# Investigating the influence of moisture and temperature on adhesion and barrier properties in laminates

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This Master's thesis has been done within the Erasmus Mundus Joint Master Degree FIPDes, Food Innovation and Product Design.

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A packaging material analysis project for Tetra Pak

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# Abstract

A world leading food processing and packaging company, Tetra Pak works with the vision of ‘Protect what’s good’ and aims to be a pioneer in developing sustainable packaging. Tetra Pak offers a wide range of carton packaging out of which aseptic carton packages are the most common. Aseptic cartons have laminate packaging material with six layers consisting of paperboard, aluminium (Al) and polyethylene (PE). While recycling the cartons post-consumer consumption, it is difficult to separate Al and PE layers. Hence, Tetra Pak is focusing on developing alternative packaging materials with decreased amount of aluminum and polymer and increased amount of fibers.

One of the new packaging materials (NPM) is a non-foil packaging material with reduced Al content. The change in material structure influences its barrier properties against oxygen and moisture along with mechanical properties such as adhesion. The barrier properties and adhesion are mainly influenced by two factors such as temperature and moisture.

This thesis work is conducted in collaboration with Tetra Pak and focuses on studying the influence of temperature and moisture on the parameters adhesion, oxygen transmission rate (OTR) and water vapor transmission rate (WVTR) over time on five variants of NPM. Adhesion, OTR and WVTR tests were performed at different temperature and humidity levels. The evaluation of adhesion results showed that PM 10802 had clear split between the metallized layer and polymer layer. The OTR test revealed that PM 10802 had values closest to the reference value when compared to the other variants. While the WVTR values obtained were far away from the reference value, PM 10802 was comparatively closer to the reference value. Overall, this variant exhibited better properties when compared to the other variants.

**Keywords:** sustainable packaging materials, temperature, moisture, adhesion, barrier properties.

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Lund, May 2021

Ramyashree Srinath

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# List of acronyms and abbreviations

| | |
|---|---|
| Al | aluminium |
| ANOVA | Analysis of Variance |
| ASTM | American society of Testing and Materials |
| Delam. | delamination |
| LDPE | low density polyethylene |
| NPM | new packaging material |
| OGTR | oxygen gas transmission rate |
| OTR | oxygen transmission rate |
| PE | polyethylene |
| PET | polyethylene terephthalate |
| PM | packaging material |
| LDPE | low density polyethylene |
| RH | relative humidity |
| SEM | Scanning electron microscope |
| Temp. | temperature |
| WVTR | water vapor transmission rate |

# 1 Introduction

*This section presents the background, the purpose, and the objectives of this project. The brief description of the company is presented along with the outline of this project.*

## 1.1 Background

Customers from across the food industry in 160 countries around the world buy products and services from Tetra Pak. More than 190 billion Tetra Pak packages were sold in 2019 with a net sale of €11.5 billion (*Tetra Laval 2019-2020*, 2020) out of which carton packages constitute the most and are used for various food and beverage products.

Tetra Pak is a world leading food processing and packaging company which works with the vision of 'Protect what's good' and provides safe, innovative, and environmentally sound products & packaging. Tetra Pak investigates new possibilities constantly to deliver the best solutions for liquid food packaging and identified four core customer needs which lead to four new strategies to be achieved by 2030: Deliver food safety and the best quality; lead the sustainability transformation; integrate and optimize customer operations; innovate for customer growth. While making the food safe and available everywhere, Tetra Pak has also taken the responsibility to address the increasing sustainability requirements in the society. The company offers a wide range of carton packages (mostly aseptic) in various size and shape along with consideration of consumer convenience, optimal shelf life and easy opening.

Composite aseptic carton packages are constituted of paperboard, aluminium (Al) and plastic layers (Zhang et al., 2014). The plastic layers are constituted of polyethylene (PE). The paperboard is usually laminated with Al foil layer by using a lamination technique which might be extrusion lamination, glue lamination or wax lamination and this step is performed before sending it for printing (StoraEnso, 2005). Further, Al/PE layers are adhered to each other by corona treatment which helps to form crosslink between them and improve adhesion and mechanical endurance of the layers. Corona treatment is a surface technique where corona discharge is used to deposit the thin films on Al to improve corrosion resistance which further means that the adhesion between them is strong (Popelka et al., 2017). For most of the aseptic carton packages a sterilant, hydrogen peroxide is used which is followed by hot air ($60^{o}$-$125^{o}$C) (Pakaging, 2016). This implies that the packaging material must be resistant to higher temperatures, as the whole sterilization process is done at higher temperatures.

To avoid food spoilage, the material used in the packaging must possess good oxygen and moisture barrier. This makes it important that the layers of the carton packaging have high resistance to oxygen and different temperature and moisture levels. Parameters such as oxygen transmission rate (OTR) and water vapor transmission rate (WVTR) are used to test the amount of oxygen and moisture that enters the carton packages. These rates when low, indicate good barrier properties. Apart from barrier properties, the material should have good mechanical properties, especially adhesion. If adhesion is affected by temperature or moisture, it might lead to separation of layers. This further will result in packaging material losing its functionality. Although good adhesion is a requirement, it is also necessary to be able to separate the layers while recycling.

There are limitations in the recycling industry to recycle the laminates used in cartons after being consumed, especially Al and PE layers since it is a tedious procedure to separate these into elemental components due to strong adhesion between them (Zhang et al., 2014). Even after being separated, there is limited infrastructure and market for a material that contains both Al and PE.

To pursue their vision and improve the recyclability, Tetra Pak is focusing on creating a sustainable beverage carton that is fully renewable and recyclable but without compromising on food safety requirements. Tetra Pak's ambition is to deliver a package that contributes to low carbon circular economy (*Sustainable-packages*, n.d). Hence a value proposition was made aiming to improve the recyclability, further increase the renewability, and deliver a lower carbon footprint of the overall beverage carton. To achieve the value proposition made, Tetra Pak is working on new packaging materials which aims to decrease the amount of aluminum and polymer and increase the amount of fibers. Hence, as an alternative, they are looking into metallized surfaces and its adhesion to PE.

When compared to the structure of aseptic carton packaging, the material structure is different in new packaging material (NPM). Regardless of the layer composition of packaging material, it must have all the properties that are required to keep the food safe. This means that the material must possess good barrier against oxygen, light and moisture. With respect to adhesion, the metallized surface and polymer layer must be able to split so that it can ease the process of recycling. Also, the Tetra Pak packages are sold to different parts of the world where the environmental conditions are very different from one another. This makes it necessary to check if the external environment conditions such as moisture and temperature has influence on the packaging material and its properties. But previous studies and experiments conducted on NPM material have revealed concerns regarding the barrier properties and the adhesion behavior of the laminates when stored at different temperatures (Tetra Pak confidential report).

## 1.2 Thesis study purpose

The studies on the material structure of new generation material and its properties helps to understand how it is different when compared to the aseptic cartons. However, the main purpose is to find out the cause and plausible solutions to the concerns raised from the previous work on NPM. To address these concerns, the thesis focuses on studying and testing five variants of NPM for its barrier and mechanical properties and to check how they perform over time and under different climates. This helps to get an understanding of how moisture and temperature affect adhesion properties between the metallized paper and polymer layers. The work further helps to get an understanding on how OTR and WVTR are influenced by moisture and temperature in NPM. Studying the above mentioned parameters will assist in concluding the overall packaging performance of the NPM.

## 1.3 Objectives

Based on the study purpose of the thesis, the key objectives of the thesis work are divided into three:

1. To find out if moisture sensitivity and adhesion mechanisms affect the adhesion measurements by performing quantitative measurements.

2. To understand how the influence of different moisture and temperature levels over time affect the adhesion between metallized paper and polymer layers by performing qualitative analysis.
3. To understand how the influence of different moisture and temperature levels over time affect oxygen transmission rate (OTR) and water vapor transmission rate (WVTR) of the NPM.

## 1.4 Research delimitations

The study focuses on investigating the influence of moisture and temperature on adhesion and barrier properties over time. Due to the time constraints, the moisture and temperature influence was studied only up to 8 weeks for adhesion. Due to the same reason only one type of WVTR test performed on the NPM. Additionally, it was difficult to perform further analyses on the material that would assist in understanding the packaging material properties better. However, the objectives of the thesis study work were achieved in the given time.

Since the study is on a NPM which is an ongoing project at Tetra Pak, it is classified as highly confidential. Due to the confidentiality, there is limited amount of information that can be revealed as part of the thesis work. Given that the study has both quantitative and qualitative research, the rich data and descriptions obtained cannot be disclosed in order to protect the ongoing research work. The lack of valuable information might lead to a few assumptions. However, the report was written in general terms and by using alternative code words to avoid revealing any important information.

As the NPM is in the development stage, there were limited literature sources available with respect to the layers used in the structure and their properties. It is important to note that this work does not generalize the findings as it is specific to Tetra Pak’s project. However, it motivates further research and development of the NPM at Tetra Pak.

# 2 Theoretical Framework

*This section is a combination of important background information from various literature sources and from Tetra Pak. This section focuses on three main topics. Firstly, the development and advantages of sustainable food packaging. Secondly, the structure of NPM currently developed at Tetra Pak. Lastly, on the key parameters of the laminates.*

## 2.1 Development of sustainable food packaging

"*Even though the main task of packaging is to protect and distribute the right product to the right end user in a safe, cost-efficient and user-friendly way, it is often regarded as a burden for the environment*" (Rezaei et al., 2019).

The importance of food packaging has been existing over a century now and there have been great advances to improve food quality and safety, one such example is the invention of canning. Over the time, the consumer preferences have changed, and this led to new advances in the food industry. Eventually, the concerns have shifted towards sustainable packaging and have brought the value of sustainability to the forefront of the corporate agenda (Rezaei et al., 2019).

The rise in sustainability concerns has brought general awareness on products and services that create a negative impact on environment, and this has made the industries to focus on designing products and services that are sustainable. Further the development of packaging design was introduced as an integrated process in the industries and companies both theoretically and practically (Rezaei et al., 2019).

Tetra Pak has successfully integrated development of packaging design along with their visions and sustainable goals. As a result, they have been working on designs that create a good impact on the environment. Out of the wide range of packaging designs, Aseptic Cartons are a good example of a packaging design that has many environmental advantages (Natalie Jacewickz, 2018).

## 2.2 Aseptic Carton packaging

Aseptic packaging is absolutely different from conventional packaging technology, which allows a similar shelf life like retort processing or canning (Götz et al., 2014). It is suitable for low-acid food (pH > 4.5), products like milk and milk products, for example, puddings as well as non-dairy desserts, fruits and vegetables juices, soups, sauces, and particulate foods. Aseptic packaging is a well-established technique that is used for decades.

In general, aseptic packaging follows the steps of filling the sterile product followed by hermetic sealing to prevent contamination. The sterilization should be achieved through adequate thermal treatment under aseptic conditions (Götz et al., 2014).

The legislative authorities and the consumers play an important role in setting parameters for the sensory and nutritional parameters of the food products in packages. Shelf-stable, high-quality food with a maximum degree of safety has been a focus in processing of liquid and semisolid foodstuffs.

To address these demands and satisfy the customers, Tetra Pak has been producing a wide range of aseptic carton packages. Composite aseptic packaging materials are composed mainly of paper, Al, and low-density polyethylene plastics (LDPE); a small amount of printing ink, coatings, and adhesives are also present (see figure 1).

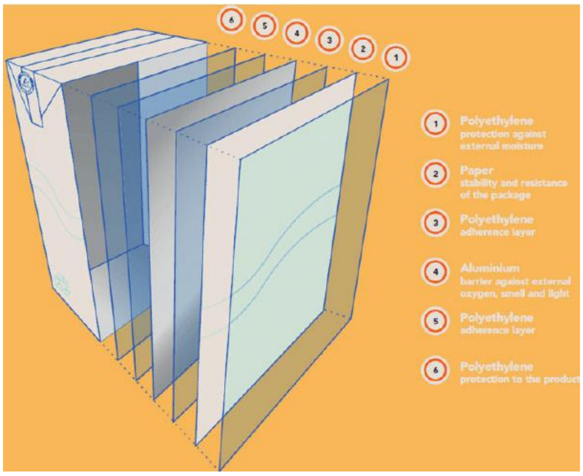



**Figure 1. Composition of aseptic packaging material (Tetra Pak)**

Currently Tetra Pak is working on sourcing materials (especially paperboard) responsibly year-round to combat deforestation as 71% of the raw materials used in the carton packages is paperboard. The current standard beverage cartons also contain thick layers of polymer plastic that keep the product fresh and prevent moisture from getting in and out.

Tetra Pak agreed on launching fully renewable aseptic carton packaging to the market. Further decided to develop bio-based products as a result of which a range of renewable aseptic cartons were introduced (see figure 2).


**Figure 2. Sustainable aseptic carton packages (Sustainable packages, n.d.)**

### 2.2.1 Tetra Brik® Aseptic

One of the world's most efficient beverage carriers is Tetra Brik® Aseptic and uses no unnecessary material, weight, space or energy. Its rectangular package shape stacks neatly on pallets, in transport containers, on supermarket shelves and in the store cupboard at home. It has ten different formats depending on different needs, does not require refrigeration and is cost-efficient (*Tetra Brik aseptic*, n.d.). The structure of the packaging material (PM) used in Tetra Brik® Aseptic acts as a reference for the NPM.

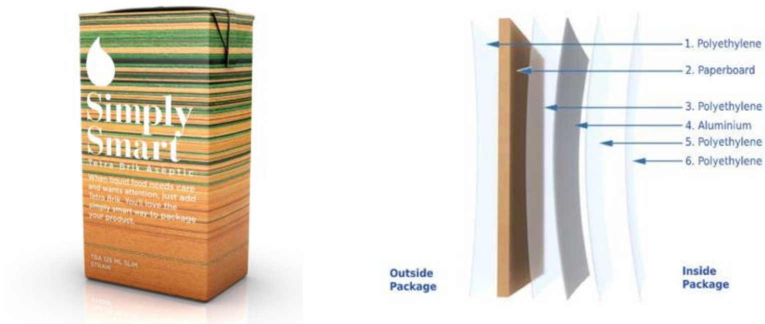



**Figure 3. Structure of Tetra Brik® Aseptic**

## 2.3 Aluminium foil (Al)

Aseptic cartons include Al layer because it prevents oxidation, light damage and keeps the perishable food safe without refrigeration. However, its production can be associated with environmental and social issues. The issues range from land use and production of hazardous by-products; to working conditions, health and safety. Addressing these challenges is complex and requires continuous research and innovation, as well as working with multiple stakeholders.

As mentioned above, to provide vital protection from oxygen and light, a thin layer of Al is used in aseptic cartons. Despite the advantages of using Al (see table 1), Tetra Pak notes that this layer is responsible for about one-third of the climate impact from its base materials. In 2018, Tetra Pak set a target for reducing the carbon dioxide emissions for its Al foil suppliers and hence is continuously investigating alternative barrier materials (Beth newart, 2019).

**Table 1. Key advantages of Al used in packaging (Beth Newart, 2019)**

| Property | Advantages |
|---|---|
| Appearance | Bright, reflective gloss makes for an attractive appearance |
| Stability | When exposed to air, aluminium forms an oxide layer that prevents further oxidation. It is also inert and does not form toxic compounds when exposed to most chemicals, including most foods and cosmetics |
| Barrier properties | Heavier gauges form a complete barrier to gases and water. Aluminium reflects light, making it a suitable material to protect light-sensitive products |
| Hygienic properties | Aluminium's smooth metallic surface is non-absorbent. It can be easily cleaned and sterilised |
| Formability | Aluminium's ductility makes it easy to form. It has excellent dead-fold properties. Its friability (ability to crumple) makes it useful for blister packaging |
| Conductivity | Aluminium conducts electricity and heat, making it useful for applications such as induction heat sealing of containers |
| Recyclability | Aluminium can be recycled at relatively low cost (recycling requires about 5% of the energy required to refine aluminium) |

In aseptic cartons, the Al layer is adhered to PE (inside layer) using adhesive. Post consumption, the Al–plastic composite is difficult to be effectively separated and fractionated into elemental components; therefore, much of this kind of liquid packaging are now burnt or embedded in disposable consumer goods, which results in resource waste and environmental pollution (Zhang et al., 2014). Although the study presented in Zhang et al.,2014 suggests a separation method of Al/PE, the disadvantages of Al especially in terms of its impact on the environment has raised concerns.

## 2.4 Company Background

### 2.4.1 New packaging material (NPM)

The traditional PM is with Al foil, but the ambition is to reduce the amount of Al. For this purpose, Tetra Pak is exploring a large number of possibilities with alternative barrier materials to reduce or replace the Al layer and improve recycling. One of the alternatives is metallized paper. The structure of NPM is inspired by Tetra Brik® Aseptic.

The new packaging material focused in this study constitutes of polymer, paperboard, and metallized paper. As mentioned in the background, the future carton package comes with aim of increasing the fiber content in packaging material structure with reduced Al content and reduced plastic.

Research and development on the NPM have been carried out for a few years. During this time the material was subject to continuous changes and improvements to enhance the overall performance of the material. It is quite challenging to develop the material structure that will behave the same as the reference material (Tetra Brik® Aseptic). Continuous testing of the material has led to new variants of packaging material with different types of barriers.

#### *2.4.1.1 Metallized paper*

As mentioned before, Tetra Pak is exploring alternative barrier materials with reduced Al content. One of the alternatives is metallized paper. The reduced Al content is an advantage for recyclability compared to Al foil used in other flexible packaging. The properties of reduced Al remains similar to Al foil i.e., it provides barrier properties to the substrate by forming a barrier against light, water vapor, odour and oxygen. But overall barrier properties of metallized paper might be less good when compared to Al foil.

Al is metallized more commonly on plastic films than on paper which is the reason that a lot of research and studies are performed on metallized films and not on metallized paper. Overall, metallized films exhibit good barrier properties, are flexible and light (*Metallized PET Films*, n.d.). The barrier and mechanical properties vary depending on the type of film used and the same applied when Al is being metallized on paper.

*2.4.1.2 Inside layers*

The NPM includes layers of PE. The PE layers provide protection and prevent the passage of moisture, water and oxygen from inside the package. The cast films are used more commonly than blown films because of advantages such as resisting tear propagation and because they are clearer. Even though cast and blown films have similar properties, one of the differences in terms of method is that the cast film is produced by horizontal process and the blown film by vertical process (Lantech, 2018). Cast films have better optical properties and also adhere well due to the corona treatment whereas blown films exhibit balance between the machine and transverse directions (Wagner, Mount and Giles, 2014; Wagner, 2016).

## 2.5 Key Parameters in Laminates

### 2.5.1 Adhesion

Good adhesion between the layers in laminate packaging is considered to be very important as the packages are used to provide long shelf-life. Yet many manufacturers face problems due to poor adhesion of laminates. In laminate packaging, the adhesion is usually between polymer and Al as well as polymer and paperboard. To increase the value of products and improve its overall properties, polymer is laminated on to the paperboard. The factors that affect the adhesion of paper to the polymer include physical and chemical characteristics such as roughness and surface energy. The adhesion between the polymer and Al is usually affected by influence of stress, influence of water. Also, PE is hydrophobic in nature, due to which it exhibits low wettability affecting the adhesion. The polarity of the polymer and its surface energy also has an impact on adhesion (Popelka et al., 2017). To improve the adhesion, manufacturing industries use various treatments such as corona, flame and ozone (Madeira et al., 2018). As much as it is important to have strong adhesion, it is also important to note that the layers Al/PE must be easily separable to be able to facilitate easy recycling. It is quite challenging to be able to compromise on either of the factors: strong adhesion of Al/PE or easy separation of PE/Al.

In NPM, the adhesion performance between the metallized paper and polymer depends on the type of substrate used. Apart from the factors mentioned above, different moisture and temperature levels also affect the mechanical properties of the material, mainly adhesion. It is important to consider these factors as the packages are stored and sold in different parts of the world with different temperatures and humidity levels. There are no exact studies showing the influence of temperature and humidity over time on adhesion. Also, there are very limited studies relevant to adhesion properties of metallized paper and inside layers. Therefore, the measurement of the adhesion between the metallized layer and the polymer is of general importance in product development and quality control of flexible packaging materials.

There are different types of adhesion out of which ‘practical adhesion’ is the most common and this is defined when there is separation between two different layers. This is generally measured in terms of forces or the work required to separate the layers apart. Factors such as adhesion mechanism, test speed, thickness of the metallized layer and substrate and temperature affect the practical adhesion (Jesdinszki et al., 2012).

The results from the previous adhesion tests performed on NPM material raised certain concerns regarding the material. In most of the previous measurements the main observation was that the different amount of metallized layer followed the peel arm or in other words metallized layer was stuck to the inside layer. Further there was also delamination in paper barrier of some samples. Although the exact reason of why this phenomenon occurs couldn’t be found, it is probable that the amount seemed to differ either based on the type of paper barrier used or on the different humidity levels. This further led to the need of some more improvements and observations of the barrier.

### 2.5.2 Barrier properties

Oxygen and water when in contact with food leads to many undesirable reactions which reduces the shelf life and leads to food spoilage. Hence, the intention of using barriers in food packaging is to provide protection from light, oxygen and moisture. In most of the aseptic cartons produced by Tetra Pak, Al provides the necessary barrier along with the other layers. The two commonly used parameters that help to measure the barrier properties are oxygen transmission rate (OTR) and water vapor transmission rate (WVTR). Low levels of OTR and WVTR are desirable as they indicate that the material has good resistance to oxygen and water (Kjellgren, 2005).

The barrier properties are mainly affected by temperature and moisture. Al can be used for frozen food and for storage of foods at higher temperature and moisture levels which means that it is stable at quite extreme temperatures (Kerry, 2012). Heavier gauges of Al foil (above 17 mm) provide a complete barrier to gases and liquids. Thinner gauges do allow some transmission: as an example, the typical WVTR for foil of 9 mm thickness is 0.3 g/$m^2$ per 24 hours (at a temperature of 38°C and a relative humidity of 90% RH) (Kerry, 2012). The typical OTR value is less than 0.006 cc/$m^2$ per 24 hours (*Barrier Properties of Film*, n.d.).

In NPM, as the Al foil is replaced by metallized paper, the barrier properties also differ. This further means that the OTR and WVTR values are different. It is indeed important to test NPM for its barrier properties and compare it to a reference material. Previous OTR and WVTR test results of NPM are available for only a few variants and the values were obtained from a Tetra Pak confidential report. These results were compared to the set reference values of NPM and also with values of a reference material (with Al foil).

Overall, there is very limited/no literature available explaining the barrier properties of materials consisting of metallized paper or the barrier alone. However, there are studies that mention about the OTR of different films and metallized films. For a 12-micron metallized PET along with adhesive and LDPE, the OTR and WVTR values are 0.6 cc/ $m^2$ per 24 hours and 0.4 g/ $m^2$ per 24 hours consecutively (*Barrier Properties of Film*, n.d.). (Kjellgren, 2005) study shows OTR values of thick films of various polymers (table 2), initially which was obtained from the study Savolainen et al., 1998. It is important to note that the values here are for thick films and the values of OTR will be lower as the thickness of the film reduces. The same is observed for WVTR as well, the values depend on the thickness of the material and the values decreases for materials with lower thicknesses. Although these studies are on films, this also applies to the metallized paper.

**Table 2. OTR values of 50 µm film at 23 °C (cc/$m^2$ 24 h bar) of various polymers (Kjellgren, 2005)**

| Material | OTR |
|---|---|
| PE-LD (polyethylene low density) | 3500 |
| PS (polystyrene) | 2000 |
| PP (polypropylene) | 1800 |
| PE (polyethylene high density) | 1300 |
| PVC (polyvinyl chloride) | 100 |
| PET (polyethylene terephatalate) | 80 |
| PA-6 (polyamide 6) | 25 |
| PVDC (polyvinylidene chloride) | 2 |
| EVOH (ethylene vinyl alcohol) | 0.2-2 |

When measuring the oxygen transmission rate on barriers and packaging material (PM) with coating and different substrates, the OTR generally doubles when the relative humidity increases from 50%RH to 80%RH. This occurs due to the close proximity of the product with the barrier (Tetra Pak confidential report). The main water and oxygen barrier is the inside polymer and a reduced layer of Al in NPM. The WVTR and OTR are generally measured at standard temperatures and humidity levels of $38^o$/90% and $23^o$/0% respectively. When testing the materials, depending on the type of material and the purpose, the temperature and humidity levels are set. This makes it crucial to understand if and how the OTR and WVTR changes over time.

# 3 Materials and Methods

*This section presents the types of experimental analyses and describes each method used along with the materials required. The study on the environmental conditions required for the analysis are also described.*

## 3.1 Materials

The experimental analysis of NPM material focused on three different tests: testing the adhesion between the Al and LDPE layer (Adhesion test), OTR and WVTR. All the tests were performed on flat packaging material and at different temperature and relative humidity (RH) levels. Five packaging material (PM) variants were provided for the tests and with different coatings (table 3). The NPM variants are represented as A, B, C, D and E respectively. There are two different blend types used. The paper barrier of first three variants (A, B, C) is coated with a blend type A consisting of two different components and the last two variants (D, E) are coated with blend type B consisting of two layers of the same component.

**Table 3. Five variants of New packaging material**

| Variants | Coating | Inside film |
|---|---|---|
| A-10624 | Blend A | Film 1 |
| B-10626 | Blend A | Film 2 |
| C-10806 | Blend A | Film 1 |
| D-10802 | Blend B | Film 1 |
| E- 10809 | Blend B | Film 2 |

## 3.2 Environmental conditions to study

The packaging performance, especially of food packaging mainly depends on barrier properties such as OTR and WVTR of the material. When compared to mono layer packages, laminates provide better barrier protection (Galic, 2015). Paperboard being one of the major components of Tetra Pak cartons, it is necessary to make sure that it does not come in contact with moisture. High humidity and temperature levels can affect the paperboard packages and hence the store location must be selected carefully (Tutak *et al.*, 2019). To resist the external temperatures, it is necessary that the PM is tested for its barrier properties, especially oxygen and water since they can cause food spoilage in packages.

At Tetra Pak, the cartons are usually stored in a controlled environment. The standard temp./RH used at Tetra Pak to store the samples is 23$^o$/50%. To test the samples for hot and humid conditions, the standard temp./RH 35$^o$/80% is used. The other temperatures used to store the samples varies on the type of the sample and requirement. These storage and test temperatures have been used at Tetra Pak for a long time.

In NPM, apart from barrier properties, adhesion between the layers is also affected when exposed to high humidity and temperature levels. For adhesion, the samples were stored at two different temperatures and humidity levels 23$^o$/50% and 35$^o$/80%, signifying two environments with low and high temp./RH levels. As mentioned above, these are the standard temperatures used and for NPM, the storage temperatures were chosen based on the availability at Tetra Pak. More variations of temperature can be studied depending on the type of material and needs and one such study is shown in Tutak *et al.*, 2019.

The temperature and humidity levels are also standardized for OTR tests by Tetra Pak. However customized temperatures can be used for OTR. The temp./RH of 23$^o$/50%, 23$^o$/80% and 35$^o$/80% were used to check the oxygen barrier of the NPM at high humidity. Since the weight loss method was developed for checking WVTR, the test method already had a set standard temp./RH of 50$^o$/95%. This condition was chosen by Tetra Pak to accelerate the WVTR test. It helps by reducing the time to obtain the results unlike in standard WVTR test.

## 3.3 Adhesion Test

Various adhesion test methodologies have been developed depending on the type of application namely Scotch®-Tape test, peel test, scratch test, pull test, fragmentation test, etc. Practical adhesion/measured adhesive strength is commonly used in most of the adhesion measurements and is affected by various factors such as humidity, thickness of substrate film and temperature (Jesdinszki *et al.*, 2012). Although there are different tests, peel test is widely and commonly used to measure the adhesion strength of a metallized layer on the inside layer (polymeric substrate) (Jesdinszki *et al.*, 2012). This test is particularly used when quantitative evaluation of the metal adhesion has to be performed.

The peel test procedure is performed as per the standards set by American society of Testing and Materials (ASTM) and ASTM D2918 is the standard test method for Durability Assessment of Adhesive Joints Stressed in Peel (ASTM D2918-99(2020)). Based on this procedure, the test method for NPM was developed to test the adhesion between the metallized paper and polymer.

### 3.3.1. Experimental design

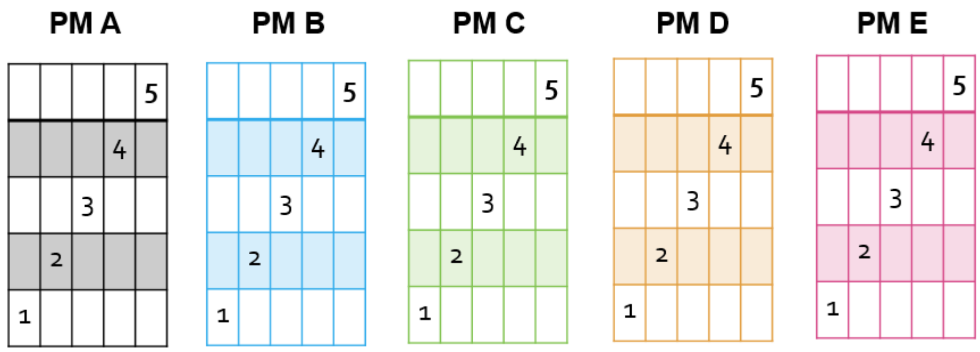
#### a) Test equipment

The equipment required for conducting the test include 'INSTRON tensile tester' with a load cell, range of 0-100 N and has an accuracy of ±0.5%. A tensile tester is an electromechanical test system that applies a tensile force to a material to determine its strength and deformation behavior until it breaks. The equipment is linked with 'Bluehill' software that helps to record the measurements, analyze, and store the data (figure 4). A cutting device was used that has a specification of 15.0±0.1 mm wide. To imitate the usage of hydrogen peroxide bath, a water bath with a temperature set to 75$^o$±3$^o$C was used. A storage chamber to store the samples before testing and other tools such as pliers to hold the samples, thermometer to check the water temperature, water for cooling (room temperature) in a beaker of 1000cc, and ordinary scissors were used.

**Figure 4. Tensile tester (INSTRON)**

### b) Sample and test preparation

The experiments were performed on flat samples and hence the packaging material used for the test is in the form of sheet rolls. The sheets were cut from the roll in a way that it includes 5 packages. The samples were cut using a cutting device set to 30mm. Each package includes 5 panel sides and for the experiment it was necessary to cut 5 sample strips, one from each package. Each strip was taken from different panels (front, rear and side) and different packages coming from the same lane (figure 5). Following this method helps to obtain test results for a range of samples from different packages. Having replicates further helps to obtain an average value and minimize the error. The water bath must be heated to 75°±3°C and a beaker filled with cold water should be placed close to the water bath.




*PM- Packaging material

**Figure 5. Sample cutting of the variants for adhesion test**

### c) Test method

In the test method of NPM, it is important to note that all the measurements must be performed in a laboratory with controlled climate (23°/50% RH). The evaluation was performed in two steps:

- Quantitative evaluation by peel test using a tensile tester.
- Qualitative evaluation by visual grading of amount metallization on peel arm after peel test.

For the quantitative evaluation, 180° Peel test was used for NPM and was performed using the tensile tester. Before placing the sample in the tensile tester, it was treated in hot water bath at 75° for 20 seconds and dipped in cool water. This step was performed to imitate the step performed in industrial process where the material is passed through hydrogen peroxide. Further the sample was cut using a 15mm cutting device. The tensile tester has two clamps, one holding the peel arm and the other holding the substrate. This is important as one of the adherends must be flexible enough so that it can fold back on itself (figure 6) (Ebnesajjad and Landrock, 2015). The test results obtained through the software program shows the graph for force (N) vs displacement (mm) similar to the graph shown in figure 7.

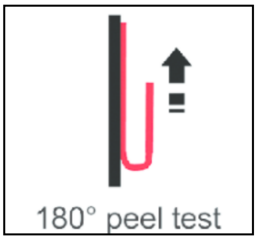



**Figure 6. Direction of the peeling in 180º Peel test**

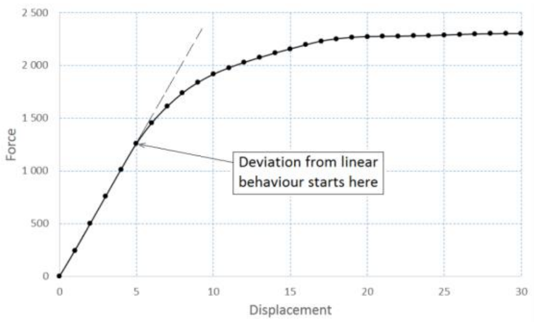



**Figure 7. Force vs displacement graph**

The qualitative evaluation i.e., the grading of the sample is conducted after the quantitative evaluation (peel test). The evaluation was performed by grading the samples by visually observing the amount of metallization stuck on the peel arm after the peel test. The grading scale ranges from 0 to 6 indicating different percentages of metallization on the peel arm (table 4). The grades also include comments based on the type of delamination and interface in which the delamination occurs. The grade is noted by observing an area with the least metallization on the peel arm. This area also corresponds to the average force value taken from the same position/area on the peel test graph.

**Table 4. Definitions of grades**

| Grade | Definition |
|---|---|
| 0 | Delamination within inside PE layers |
| 1 | 0% metallization on peel arm |
| 2 | 1-5% metallization on peel arm |
| 3 | 6-15% metallization on peel arm |
| 4 | 16-50% metallization on peel arm |
| 5 | 51-99% metallization on peel arm |
| 6 | 100% metallization on peel arm |

## 3.4 OTR Test

One of the important parameters to test the barriers in a packaging material is OTR which correlates to the packaging performance. It is a common method of testing with standardized procedure depending on the type of material and parameters. The test is performed to measure the transmission rate of oxygen through film, sheeting, laminates, coextrusions or packaging material. ASTM D3985 provides the standard procedure for OTR test which helps to determine the rate of transmission of oxygen gas, at steady state and at a given temperature and %RH level. Oxygen gas transmission rate (OGTR) is one of the important parameters for packaging material at a given temperature and %RH and ASTM F1927 provides the test method for OGTR (Ebnesajjad and Landrock, 2015).

For NPM, the OTR procedure used was taken from the standard procedures, ASTM D3985-05 and ASTM F1927-14. The test methods were performed using a coulometric detector and sensor. The test determines OGTR, permeability and permeance at controlled RH through barrier materials and through plastic film. The test was conducted on the whole packaging material and in a laboratory that complies with the environmental requirements of the instrument.

### 3.4.1. Experimental design

### a) Test equipment

Ox-Tran is the instrument used to test the oxygen permeability and is highly accurate (figure 8). It has lowest detection rate with the range of 0.005-200 at 50 $cm^2$. It is manufactured by Mocon and each type of Ox-Tran has its own operator manual. It consists of a coulometric sensor which avoids the need to calibrate the instrument (*Ametek-Mocon*, n.d.).


**Figure 8. OTR test instrument (Ox-Tran)**

The test cell principle used in the instrument is shown in figure 9. The test material acts as the membrane separating a stream of dry nitrogen from an oxygen stream on the other side of the chamber. This creates a partial pressure difference which further provides a driving force for oxygen molecules to diffuse through the film to the low-pressure side. The film barrier determines the rate of oxygen permeation and this is measured by the coulometric sensor present in the instrument which is present in the outgoing stream on the nitrogen side. The test is complete when equilibrium or steady state is achieved i.e., it is complete when the sensor detects a constant amount of oxygen in the nitrogen carrier stream (*Polyprint- OTR*, n.d.).

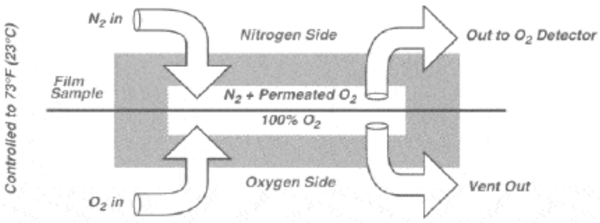



**Figure 9. OTR test cell principle**

**b) Sample preparation**

The test samples used were flat materials and free of defects. All of the samples were conditioned at RH and temperature at which they were measured at. For NPM, the samples were conditioned at $23^{o}$/50%RH, $23^{o}$/80%RH and $35^{o}$/80%RH. The samples were cut out depending on the cell size of Ox-Tran instrument used.

**c) Test method**

The samples were placed in the test cell and depending on the parameters set, measurements were taken until the transmission rate had reached the equilibrium. The built-in software calculates the individual OTR results of the measured samples automatically. The environmental conditions along with the number of samples and mean values were also reported.

## 3.5 WVTR Test

Like OTR, WVTR is also one of the parameters which correlates to the packaging performance. A critical function of the packaging material is to protect the products and not allow the moisture to transmit through the barriers. The test method is to measure the water vapor transmission rate through the barriers and their ability to resist moisture transmission. The test principle is similar to OTR test, but the oxygen stream is replaced with wet nitrogen stream. The measurements are performed at a set temperature and RH (*Polyprint- WVTR*, n.d.). ASTM F372-99 and ASTM F1249-13 provide the standardized method for WVTR which uses an infrared detection technique.

The test method developed for NPM is called 'Weight loss method' which is based on gravimetric measurement of WVTR through a flat sample like films or packaging materials. The typical chosen conditions are $50^{o}$/95%RH indicating a very hot and humid condition. This method is used either for thin films like PE, PET (or any coated films) or on whole packaging material consisting of multilayer structures.

The test principle of gravimetric measurement is to weigh the mass variation of the material by an analytical balance ('Breakthrough Made in Water Vapor Permeability Instruments of Gravimetric Method', n.d.). Gravimetric measurement is further divided into desiccant and water method. For NPM, the desiccant method is used where one face of the material is exposed to high humidity level and the other side to low humidity and here RH acts as a driving force (figure 10).

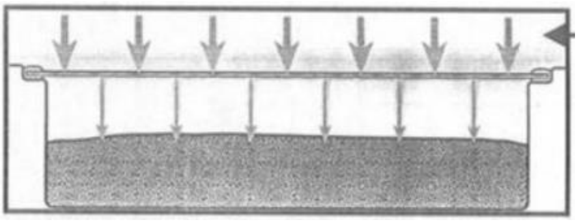


**Figure 10. Desiccant test method principle**

### 3.5.1. Experimental design

#### a) Test method

As mentioned above, it is important that the samples used are free of any mechanical damage. The samples were cut to obtain at least 3 replicates of each sample. The sample was placed in the cell with 50ml water at the bottom and closed. This cell was pre-conditioned i.e., cells were stored in a dry chamber at least for 2 hours. The cells were removed from the chamber and the weight of the samples are recorded using a balance. The cells were closed again and placed in the chamber for 48 hours. The RH and elapsed time were noted and later the samples were taken out and weighed. It is important to note that the samples are not placed in ambient temperature for more than 30 minutes. Usually, the test is carried on for one to two weeks depending on the type of the sample.

#### b) Calculation for weight loss of the samples

The results were noted in an excel file and the weight was recorded after 24h (cell conditioning). The excel formula used for calculations are mentioned below:

Weight 1 (W1) Elapsed time 0h before climate chamber

Weight 2 (W2) Elapsed time in climate chamber (exp. 24h)

Weight 3 (W3) Elapsed time in climate chamber (exp. 48h)

Weight 4 (W4) Elapsed time in climate chamber (exp. 120h)

Weight 5 (W5) Elapsed time in climate chamber (exp. 144h) …so on.

Weight loss

W1-W2 Elapsed time (W2-W1) Exp. (24-0) = 24

W2-W3 Elapsed time (W3-W2) Exp. (48-24) = 24

W3-W4 Elapsed time (W4-W3) Exp. (120-48) = 72

W4-W5 Elapsed time (W5-W4) Exp. (144-120) = 24… so on.

Weight loss (g/cell/24h)

= +W1-W2/$ Elapsed time $*24

= +W2-W3/$ Elapsed time $*24

= +W3-W4/$ Elapsed time $*24… so on.

## 3.6 Methods/tools used for data analysis

To analyze the large amount of data obtained from the tests, statistical analysis was chosen. For the analysis, a software tool 'Minitab' was used. Minitab is a statistical tool that helps evaluating large amount of data by automating calculations and creating graphs. This tool was used mainly for adhesion test as overall 225 samples were analyzed. The tool also helps to plot statistical graphs to understand the data and what are the factors affecting it. The OTR test and WVTR had lesser samples and were analyzed by plotting suitable graphs using excel.

## 3.7 Sample storage and test schedule

The storage and tests of the samples were conducted at Tetra Pak. The samples were stored at different temperatures for different tests and the selection of the temperatures were based on the standard temperatures used at Tetra Pak for tests. The tests were performed for all 5 variants of new packaging material. For adhesion test 5 samples of each variant were tested at two different temperatures/RH ($23^o$/50%, $35^o$/80%).

As mentioned earlier in section 3.1, all tests were performed on flat materials. Adhesion test was performed once every 2 weeks consecutively up to 8 weeks and 50 samples were analyzed in a day except at 0week point (25 samples). OTR was performed at 3 different temperatures and WVTR was performed at 50°/95%. OTR and WVTR was performed by a laboratory professional at Tetra Pak. The storage schedule is shown below in table 5.

**Table 5. Sample storage and test schedule**

| **Tests<br>Temperature (° C)/ RH (%)** | **Weeks of 2021** | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | **11** | **12** | **13** | **14** | **15** | **16** | **17** | **18** | **19** | **20** |
| **Adhesion test<br>23°/50, 35°/80** | | 0w (only 23°/50) | | 2w | | 4w | | 6w | | 8w |
| **OTR test<br>23°/50, 23°/80, 35°/80** | | | | 0w | | | | | | |
| **Weight loss test<br>50°/95** | | | | | 0w | | | | | |

## 3.8 Quality of the Research

To measure the quality of the research done, it is important to critically reflect on the results obtained. A critical reflection helps to identify and question the assumptions or biases made on the results. This reflection further helps to identify problems at their core. To critically reflect on the experimentation done in this thesis work, two concepts are used: Validity and reliability. These concepts explain how well a method, technique, or test measure something and are mostly used for quantitative research.

### 3.8.1 Reliability

Reliability is often referred to consistency of a measurement. This helps to assess if the results can be reproduced when the measurements are repeated under the same conditions. The three tests (Adhesion, OTR, WVTR) performed on the NPM have been evaluated for the reliability of their measurements.

The adhesion test measurements were performed using a tensile tester and the samples were stored in separate storage chambers depending on the different climatic conditions. Each PM variant had five replicates of the sample. In general, having replicates helps minimize the errors and give an average value. The adhesion measurement instrument and the samples for the adhesion test were placed in different locations. The samples that were stored in a controlled environment had to be carried in an external environment to reach the adhesion test lab. Although the samples were handled by storing them in plastic bags (wrapped with Al foil) during transport, slight errors in the measurements can be considered as the material will have a slight temperature difference due to the external environment. As the measurements were performed at 5 test points (0, 2, 4, 6, and 8 weeks), it gives the results over time. Although there might be small errors in measurements due to the transport factor, the rest of the factors in the measurements make the results reliable. Overall, having replicates for NPM helps to avoid errors and get an average value of the peel force. Also, the repetitive measurements for the replicates helps to rely on the results as reliability is checked by consistency of test results.

As the OTR and WVTR test was performed by the laboratory professional and in a controlled environment at Tetra Pak, it is difficult to comment on the errors. However, it can be assumed that there might be instrument errors. These tests were not repeated on the samples but had replicates (five replicates per sample). As mentioned earlier, having replicates helps to get an average value in case of errors. The repetition of these tests in the same environment might have similar values as they are performed using the same instrument and by a professional.

To conclude it is possible to say that the measurements are highly reliable. Lastly, it is important to note that the measurement values can be reliable but not always valid. This means that the results might be reproducible but not necessarily correct.

### 3.8.2 Validity

Validity here refers to the accuracy of the instrument/measurement. In other words, validity can be checked by comparing the results to established theories or to other measures of the same concept. One of the indicators of the measurements being valid is reliability. As per the discussion in 4.1.1. the data is highly reliable which mean that it might be valid too. Even though reliability is not the only factor that supports validity, the other factors such as the type of instrument used, and the environment used to test can support the validity. The instruments used for all three tests were in perfectly working conditions and for consistency, the same instruments were used for all samples. Also, as mentioned earlier the controlled environment was maintained for all the tests.

Since NPM is being developed recently, there were limited theories stating results of the similar material. Regarding adhesion test, there was no literature study indicating the peel force values. However, a few studies mentioned the OTR and WVTR of reference material (Al foil). From the comparison, the values of NPM (table 7 and table 9) were higher than the values of Al foil (section 2.5.2). However, it is difficult to compare and comment as the factors such as, conditions of test and material thickness were different.

To validate the data further, the comparisons were made to the measurements from the previous tests conducted on NPM. But this comparison was done only for OTR and WVTR and not for adhesion. These comparisons helped to conclude the properties of the PM variants. Even though the adhesion test measurements were not compared, the rest of the factors involved in the measurements make the results valid. As the project is ongoing and continuous developments are being made, the results are compared to previous measurements more than the established theories. This is also the reason that there is no previous data available for certain PM.

Overall, the data from the measurements can be considered as moderately valid. This means that certain measurements are valid, and some cannot be commented on as there is no comparison or exact reference values. Unlike reliability, valid measurements are generally reliable which means that if the test gives accurate results, they can be reproducible. To improve the validity, samples can be tested with other measurement tool. The same measurement tool can be used to test samples with known adhesion, OTR and WVTR.

# 4 Results and Discussion

*This section presents the results and evaluation of all the experimental analyses: Adhesion test, OTR and WVTR tests. The results are discussed to answer the research questions. Further, the quality of the research is assessed.*

## 4.1 Adhesion test

The adhesion test was performed to check how parameters such as humidity and temperature affect the peel strength between metallized paper and the polymer. The results were recorded and evaluated for five different variants of NPM. As mentioned earlier, the results were recorded in the 'Bluehill' software in a quantitative data format which include average peel force values and graphs. The amount of force acting on the width of the bonding surface when peeled at a constant velocity is the measurement of peel strength. The amount of peel strength represents the work of cohesive fracture or detachment (Ashter, 2014). The peel strength can also be defined as the measure of average force required to separate two bonded materials (*Com-Ten Industries*, n.d.).

The adhesion test between the metallized paper and polymer was performed to evaluate the peel strength and to find out if there is a change of interface and if cohesive fracture occurs. In case of occurrence of cohesive fracture or delamination, the samples were evaluated to find where the fracture occurs. The past test measurements and observations on NPM material revealed that different amount of metallized paper follows the peel arm. This led to research questions such as:

- Where in the coating does the split happen?
- How are the coating layers impacted by humidity level in paper?
- How does the amount and quality of coating impact the performance?
- What is the reason for different amount of metallization on peel arm?

As mentioned in section 3.3.1 (c) the tests include both quantitative and qualitative evaluation. As part of the quantitative evaluation, the influence of different temperatures and humidity levels on PM at different storage times (0, 2, 4, 6, 8 weeks) was analyzed by first noting down the average peel force values. Further, as part of the qualitative evaluation, the samples were graded based on the amount of metallized on the peel arm (described in table 4). The tabulated data consists of PM variants, replicates, storage time, temperature, RH, average peel force and metallization grade. An example table of the data for one PM variant is shown in table 6.

**Table 6. Tabulated data of PM 10624 in Minitab**

| C1 | C2 | C3 | C4-T | C5 | C6 | C7 |
|---|---|---|---|---|---|---|
| PM | Replicate | Time in weeks | Peel force (N/m) | Temperature | %RH | Delam. grade |
| 10624 | 1 | 0 | x | 23 | 50 | 0 |
| 10624 | 2 | 0 | x | 23 | 50 | 2 |
| 10624 | 3 | 0 | x | 23 | 50 | 0 |
| 10624 | 4 | 0 | x | 23 | 50 | 2 |
| 10624 | 5 | 0 | x | 23 | 50 | 0 |

### 4.1.1 Individual value plot

To visualize the distribution spread of the data obtained, individual value plot was chosen, and this plot helps to provide information on the variability of the data. The individual plot for the NPM variants is shown in figure 11 which mainly shows the levels of delamination in NPM.

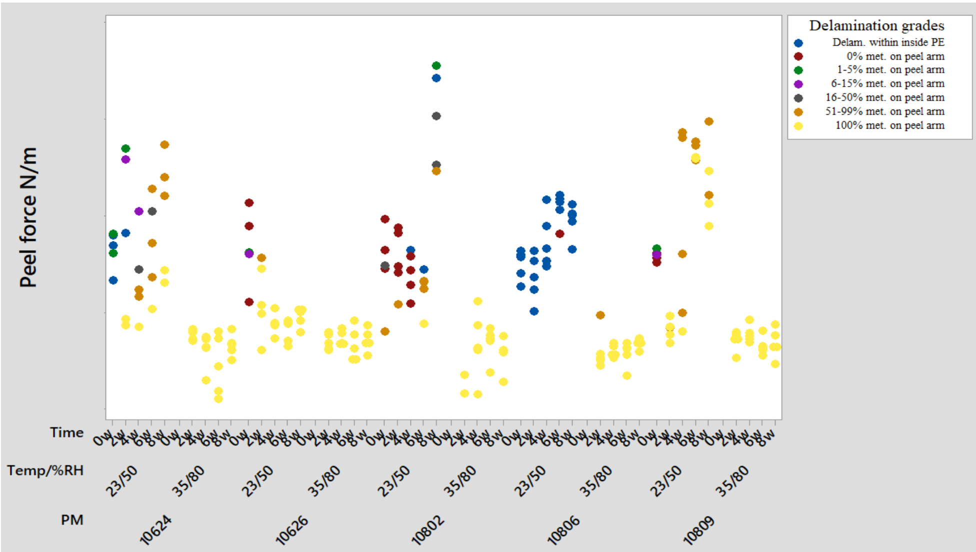



**Figure 11. Individual value plot indicating the levels of delamination in the NPM**

As shown in figure 11, the graph was plotted with Temp. (°C)/%RH, Time and PM variants on the x-axis and Peel force (N/m) on y-axis (due to confidentiality the y axis values cannot be revealed). To better visualize the distribution of the data and observe the variation, the delamination grades were grouped for each PM variant. The different colored dots represent the grades 0-6 for 5 replicates of each PM variant. From the plot it can be seen that grade 6 (yellow dots) which represents 100% metallization on peel arm, is the most common grade obtained for the variants especially at 35°/80%. Grade 0 (blue dots) that represents delamination within the inside layer was obtained mostly for the variant PM 10806 at 23°/50%. PM 10802 at 23°/50% had more of grade 1 (brown dots) samples, which represent 0% metallization on the peel arm. The rest of the variants had grades varying between grades 2-5. The grades correspond to the amount of metallization on the peel arm (mentioned in the legend) and the two most common ones are highlighted here: 100% delamination (grade 6) means a complete delamination where the metallized layer follows along the peel arm; 0% metallization (grade 1) occurs when there is a clear split between the layers.

### 4.1.2 Main effects plot

To understand the effect of certain parameters on the peel force, main effects plot was used (figure 12), which helps to describe the relationship between the set parameters and a continuous response. The main effects plot uses a fit regression model to evaluate the data. The set parameters for NPM were time, temperature, delamination grade and PM (only four parameters can be chosen at once) and peel force acts as the continuous response.

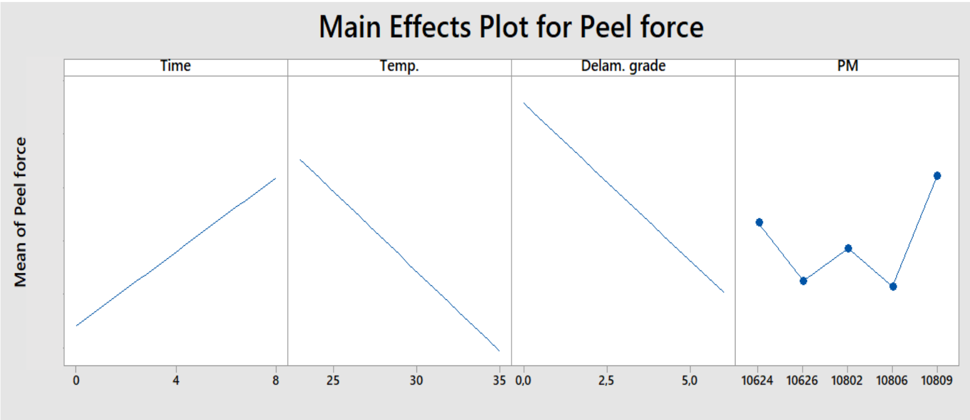



**Figure 12. Regression fit models showing the effect of four factors on mean peel force**

The evaluation of main effects plot for peel force are as follows: The mean peel force of all the PM variants increases over time (0 to 8 weeks) whereas decreases as the temperature increases from 23° to 35°. PM 10809 has the highest mean peel force and PM 10624 is the next highest with a mean peel force. PM 10626 and 10806 have very similar mean peal force values. Overall, PM 10802 has an average mean peel force. In terms of delamination grade, as the grade reaches 6, the mean peel force decreases. At 23°/50%, the delamination grades for the variants vary from 0-6 over time but at 35°/80%, the grade was noted to be 6 which remained constant for all the variants over time. These grade values influence the analysis of other values of the variants obtained at 23°/50%. Thus, makes it difficult to comment on the overall trend between peel force and delamination grade.

### 4.1.3 ANOVA

ANOVA is similar to main effects plot, used to observe the variance in the data. The analysis uses the statistical method of least squares which is a type of regression analysis. Similar to main effect plot analysis, to perform ANOVA, set and continuous parameters are required. The main effects plot obtained by performing ANOVA is shown in figure 13.

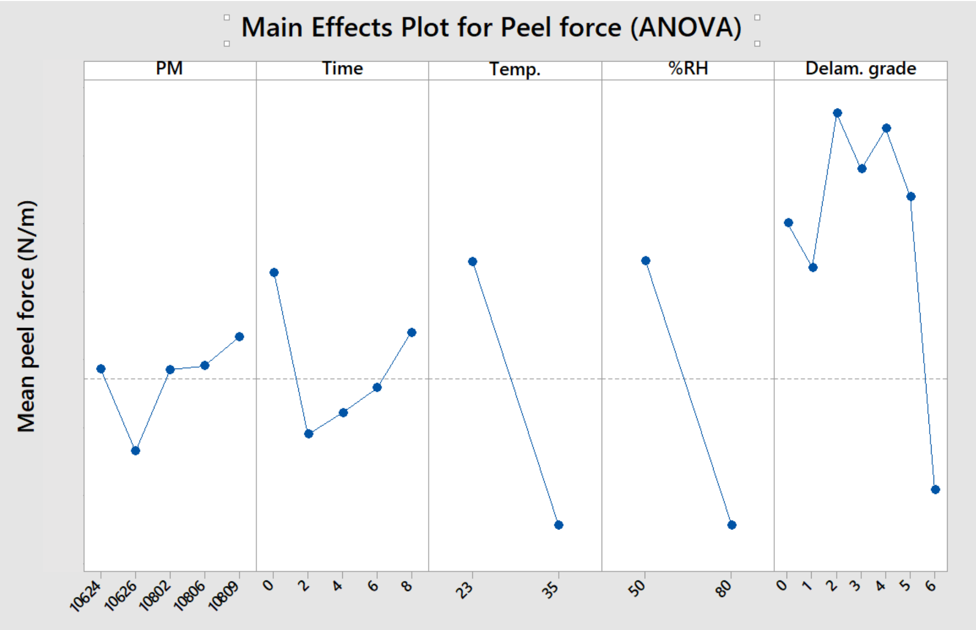



**Figure 13. Regression fit models (ANOVA) showing the main effects of five factors on mean peel force**

The dotted horizontal line denotes the reference mean value that connects all the parameters. The reference mean value is also called as response mean value. A mean value is set to observe how the data values have an effect on the parameters. The points below the reference line (response mean) are considered to be ‘outliers.’ Also, the outliers are the points which can have a main effect on the whole group of data and affect the overall results.

Although the plot is similar to the plot shown in figure 12, ANOVA is useful to check if and how the data varies from the reference mean value. Further helps to understand the trend of the parameters with respect to the peel force values. The evaluation of the plot is as follows: PM 10626 is the only outlier and has the lowest mean peel force when compared to the rest of the PM variants. The other PM variants have almost similar mean peel force values. With respect to time, the mean peel force value is the highest at 0 week (measured at 23$^{o}$/50%). It can be clearly seen that after 2 weeks of storing the samples at 23$^{o}$/50%, the mean peel force value is low and is an outlier. The points at 4 and 6 weeks also are outliers. This means that the values measured at 2, 4, 6 has the least mean peel force values and further has an impact on the overall data obtained. Additionally, it also shows that the temp/%RH has an impact on the material. The effect of temperature and RH are plotted in a linear line, which indicates that with the increase of these parameters, the mean values decrease. The steepness of the line denotes the magnitude of the effect. This means that the mean peel force values are affected by temp./RH have to a greater magnitude. A significant outlier can be observed in delamination grade which is grade 6. This signifies that grade 6 has a main effect on the whole group of data. The lowest mean values are for grade 6 when compared to the other grades where the mean values are much higher. However, as grade 6 has a main effect it has an impact on the analysis of other values. This plot makes it clear that the mean values do have an impact from the various factors and the trend cannot be commented on because of the effect caused by the outliers. The most probable way to see the effects in depth is to create an interaction plot and check the effects. However, this plot usually has more than single error while calculating the data unlike main effects plot of regression fit model.

### 4.1.4 Further discussion

Addressing the research questions mentioned in section 4.1, a few observations were made. The different amount of metallization on the peel arm varies depending on the peel force. The peel force mainly depends on the thickness of the material and is a direct function of thickness of the polymer i.e., the peel force increases as the thickness increases. There is also a direct influence of temperature on the peel force (Ashter, 2014). Sometimes the peel angle can also have an effect on the peel force i.e., as the peel angle increases, the peel force decreases. Delamination is the common form of failure that occurs in laminates and often affected by different temperatures, moisture and thickness of the material (Awalekar et al., 2018).

When the material absorbs moisture, the chemical bonds in the adhesive degrade (Awalekar et al., 2018). This means that the barrier layers used must have good barrier properties and not allow moisture to pass through the material. Further this relates to the type of coating blend used and their barrier properties which means that in NPM, the adhesion properties vary depending on the blend type used. According to the results obtained, PM 10802 (blend type B) had clear split for most replicates even after storing them up to 8 weeks at 23$^{o}$/50%. PM 10806 (blend type A) mostly had delamination within PE or partial mixed delamination where tiny spots of metallized aluminum were spotted on the peel arm along with PE. This infers that blend type B which has two layers of the same component performed better in terms of the barrier properties and adhesion when compared to the blend A which is a mix of two components.

The rest of the variants did not have clear split and had metallization on the peel arm. However, over 8 weeks it was observed that the temperature and RH had an effect on the material. Regardless of the blend type used, at 35$^{o}$/80% all the samples had complete delamination (grade 6). Overall, just by performing adhesion test, it is difficult to point where the split occurs and, in such cases, scanning electron microscopy (SEM) analysis is recommended to get a clear observation of the laminates.

## 4.2 OTR test

The OTR test was performed on flat samples at different temperatures to measure the amount of oxygen that passes through the samples. The results were recorded and evaluated using Minitab. Table 7 shows the OTR values for all five variants of NPM along with the OTR value of the reference material (with Al foil). Due to confidentiality, the reference values at 23/50% and 23/80% are indicated as x and y respectively. There are no reference values at 23/80%.

**Table 7. OTR results of NPM along with reference values**

| PM | Coating | OTR (cc/m²/24h) | | |
|---|---|---|---|---|
| | | 23º/50% | 23º/80% | 35º/80% |
| **Reference** | - | x | y | - |
| **10624** | Blend A | 8x | 8y | 2(8y) |
| **10626** | Blend A | 5x | 5y | 2(5y) |
| **10806** | Blend A | 2x | 2y | 2(2y) |
| **10802** | Blend B | 2x | 1.5y | 1.6(1.5y) |
| **10809** | Blend B | 3x | 3y | 1.5(3y) |

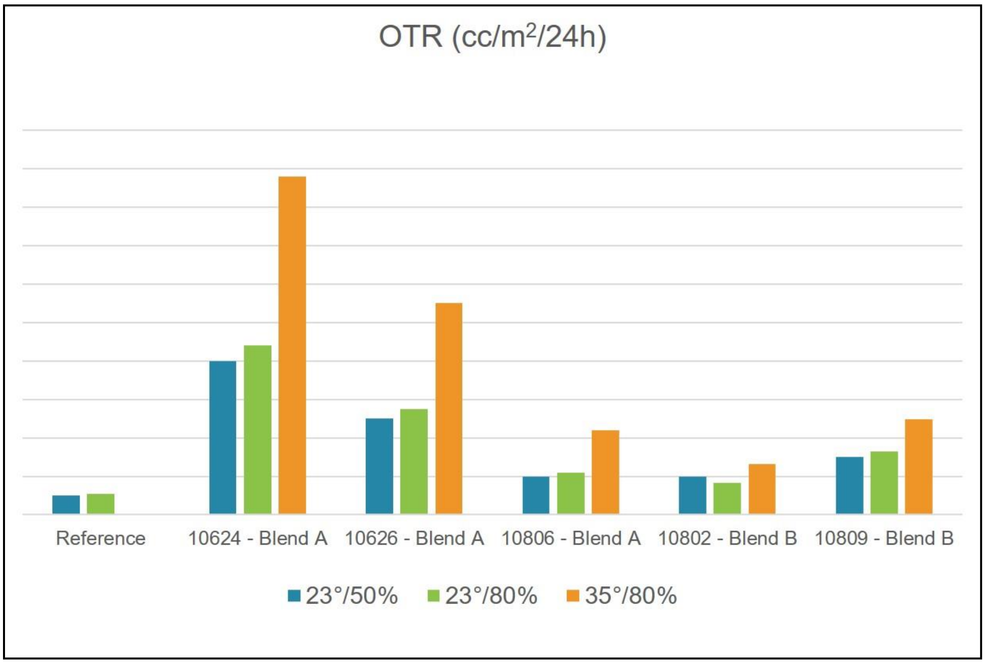



**Figure 14. Bar graph showing the comparison of OTR values between the 5 variants and reference at different temp./RH**

The packaging material must be able to provide an adequate oxygen barrier and must be able to maintain a low oxygen concentration inside the packages. The evaluation (figure 14) showed that the OTR values at 23/50 and 23/80 for NPM variants were higher (ranging between 1.5-8 times) than the values of the reference material. PM 10624 was almost 8 times higher than the reference value indicating poor oxygen barrier. The values of PM 10802 and

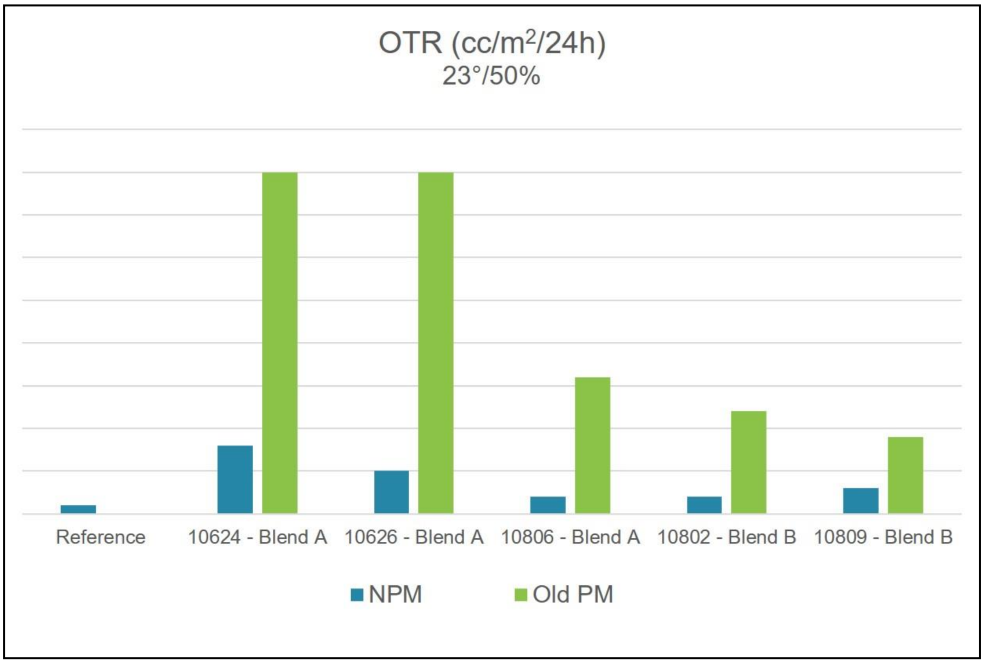
10806 are comparatively closer to the reference OTR values. They are approximately 2 times higher than the reference values which is better when compared to the other OTR values.

Since there is no reference value for 35/80, it was compared to the OTR values of NPM obtained at 23/80. As shown in the table 7 all the values at 35/80 were doubled meaning that it is two times higher than the values obtained at 23/80. This further means that the increase in temperature had an effect on the material indicating poor oxygen barrier at higher temperature. According to the research and experiments performed, Tetra Pak, the confidential reports stated that the OTR in real environment might be three times higher than the OTR in laboratory conditions.

To check if the obtained OTR values of NPM meet the requirements, it was also compared to a reference material with reduced Al content. Tetra Pak uses the reference values of previously tested NPM material which is shown in table 8. The values were normalized by considering the OTR values of the NPM which are mentioned in table 7 in terms of x and y.

**Table 8. OTR values of reference material with reduced Al content**

| PM | Material structure | OTR (cc/m$^2$/24h) | | |
|---|---|---|---|---|
| | | 23$^o$/50% | 23$^o$/80% | 35$^o$/80% |
| **10624** | Paper but different inside | 5(8x) | 5(8y) | - |
| **10626** | Paper but different inside | 8(5x) | 9(5y) | - |
| **10806** | Paper but different inside | 8(2x) | 8(2y) | - |
| **10802** | Same material | 6(2x) | 7(1.5y) | - |
| **10809** | Same material | 3(3x) | 4(3y) | - |



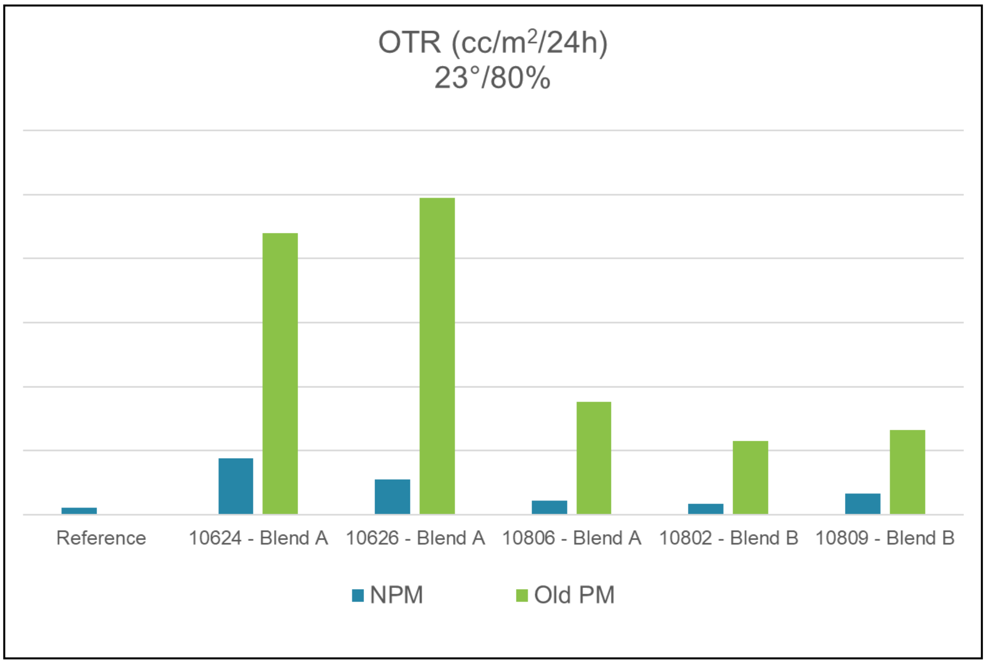




**Figure 15. Bar graph showing the comparison of OTR values between NPM, Old PM and reference**

The comparison of old PM and NPM is shown in figure 15. The OTR values of the variants obtained previously are much more higher when compared to the values obtained in the recent tests (23/50, 23/80). This clearly shows an improvement in the oxygen barrier properties of the material and indicates that the oxygen barrier of the NPM variants is better than the traditional PM variants. However, there were no reference values at 35/80 so they haven't been compared here.

There are various factors affecting the OTR of the material: The thickness of the barrier layer i.e., thicker the barrier, better the barrier; base surface compatibility i.e., the physical and chemical characteristics of the base surface after metallization have a major effect on the OTR; Even though having coating layers can be advantageous in terms of improving OTR of the material, most of the times they are not coated equally which might lead to poor barrier properties. (*Polyprint- OTR*, n.d.)

Different types of coating exhibit different chemical characteristics that play an important role in providing good barrier properties. Sometimes the coating layer alone exhibits good barrier against oxygen but will need to be laminated or coextruded along with a layer that provides moisture barrier and mechanical properties. The new packaging materials with reduced Al content, reduced plastic and increased amount of fiber, have an increased tendency to absorb moisture and oxygen especially when stored at higher temperature and moisture levels for longer period of time.

## 4.3 Weight loss test

The weight loss test was performed to check the amount of water vapor that passes through the NPM material flat samples. The system linked to the instrument records the weight loss of the samples over a period of time at very high temperature and RH (50$^{o}$C/95%). The results are obtained in an excel sheet along with calculations of average values and standard deviation. The reference value mentioned in table 9 are of the material with Al foil and is denoted as 'z'. The values obtained for NPM are normalized in terms of the value 'z'.

**Table 9. Weight loss test results of NPM**

| PM | Coating | WVTR ($g/m^2/24h$) |
|---|---|---|
| | | 50/95% |
| **Reference** | - | z |
| **10624** | Blend A | 8.5z |
| **10626** | Blend A | 8z |
| **10806** | Blend A | 8z |
| **10802** | Blend B | 6z |
| **10809** | Blend B | 8z |

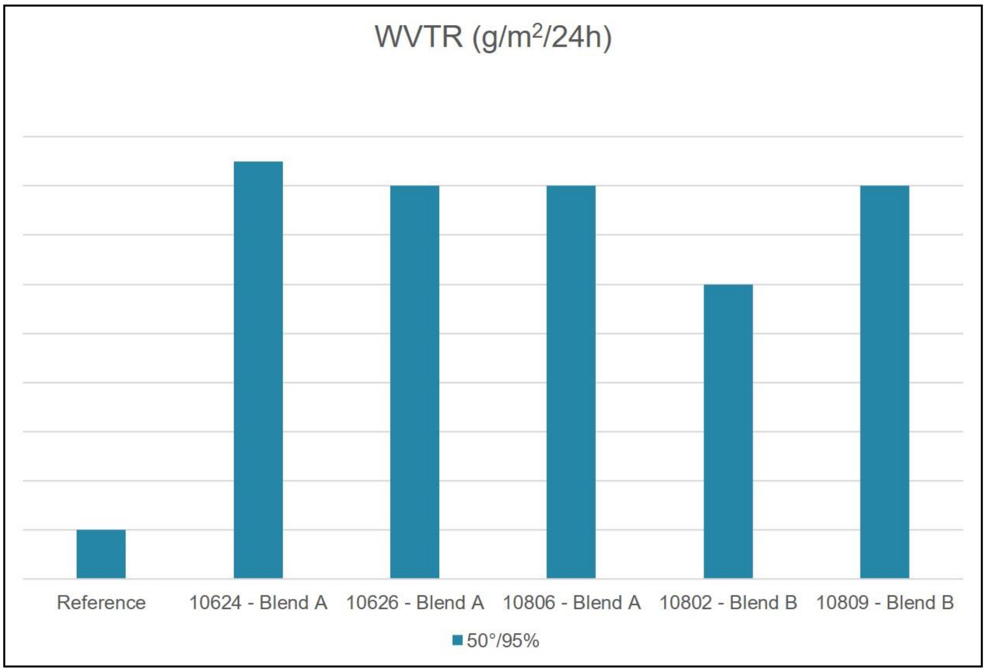



**Figure 16. Bar graph showing the comparison of WVTR values between 5 variants and reference**

As shown in the figure above, the weight loss value obtained for NPM material was 6-8 times higher than the reference value. Lower value refers that the water transmission rate through the material is less which means better moisture protection. In NPM, all variants have higher weight loss and the factor that majorly affects high WVTR is the material used. In NPM, paper can have an effect on the water transmission rates; or process conditions can have an effect on the material and its moisture absorption levels; or the instrumental error also plays a role in obtaining higher values (*Polyprint- WVTR*, n.d.). Similar to OTR, the values obtained for NPM were compared to the weight loss values of the reference material with less Al content (table 10). These reference values are obtained from previous weight loss tests performed on NPM.

**Table 10. Weight loss values of reference material with less Al content**

| **PM** | **WVTR ($g/m^2/24h$)** |
|---|---|
| | **50º/95%** |
| **10624** | 0.12z |
| **10626** | 0.14z |
| **10806** | 0.07z |
| **10802** | 0.12z |
| **10809** | 0.1z |

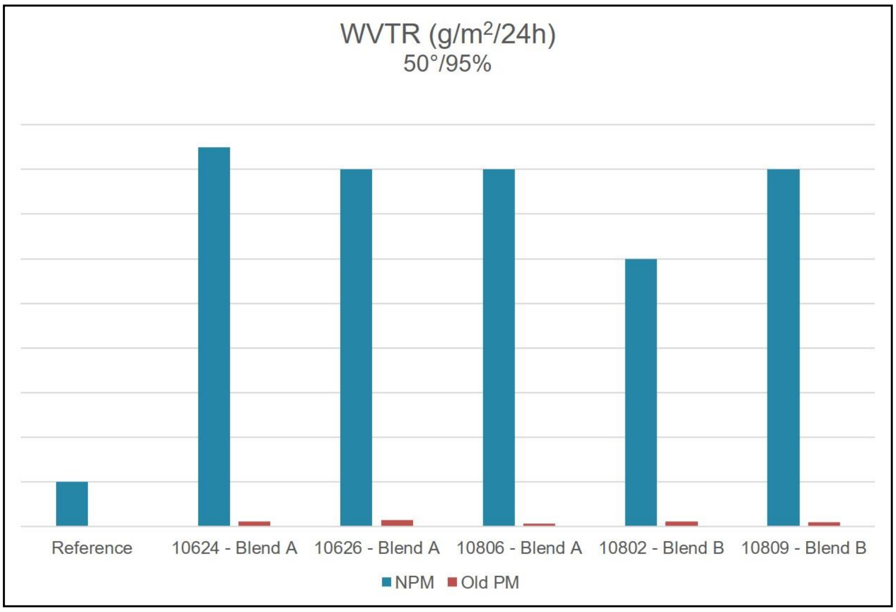



**Figure 17. Bar graph showing the comparison of OTR values between NPM, Old PM and reference**

Overall, all the old PM variants had lower WVTR when compared to the values of NPM and this can be observed in figure 17. In fact, the values are lower than value of the reference material with Al foil. Lower values indicate a good water vapor barrier which means that all the variants here have a really good water vapor barrier. However, the current variants used have very high values of WVTR which signify that they are not acting as good water vapor barriers. When compared amongst the values of NPM, PM 10802 still has a lower value. It would be difficult to comment on the reason behind obtaining the high values without performing further analyses.

However, as mentioned earlier, the type of coating and the thickness of it plays an important role in providing good barrier properties. This means that the type of coating used must provide a good moisture barrier. Either the barrier or the coatings used in NPM could be the factors for obtaining higher values indicating high moisture absorption.

The conditions maintained for the test was 50$^{o}$/95% and this was to create an environment of hot and highly humid climate. The results obtained at these conditions might not imply on the real environmental conditions. This makes it difficult to comment on how the material would behave at lower temperatures and humidity levels. However, higher values indicate that there might be chances of cracks and bursts in the paper or paperboard which might affect the overall packaging performance (Tutak et al., 2019).

## 4.4 Comparison to prior studies

The key findings of adhesion, OTR and WVTR tests have been mentioned in the above sections. The comparison of the key findings obtained from the experimental analysis and from the theoretical background are discussed here. For the adhesion test, there was limited theory obtained from the literature and the results here are discussed with respect to the findings obtained from company project background. The previous tests on NPM had revealed different amount of metallization on the peel arm but there was no qualitative analysis performed. There was also no report stating the delamination of the samples. The methods followed for the NPM consisted of qualitative analysis in the form of a grading system. This analysis combined with the quantitative analysis helped to analyze the results better statistically. Having grades made it easier to comment on the delamination of the samples. Further comparisons could not be done as there was no data (peel force values) available for the same variants. Also, the impact of moisture and temperature on adhesion was specific to this study. Hence, no comparisons could be made.

With respect to OTR test, the previous tests on NPM had shown high oxygen transmission rates. From the theory, the OTR of Al foil was less than 0.006 $cc/m^2$. When these rates were compared to the current results, OTR values are higher than the value of Al foil. But when compared to the previous results of NPM, the rates have decreased. This means that the current variants behave as better oxygen barriers. According to the literature, since there were no tests performed at the same environmental conditions, the exact values could not be compared. But comparison of OTR values of the metallized films, the values obtained for the metallized paper were quite closer.

For the WVTR, the previous results showed very low WVTR. From the literature, it was found that Al has WVTR of 0.3 $g/m^2$. The WVTR of metallized films also had low values when compared to the metallized paper used for NPM. The WVTR of the current variants were much higher than the values obtained from both literature and previous work. The values from the previous studies were lower than the reference. The reason for this must be investigated further.

# 5 Conclusion and future research recommendations

*This section concludes the thesis work by highlighting the objectives of the project and the conclusions obtained from the results and discussion. Further, it also presents the research limitations, future research recommendations and research implications of the project.*

## 5.1 Conclusions

As presented in Chapter 1, the three objectives of the thesis work are:

1. The first objective is to find out if moisture sensitivity and adhesion mechanisms affect the adhesion measurements by performing quantitative measurements.
2. The second objective is to understand how the influence of different moisture and temperature levels over time affect the adhesion between metallized paper and polymer layers by performing qualitative analysis.
3. The third objective is to understand how the influence of different moisture and temperature levels over time affect oxygen transmission rate (OTR) and water vapor transmission rate (WVTR) of the NPM.

All three objectives are linked to studying the influence of moisture and temperature on the key parameters of the material. For a better understanding of the parameters and tests performed, the literature research gave adequate knowledge. Since the literature provided limited information about the studied phenomena, it made it quite difficult to understand certain material properties and the influence of moisture and temperature on them. However, the background of the project obtained from Tetra Pak helped to set the objectives of the thesis work. The research questions which are discussed in the Chapter 4 were framed based on the previous work done on similar material.

To address the first two objectives, adhesion test was performed. As mentioned earlier the importance of performing the test was to check the influence of moisture and temperature. This was achieved by following a standard protocol and storing the samples for 8 weeks at set temperatures. The evaluation of adhesion test results indicated that there was an influence of different moisture and temperature levels especially at higher temperature and RH on the adhesion measurements. Further, this signifies that over time these factors have an effect on the layers in the material. In NPM, the effect is on the adhesion between the metallized layer and polymer layer. These factors can lead to changes in the material properties, especially the barrier properties. Overall, it was a clear observation that the PM 10802 with blend B coating had a better performance compared to the other variants during the adhesion tests. From this, it can be inferred that the coating also plays an important role in enhancing the barrier properties.

The third objective completely focused on testing the material for its barrier properties against oxygen and water through OTR and WVTR tests. The OTR tests were performed at different temperatures to compare the material performance in different environmental conditions. At higher temperature and RH (35/80), there were no reference values. But they were compared to the values obtained at 23/80. The results clearly showed that the OTR values at 35/80 were double the values at 23/80 indicating the influence of higher temperature. Overall PM 10802 had closer values to the reference making it the variant with a good oxygen barrier. However, it is important to note that the oxygen barrier of NPM has improved when compared to the reference values of previous OTR test on NPM.

With respect to WVTR, the temperature and moisture had a very high influence. The values obtained were very high compared to the reference values indicating a poor water barrier of the material. But as mentioned earlier the conditions set (50ºC/95%) were to imitate a hot and humid climate. But it is too high, and it does not exactly imitate the real environment conditions. PM 10802 has the lowest WVTR compared to the other variants. However, the values were at least 6-8 times higher than the reference values. This can also mean that even at slightly lower temperatures, the WVTR could still be higher than the reference values.

Although the WVTR was high for PM 10802, the overall evaluation shows that it has better barrier properties when compared to the other variants. But the variant certainly needs enhancement in terms of providing a good water vapor barrier. The test results of adhesion also showed that the variant 10802 performed better than the other variants. So, in conclusion, the variant 10802 performed better than the other variants and with improvements it could potentially have a good packaging performance.

## 5.2 Research limitations

Limited literature studies on this material were an obstacle in being able to validate the results. It also limited the knowledge on the material and its properties. As the project is also about studying the influence of moisture and temperature on the NPM, there were only a few studies available on other materials but those were not similar to NPM. Not having enough literature limited the study on the NPM's behavior. All the variants had similar barriers used and there was no reference material tested. Further, due to limited time, only a few tests and analyses could be performed on the material. This limitation makes it difficult to answer all research questions. For the adhesion test, limited statistical analysis was done which is a limitation when understanding the material behavior. Also, the results were not compared to any previous data or established theory, making it quite difficult to validate the results. While for OTR and WVTR, some of the reference values were not available. Only weight loss test was performed to check the WVTR and the values obtained were really high. The high temperature and moisture used in weight loss test can also be a limitation as it does not represent the real environment. There was no standard WVTR test performed to compare and validate the results. Since the project is in the development stage, continuous changes might lead to different results. This often makes it difficult to compare the results of NPM. Although ANOVA gave better perspective in terms of the effects, it can have multiple errors when compared to the regression fit plot which usually has only single error. This might lead to inaccurate results.

## 5.3 Future research recommendations

Firstly, with respect to the material structure, few improvements or changes can be considered. More types of paper having good barrier properties can be researched on. With respect to paper, the influence of its thickness can be studied, as this is an important parameter that affects the properties of the material. Apart from the type of paper, the coatings also have an influence on the barrier properties. There are many other different types of barrier coatings that perform better than the ones used currently in the NPM structure. These coatings can be explored.

For the adhesion test, the material was tested only up to 8 weeks due to the time constraint. But the material could be tested for more time depending on the shelf-life requirements of the material. The possible errors of the adhesion test can be avoided by having an instrument near the storage area or the vice versa so that the measurements are more accurate. To understand the data better, more statistical analysis should be performed by focusing on certain parameters such as the delamination grade and influence of time. To be able to check where the split can occur and why it occurs, an in-depth analysis using scanning electron microscope (SEM) could be performed. SEM helps to visualize the structure better and also to point out where the split occurs.

With respect to the OTR tests, there were no reference values at 35/80 in the project which made it difficult to compare and comment. But as part of this thesis work the OTR values were measured at 35/80 and this can serve as a future reference. For the WVTR, along with weight loss test a standard WVTR test can be performed to compare the values obtained. The WVTR standard test can be performed at different temperatures which would be advantageous when compared to the weight loss method. The only limitation for performing the standard WVTR test as part of this thesis work was because the sample would take more time to reach equilibrium. This would make it difficult to obtain and evaluate the results within the given time. Further, measuring OTR and WVTR for filled packages at different conditions can be useful in predicting the shelf life of the NPM.

Most of the thesis work concentrates on studying the influence of moisture and temperature on the different parameters. But to evaluate and discuss the results more, it would also be helpful to study additional factors affecting the parameters. The other factors that can be studied are the influence of the paper; the product influence once the packages are filled; the thickness of the layers; barrier coatings and their chemical characteristics. As mentioned in the conclusion, overall PM 10802 has a better performance when compared to the other variants. The above mentioned recommendations can be applied to this material structure which will help to improve the packaging performance of this variant.

## 5.4 Research implications

Implications addresses results, conclusions and future recommendations altogether. The effect that this research might have on future research or on similar field of work can be studied. All the results and key findings have a significance in their own ways and might have implications. The implications of this research work are categorized into three types: theoretical, practical and social.

### 5.4.1 Theoretical implications

The theoretical implications of the findings are relevant to increase the understanding about the influence of moisture and temperature on the barrier properties and adhesion of carton-based laminates. A key finding of the study regarding the OTR and WVTR of Al foil showed that it is a very good oxygen and water barrier. But there were no studies on metallized paper or its barrier properties. A few studies explained the influence of different temp./RH levels on the material but not exactly on the above properties. In general, the key finding was that with influence of temp./RH, there are chances that OTR and WVTR might be high. But in terms of adhesion there was no literature found indicating the influence. However, due to this limitation, the findings could not be compared to them. The other findings which were used in this study were obtained from Tetra Pak based on the previous studies. It was observed in the adhesion test (between the metallized and polymer layers) that there was metallization on the peel arm. Even though it was thought that the temp./RH might have an influence, it is quite challenging to know the exact reason behind this phenomenon.

Overall, this study contributes insights about the adhesion between metallized paper and polymers along with insights about the influence of temperature and humidity on OTR and WVTR of carton-polymer laminates (with metalized paper).

### 5.4.2 Practical implications

The practical implications here refer to the applicability of the findings to the NPM. In NPM, the material structure is different from the other aseptic cartons, especially the barrier used. From the findings, it can be seen that there was a clear influence of moisture and temperature on the three tests of

NPM. In adhesion test, it was observed that the mean peel force values decreased with increase in temp./RH. In the OTR test a clear increase in the rates were observed when the temp./RH increased. Although a clear influence was observed, further analysis of the results can explain the influence in more depth. This further will establish theory on how the material behaves when influenced by different temp./RH. Even though these findings cannot be verified to any literature due to the limitation in the literature findings, it can further be used as a base into a theory explaining the effect of temp./RH on adhesion, and especially on barrier properties. PM 10802 is a promising structure that Tetra Pak will develop further to increase the water vapor barrier.

### 5.4.3 Social implications

The social implications of this study refer to the vision and goals of Tetra Pak i.e., to 'Protect what's good' and reduce the carbon footprint of the future carton. To protect the product, the packaging must have good barrier and mechanical properties and must resist different temp./RH. Even when a new sustainable packaging material is used, it is important to test this material to ensure that it protects the product. Out of all the variants, PM 10802 behaved as a good oxygen barrier. Even though PM 10802 had better WVTR than other variants, this variant definitely needs improvements to perform as a good water barrier.

To be able to reduce the carbon footprint, Tetra Pak aims at improving the recycling of the cartons. In NPM, the peel test was performed to check if there is a clear split between the metallized and polymer layer. Without a clear split the mix of these two layers causes problems in recycling. However, to be able to comment on the recycling and conclude, further studies must be performed as it was not included in this thesis work. These studies can be performed especially on cartons made using PM 10802 which had the samples with clear split.

Overall, the further improvements stated in section 5.3 might be challenging. But achieving them will help to protect the product, while being able to lower the carbon footprint of the cartons.

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