimize rounding error. We also ask that you refer to MatWeb's terms of use regarding this information. Click here to view all the property values for this datasheet as they were originally entered into MatWeb.

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# **APPENDIX - H: TDS of Dry Lamination Adhesive**

### ROHM!HAAS M

### ADCOTE™ 545-80 Laminating Adhesive

#### Description

ADCOTE 545-80 is a solvent based two component polyurethane adhesive with high solid characteristics, excellent heat and chemical resistance. This polyester adhesive in conjunction with various isocyanate terminated coreactants functions as a laminating adhesive for flexible packaging and industrial applications. ADCOTE 545-80 provides optical clarity and high bond strength.

ADCOTE 545-80 adhesive adheres to a wide variety of substrates including cellophane, treated polyolefins, polyester, polyamide, aluminum foil, paper, metalized and PVDC coated materials, and the treated side of heat-sealable coextruded films.

#### Typical Uses

ADCOTE 545-80 with Coreactant F is suitable for the lamination of transparent and aluminum-containing structures with and without sandwich printing. Principal uses are in flexible materials for food pouches, vacuum or gas flushed luncheon meats and cheese, coffee pouches, condiment packaging, cable wrap laminations, liquid packaging, hygroscopic powders, chemicals and cosmetics.

### Typical Properties\*

These properties are	e typical but do not constitute specific	cations.
	ADCOTE 545-80 (OH Component)	Coreactant F ** (NCO Component)
Solids	80%	75%
Viscosity	6500 cps @ 77°F (25°C)	1800 cps @ 77°F (25°C)
Weight/Gallon	9.1 lbs (1.10 g/cc)	9.9 lbs (1.19 g/cc)
Solvents	Methyl ethyl ketone	Ethyl acetate
Mix Ratio	100 parts by weight	14 parts by weight
Diluents	Methyl ethyl ketone, toluen	e, or urethane grade ethyl acetate
Shelf Life	360 days	180 days

<sup>\*</sup>These items are provided for general information only. They are approximate values and are not considered part of a product specification.

#### Safety, Handling and Storage

- Store in cool, dry, well-ventilated area away from heat and ignition sources. Keep container tightly closed.
- Hot organic chemical vapors can suddenly and without warning ignite when mixed with air. Ignition can
  occur at typical elevated temperature process conditions. Please evaluate such processes to assure safe
  handling conditions.
- Support and ground containers before opening, dispensing, mixing, pouring or emptying. Open with nonsparking tools. If container is warm, open bung slowly to release the internal pressure.
- Wash thoroughly after handling. Wash contaminated goggles, face shield, and gloves. Professionally launder contaminated clothing before re-use.
- ATTENTION! Container can be hazardous when empty. Follow label warnings even after container is empty.
  Do not use heat, sparks, open flames, torches or cigarettes on or near empty containers. Do not reuse
  empty container without professional cleaning.
- Refer to Material Safety Data Sheet for more information.

Alcohol and similar materials containing active hydrogen can react with the ADCOTE 545-80 and Coreactant F causing inadequate cure. ADCOTE 545-80 with Coreactant F could potentially interact with other constituents of the laminated structures. Retained solvents, printing inks, slip additives, film additives, antiblock agents, coatings, contaminated solvents as well as the packed product are some of the components that may cause property changes of the film and/or adhesive.

<sup>\*\*</sup>Other coreactants are available for special uses. Consult your Rohm and Haas sales representative for further information.

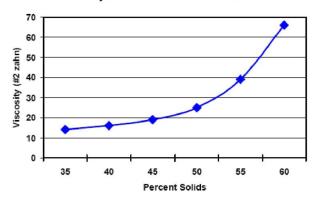
#### **Recommended Operating Conditions** Application Method Direct gravure 165 - 200 line (65 - 80 line per cm) Application Cylinder Application Solids 35 - 50% 8 hours approximately Pot Life Static 24 hours approximately Dry Adhesive Weight 1.1 - 2.5 lbs/ream (2 - 4.5 gsm) depending on structure 150 - 200°F (65 - 93°C) Drying Web Temperature Lamination Temperature 125 - 180°F (52 - 82°C) Cure Time 5 - 10 days at 70°F (21°C) Slitting/Rewinding Time 4 - 8 hours at 70°F (21°C) Cleaning Solvent Methyl ethyl ketone, ethyl acetate or other suitable organic solvents.

Initial bond immediately off the laminator is good. Within 48 hours the bond strength will be sufficient for further processing, such as slitting and rewinding. Maximum properties of heat and chemical resistance will develop within 5 - 10 days.

#### **Dilution Table**

Solids content (%)	ADCOTE 545-80 (parts)	Coreactant F (parts)	Methyl ethyl ketone or Ethyl acetate (parts)
60	100	14	37
55	100	14	50
50	100	14	67
45	100	14	87
40	100	14	112
35	100	14	145

### Viscosity of Adcote 545-80/Coreactant F



# **APPENDIX - I: TDS of Solvent**



### Technical Data Sheet

# **Ethyl Acetate**

CAS No. 141-78-6 IUPAC name: ethyl ethanoate Other names: acetic acid ethyl ester

### TYPICAL PHYSICAL PROPERTIES

Parameter	Conditions	Units	Value
Density	20°C	kg/l	0.9008
Litres per tonne	20°C		1110
Boiling point		°C	76.5
Azeotrope with water			
wt % so	olvent	% wt	6.79
boiling	point	°C	3.435
Flash point	ACC	°C	- 3
Auto-ignition temperature		°C	426
Flammable limits			
1	upper	% volume	11.5
	lower	% volume	2.2
Viscosity	20°C	mPa.s	0.46
Refractive index	n <sub>D</sub> <sup>20</sup>		1.373
Vapour pressure	20°C	mbar	96.9
Relative evaporation rate	20°C	(n-butyl acetate= 1)	4.2
Volume resistivity		Ωm	1.1 x 10 <sup>7</sup>
Hansen solubility parameters			
	$\delta_d$	MPa <sup>1/2</sup> ({cal cm <sup>-3</sup> }) <sup>1/2</sup>	15.8 (7.7)
	$\delta_{p}$	MPa <sup>1/2</sup> ({cal cm <sup>-3</sup> }) <sup>1/2</sup>	5.3 (2.6)
	$\delta_h$	MPa <sup>1/2</sup> ({cal cm <sup>-3</sup> }) <sup>1/2</sup>	7.2 (3.5)
NHE solubility parameters			·
solubility para	meter		8.8
fractional po	olarity		0.167
hydrogen bonding	index		8.4

# ABBREVIATIONS

ACC Abel Closed Cup NHE Nelson, Hemwall & Edwards

#### HANDLING AND STORAGE

Ethyl Acetate is a highly flammable liquid.

Bulk quantities of Ethyl Acetate must be stored outside in detached tanks. Storage tanks must be positioned within a bunded area. Ethyl Acetate must be stored away from sources of heat or ignition, and away from incompatible materials (oxidizing agents, acids, bases, ...).

Dry acetates have a negligible corrosive action on metals and may be stored or processed safely in either mild steel or aluminium. They can be stored under ambient conditions of temperature and pressure.

Blanketing must be provided on the storage tanks using dry nitrogen. Blanketing is required to retain the quality during prolonged storage. It also prevents the formation of a flammable atmosphere in the vapour space. Ethyl Acetate is stable under recommended storage conditions for 2 years.

Storage and transfer equipment must be adequately earthed and bonded to prevent the accumulation of static charges. Storage tanks should preferably be bottom filled. Where top filling has to be carried out, the filling should exclude the possibility of splashing.

Under normal conditions in industrial use Ethyl Acetate does not present an appreciable health hazard. Precautions should be taken to prevent entry into the eyes and to avoid prolonged or repeated contact with the skin. Suitable protective clothing including goggles and rubber or PVC gloves should be worn when handling. Adequate natural or exhaust ventilation should be provided to prevent gross exposure to vapours.

A Safety Data Sheet has been issued describing the health, safety and environmental properties of Ethyl Acetate, identifying the potential hazards and giving advice on handling precautions and emergency procedures. This must be consulted and fully understood before handling, storage or use.

#### **EXCLUSION OF LIABILITY**

Information contained in this publication is accurate to the best of the knowledge and belief of INEOS. Any information or advice obtained from INEOS otherwise than by means of this publication is given in good faith. However it remains at all times the responsibility of the customer to ensure that INEOS materials are suitable for the purpose for which they are intended by the customer.

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Ethyl Acetate - Technical Datasheet - March 2008

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 $\label{eq:APPENDIX-J} \textbf{APPENDIX-J: Material blends tried out to produce LLDPE}$ 

Material	Inner Layer (%)	Middle Layer (%)	Outer Layer (%)
Equate 7087	-	-	80
I -4 5006	20	20	20
Lotrene 5026	20	20	20
Affinity 1881	70	50	-
Elite 5401	10	30	-
PPA	100 g	100 g	100 g

Material	Inner Layer (%)	Middle Layer (%)	Outer Layer (%)
Equate 7087	-	-	80
Lotrene 5026	20	20	20
Affinity 1881	70	30	-
Elite 5401	10	50	-
PPA	100 g	100 g	100 g

Material	Inner Layer (%)	Middle Layer (%)	Outer Layer (%)
Equate 7087	-	80	80
Lotrene 5026	20	20	20
Affinity 1881	70	-	-
Elite 5401	10	-	-
PPA	100 g	100 g	100 g

# APPENDIX - K: DSC Curves of Sealant Material LLDPE

