KAUNAS UNIVERSITY OF TECHNOLOGY
MECHANICAL ENGINEERING AND DESIGN FACULTY

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AN ASSESSMENT OF GREASE PENETRATION AND DELAMINATION PROPERTIES OF MULTILAYER FLEXIBLE PACKAGING MATERIALS

Master's Degree Final Project

Supervisor
Lect. dr. **Vaidas Bivainis**

KAUNAS, 2018

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Master's Degree Final Project
Industrial Engineering and Management (621H77003)

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(Title and code of study programme)

"An Assessment of Grease Penetration and Delamination Properties of Multilayer Flexible Packaging Materials"

DECLARATION OF ACADEMIC INTEGRITY

05 January 20 18

Kaunas

I confirm that the final project of mine, **Vegesna Sunil Varma**, on the subject “An Assessment of Grease Penetration and Delamination Properties of Multilayer Flexible Packaging Materials” is written completely by myself; all the provided data and research results are correct and have been obtained honestly. None of the parts of this thesis have been plagiarized from any printed, Internet-based or otherwise recorded sources. All direct and indirect quotations from external resources are indicated in the list of references. No monetary funds (unless required by law) have been paid to anyone for any contribution to this thesis.

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Approved:

Head of
Production engineering
Department

____
(Signature, date)

Kazimieras Juzėnas
____
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MASTER STUDIES FINAL PROJECT TASK ASSIGNMENT

STUDY PROGRAMME INDUSTRIAL ENGINEERING AND MANAGEMENT

The final project of Master studies to gain the master qualification degree, is research or applied type project, for completion and defence of which 30 credits are assigned. The final project of the student must demonstrate the deepened and enlarged knowledge acquired in the main studies, also gained skills to formulate and solve an actual problem having limited and (or) contradictory information, independently conduct scientific or applied analysis and properly interpret data. By completing and defending the final project Master studies student must demonstrate the creativity, ability to apply fundamental knowledge, understanding of social and commercial environment, Legal Acts and financial possibilities, show the information search skills, ability to carry out the qualified analysis, use numerical methods, applied software, common information technologies and correct language, ability to formulate proper conclusions.

1. Title of the Project

An Assessment of Grease Penetration and Delamination Properties of Multilayer Flexible Packaging Materials

Approved by the Dean Order No.V25-11-12, 11 December 2017

2. Aim of the project

To determine rate of grease penetration on various flexible packaging materials, thermal, peel test of material and discussion of delamination properties

3. Structure of the project

Summary, literature review, grease penetration experiment, delamination properties, peel test, results and conclusions

4. Requirements and conditions

Samples of six different flexible packaging materials are required. The grease penetration experiment should be conducted in laboratory oven and 180° peel test should done on peel test machine

5. This task assignment is an integral part of the final project

6. Project submission deadline: 21 December 2017

Student _______________________ _______________________
(*Name, Surname of the Student*) (*Signature, date*)

Supervisor _______________________ _______________________
(*Position, Name, Surname*) (*Signature, date)*

Vegesna, Sunil, Varma. *Daugiasluoksnių lanksčių pakavimo medžiagų riebalų pralaidumo ir atsisluoksniavimo savybių įvertinimas:* magistro baigiamasis projektas / vadovas: lekt. dr. Vaidas Bivainis, Kauno technologijos universitetas, Mechanikos inžinerijos ir dizaino fakultetas.

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Reikšminiai žodžiai: *daugiasluoksnės pakuotės medžiagos, riebalų prasiskverbimas, daugiasluoksnės plėvelės atsisluoksniavimas, klijai, popierius, laminavimas*

Kaunas, 2018. 47 p.

SANTRAUKA

Šiuo metu įvairių maisto prekių pakavimui naudojamos daugiasluoksnės lanksčios pakavimo medžiagos. Jų gamybai naudojamos įvairiausios paskirties polimerinės ar kt. medžiagos, kurių vienos paskirtis – suteikti barjerines, geras terminio suklijavimo bei kt. reikimas savybes. Šios savybės svarbios maisto produktų pakavimui, nes pakuotės medžiaga ir jos konstrukcija turi neleisti aplinkoje esančiam deguoniui, vandens garams pažeisti supakuoto gaminio. Analogiškai supakuotas produktas ar jo sudedamosios dalys neturi prasiskverbti į pakuotės išorę. Maisto gaminiams, savo sudėtyje turintiems riebalų labai svarbi pakuotės savybė – atsparumas riebalų pralaidumui. Šias savybes užtikrina įvairios medžiagos, tai gali būti polietileno sluoksnis, tai gali būti plonas Al sluoksnis ar kt. polimerinės medžiagos.

Darbe atlikta daugiasluoksnių pakuočių medžiagų sluoksnių formavimo technologijų, savybių tyrimų bei fleksografinės spausdinimo technologijos apžvalga. Taip pat darbe atlikti maisto pramonėje naudojamų daugiasluoksnių plėvelių, skirtų spausdinti fleksografija riebalų pralaidumo tyrimai. Gauti rezultatai parodė gerą jų atsparumą riebalų pralaidumui. Ir tik medžiagos sulenkimo linijose susiformavo medžiagos defektai, kurių dėka fiksuotas riebalų prasiskverbimas.

Darbe atlikti daugiasluoksnės sviesto pakavimo medžiagos, kurios sudėtyje naudojamas vaško sluoksnis atsisluoksniavimo tyrimai. Gauti tyrimų rezultatai parodė, kad esant 40^0 C aplinkos temperatūrai fiksuotas šios pakavimo medžiagos atsisluoksniavimas. Darbo gale pateiktos išvados.

Vegesna, Sunil, Varma. *An Assessment of Grease Penetration and Delamination Properties of Multilayer Flexible Packaging Materials*. Master's Final Project / supervisor Lect. dr Vaidas Bivainis; Faculty of Mechanical Engineering and Design, Kaunas University of Technology.

Research field and area: Production Engineering, Technological sciences

Key words: *multilayer packaging materials, grease penetration, multilayer film layering, wax, paper, lamination*

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SUMMARY

At present, multilayer flexible packaging is a rapidly improving type of packaging and these materials are used in various food packaging items. Various polymers are used in the production of these materials- to provide good barrier to grease, oxygen, good thermal conductivity and other required properties etc. Grease barrier plays a key role in high-fat content products. A wide range of barrier properties is exhibited by materials, such as a polyethylene layer, a thin layer of Al or other polymer materials. They are few basic methods which determine the resistance of package to grease penetration and many methods give the best quality measure of grease penetration. Multilayer films which can be recycled are integrated with packaging and simple process is used for determining grease barrier properties of high-fat content product films and these test results of several multilayer films are presented to describe the effectiveness of these films.

Multilayer butter packaging materials are used in the work, subjected to grease penetration (rapid method), delamination and peel test. The results for show good resistance to grease penetration and only for creased materials have defects this results in grease penetration. The results of the delamination study showed that these packing material delaminates at 40° C. The results and conclusions are shown at end of the work.

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INTRODUCTION

Packaging is been part of mankind from the past by utilizing of leaves and other material to store, ship, hold and for preserving food. Later on, storage materials are persevered to change which includes wood, mud pots, woven boxes, glass, metal and paper and paperboard. The use of plastics in packaging came into consideration after the World War II and polyethylene was first among them, nevertheless remains the leading packaging material and because of the continued improvements within the field of production and use of plastics the field of packaging increased unexpectedly at some point since the 1970's [1]. Easy manufacturing of plastic materials additionally supported for the expanded use of those plastics. Combination of all these characteristics makes packaging the largest marketplace for plastics.

The fundamental capabilities of a packaging are to prevent quality deterioration, storage, and transportation of the product. At present, the packaging technology is a mixture of art, science. In past various grease containing packaging materials like metals cans, glass bottles, wax coated paper, are used for human utilization. In recent times multilayer films are used in flexible packaging to reduce material to product ratio which results in less energy consumption during the production, reduction of space while transportation and convenience to the customer. Several flexible and multilayer films are developed by using polymers especially for grease resistance like EVOH, PE, PET, PVDC, EAA depending on the desired cost of the structure.

Work aim

This research is aimed to analyze grease barrier properties of the different multilayer flexible packaging materials like PET, AL film, PP, PE, BOPP (Biaxially oriented polypropylene), OPA(Oriented polyamide) which are commonly used in butter packaging, by conducting grease penetration test (Rapid method). Delamination and peel test are done to assess delamination properties and bond strength (wax) of material.

Tasks for work aim

Multilayer flexible barrier materials creased and un-creased are subjected on one side to oil (olive) contained in weighted cotton patches. The time needed to exhibit change caused by wetting on TLC plate is measured and rate of grease penetration through materials is known. In future by use of this method, the development and selection of multilayer flexible materials suitable for grease barriers are done.

Delamination is done by heat conductivity at different temperatures (25-45°C) and estimating bond strength (wax) between two layers of flexible packaging by peel test.

LITERATURE REVIEW

1. Paper for packages

Paper is defined as a sheet made of plant fiber [2]. The main form of modern paper is cellulose from wood. It can be characterized according to its weight, moisture, thickness, brightness. When the weight of paper > 250 g/m^2. The quality of paper depends on fiber source. Longer fibers exhibit good properties like fold, tear, and puncture strength and shorter fibers will give even density over the width of the sheet and have smooth surface texture [2].

Based on market and customer needs a wide range of papers is commercially available. At stock preparation stage treatment and additives are used. Additives, surface finish, coatings are done at different stages there resulting in various types of packaging material. According to grammage thickness, there is a wide variety of paper-based products.

They are different types of paper for packaging such are greaseproof, glassine, sack kraft, vegetable parchment, label paper, impregnated and laminating papers [3].

Greaseproof: Fibers are refined at the stock preparation stage. In a beater, hydration is carried out in a batch process so that the fibers will be in smooth. Grammage range is 30-70 g/m^2.

Glassine: This paper exhibits high smoothness and glassy finish. It can be laminated to paperboard and it is non-porous. Glycerin can be used for plasticizing. It is in plain and dark brown in color and grammage range is 30-80 g/m^2.

1.2 Multilayer Flexible Packaging

Materials for packaging is commonly classified as monolayer materials and multilayer materials. Films, foils, and paper comes under monolayer materials whereas coated substrates, co-extrusion and laminates refer multilayers. From a variety of standard and new material, a particular product is chosen. Drawbacks of one material restricted by the presence of another material [4].

Co-extrusion, lamination, coatings and other several methods used to manufacture these multilayer packaging materials and these are majorly used in flexible packaging industry. According to thickness, coating, laminating materials and other property requirements like the barrier to gases, moisture and printing decisions are made whether to coat, co-extruded and laminate [4]. Combining different materials can also be economical than using only monolayer structures for suppose if the paper is coated or laminated with plastic films it gains resistant to gases and moisture.

Multilayered packaging structures are generally indicated in both types (verbally or graphically). In the graphical show, each line shows layer and in verbal it is separated by forwarding slash (-). A structure can also be defined as the class and generic levels.

Class: Film/Adhesive/Foil/Adhesive/Film

Generic: AL6/ PE10/ PAP35/PE15 MATT

1.3 Flexography

It is introduced at the beginning of 1950 and called as anilox printing. The other name for Flexography is *'Flexo'* due to the use of flexible plates. It is an impression based process used to print any material including plastic, foil, acetate films, brown paper, and metals etc. which delivers high-end graphics to the substrate [1, 3].

It is important printing method in flexible packaging. The printing process can be done with rapid speed and low cost. In flexo printing the plates used are flexible made from photochemical or photopolymer plates that are thin plastic or made from rubber and attached to printing rollers and after the introduction photopolymer plates flexographic printing is vastly used. Ink is picked by cavitated anilox roll which is transferred to printing plates and it is printed to the substrate [1, 3].

Inks Used

The main properties of flexography inks are drying and have low viscosity consists of colorants. They are two types of inks used solvent based, water based. These solvents mainly consist of alcohol, glycol, and ammonia as the main solvent which helps in the emission of Volatile Organic Compounds helps in fast drying. U-V curable inks increases the image quality and it is commonly used. In recent times the use of the inks are rapidly high but printing equipment cost is relatively high. Some of the Solvent-based inks are polyvinyl chloride (PVC) polyvinyl butyral (PVB) and Nitrocellulose (NC) [1].

Nitrocellulose inks are used for simple laminates which shows good printability with high water resistance with the heat seal. Combination of nitrocellulose and polyurethane improves lamination bond strength for pasteurization.

For high-quality laminate polyvinyl butyral (PVB) and polyvinyl chloride (PVC) are used and due to its different properties and limitations. For many varieties of flexible packaging films, PVB is used for good adhesion as in there another case to gain high resistance in sterilization process PVC inks are used on PET and PA films [1].

Figure 1.1 shows a schematic of a Flexographic printing set up.

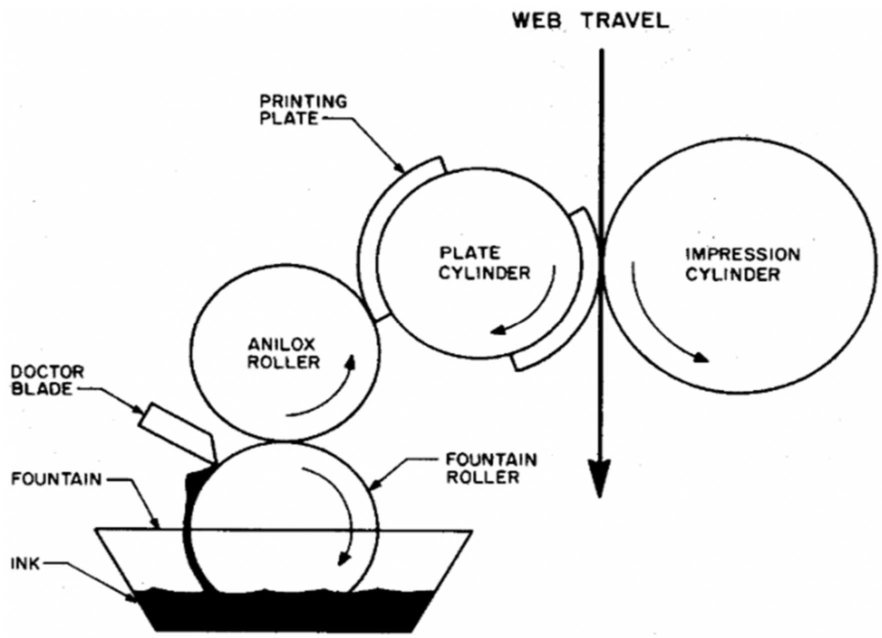


Figure 1. 1 Flexographic printing process [11]

Substrate Specification	**Importance of printing**
Surface strength	+
Smoothness	+++
Moisture	++
Water resistance	++
Surface chemistry	++
Liquid absorption speed	++
Liquid absorption capacity	++

Table 1. 1 Basic paper specification for flexography **(+++)** - High importance [1]

2. MATERIALS

The packaging materials are majorly divided into rigid, semi-rigid and flexible materials. Materials like paper and paperboard, metal and glass containers are widely used. Most economical packaging materials are paper and paperboard and for good barrier properties, strength and rigidity metals are used whereas when it comes to glass the main disadvantage is breakage due to transportation but it gives the product a good look and exhibits good barrier properties [3].

Glass

Glass which is used commercially are based on silica and it is represented by chemical formula SIOx. Mostly soda-lime glass is used in glass packaging which is made up of silica, sodium and calcium compounds. Glass can be melted in high temperatures and cooled quickly so it becomes solid in a transparency condition. Transition metal compounds are added to give color to glass and the main drawback for glass packing is weight and it breaks easily [2].

Glass packaging is mainly utilized to store reactive chemicals, carbonated beverages and can withhold high temperatures also for retort-able products. Retorting is about packing hot filling and pressure to kill microorganisms. Main advantage of glass packing is chemically inactive, tasteless and odorless [2].

Metals

Among all rigid containers, Metal cans are of 60% used in the USA for beer and food beverages [3].Aluminum and steel are main metals used in packaging industry. Beer and soft drink segments dominate aluminum cans while steel can dominate food industry. These metal can be used in very thin structures by which it exhibits lightweight and shows good ductility and strength properties [4].

Foils

Very thin sheet metals like Tin, Steel, and Aluminum are known as foils. The word foil generally refers to aluminum foils and Tin, Aluminum foils are not importantly used in packaging. Thickness ranges from 26 gauges to around 700 gauge (6.5 μm to around 180 μm). In a large variety of packaging, Aluminum is used and it provides a barrier to gases, flavor loss, oxygen, and good heat conductivity and gives an attractive look to the package. The main drawback of aluminum foil is due to formation pinholes and to stop these pinholes multilayers of foils should be combined due to this moisture barrier, other barrier properties can be achieved and also by laminating it to a plastic [4].

2.1 Films for packaging

Flexible packaging is accepted by consumers because it is very easy to dispose of and it takes less space in waste these factors makes it fastest growing in technology.

A thin sheet of plastic can be defined as films and classification of sheet and film differs according to their thickness which relates flexibility. Films are less 0.0762 mm and thicknesses more than 0.0762 mm represents sheet [1].

Polymers are the generic term for plastic and these the classified as crystalline, semi-crystalline or amorphous depending on the molecular chains orientation. These molecular chains are orderly fashion in crystalline polymers for amorphous polymers they are randomly oriented. For most varieties of products like bags, cups, bottles are made from plastic films. By flat die extrusion, solution casting, calendaring films are made. Depending on the resin characteristics and the desired film properties blown-film extrusion is done.

The combination of two or more films can be describe the composite structure and it is made by applying adhesive and heat it is called as laminates. Co-extruded structures are formed by extruding multiple layers together [4]. Polystyrene, Polypropylene, Ethylene copolymers, Polyethylene, Polyester, Polyvinyl chloride, Polyamide and polycarbonate films are majorly used.

Polyamide (PA)

In flexible packaging Polyamide is largely used for putrescible or perishable food due to its peculiar properties. This films may be processed by cast and blown film process. To gain abilities like stability in dimensions, ready to-use-film of high-quality, post treatment by annealing and humidification is done. About 260000 tons of polyamide is consumed worldwide in the year 2002 as shown in figure 2.1 synthetic polymers like PA66 (nylon).

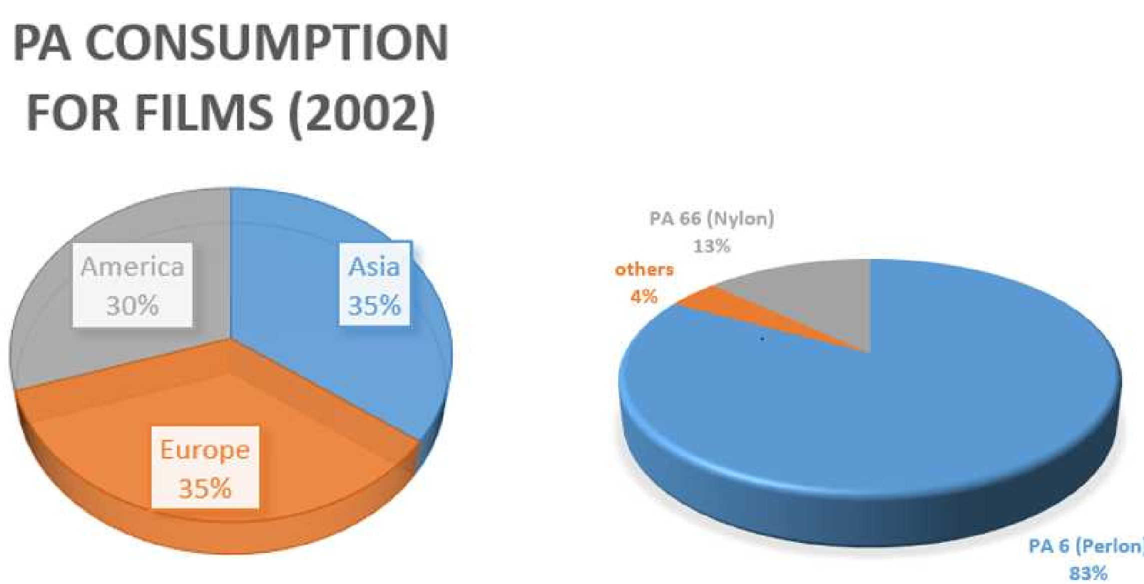


Figure 2. 1 Polyamide consumption for films in 2002 [17]

Polyester (PET)

Both Ethylene glycol and Terephthalic acid react to form a high-performance PET film. Here the by-product is water for this reaction so it is called condensation. It is linear thermoplastic material and exhibits high tensile strength, toughness, stiffness, chemical resistance, clarity and some other barrier properties. Alignment of the film can be done by blown-extrusion process thereby increasing its properties [2, 4].

To improve heat resistance addition of nucleating agents and heat treatments can be done to PET films and which also achieves a higher level of crystallinity.

Polyvinyl Chloride (PVC)

It is majorly used in sheet forms for thermoformed materials. Thermoforming is defined as heating the polymer above its Glass transition temperature and pressing it over a mold with application of air and vacuum. PVC is characterized as rigid plastic but can be made into soft unbreakable or in a flexible film form with the addition of some chemicals known as plasticizers. PVC exhibits resistant to oils, grease and has adequate barrier properties which are ideally used in packing meat, fish sausages also widely used in pharmaceutical industry [2].

Ethylene Copolymers

Ethylene polymers like (EAA) and (EMAA) are the sealants used to increase seal-ability and strength. Thermos plastic material is heated above a certain temperature and cooled at room temperature to achieve seal-ability. The molecular chains undergo segmental motion at a certain temperature this is term as glass transition temperature, Tg. Polymer heat seal is always above the Tg of that polymer. (EVOH) is used for inside coatings and to make a multilayer structure which improves moisture and gas barrier properties. To improve adherence and heat seal-ability Ethylene vinyl acetate (EVA) is combined with other plastics [2].

The least expensive material in packaging is polyethylene. Density varies from 0.910-0.965 g/cm^3 and has different classifications as Low-Density Polyethylene (LDPE) and High-Density Polyethylene (HDPE). For good moisture, odorless and tasteless properties LDPE is used. For inks and adhesives, LDPE is subjected to surface treatments [1]. It is used in meat packaging and combined with other packages to increase the barrier properties to some packaging applications. HDPE is the most rigid and least clear in PE films and exhibits high moisture barrier, strength, and better chemical resistance it is used in milk, crackers, cereal bags and other packaging [2].

Polypropylene (PP)

In the field of packaging, Polypropylene is majorly used. If it is compared to PE films PP films exhibit higher temperature and better water vapor barrier properties. PP becomes fragile at freezing temperatures, but alignment is proven to reduce this problem. The seal range is limited and should a have close inspection of a packaging line. To achieve printability, oxidization, and applications of adhesives the film must be surface treated. Slip additives are added to reduce Coefficient of friction. The range of heat seal, slip can be upgraded by coating the film acrylic anther coatings. Oriented Polypropylene films (OPP) is metalized to gain good barrier properties like moisture, light, gases, and appearance [2]. Thin layer metal is coated to the surface of the film this process is known as Metallization and it is done with the vacuum.

3. COATINGS AND MULTILAYER COATING PROCESS

One or more fluid layers are applied to material thereby improving the properties of the coated material and this whole process is known as coating [4]. It is one of the regular method used improve the performance of single packing materials. Properties like heat seal-ability, film surface protection, barrier properties, waterproofing and preserving items can be achieved. Wax is coated on cereal boxes to protect its crispness. When thickness requirements are, less than 0.00762 mm coatings are preferred over lamination [4]. In recent times due to the innovation of science and chemistry, there is an improvement of broad range of barrier properties like moisture, gas and oil, grease resistance [2].

By coating weight and in terms of thickness on substrates the amount of coating surface is specified. It is expressed in terms of mass per unit area. According to English system grams per square meter (g/m^2) and in U.S system pounds per 1000 square inches (MSI).

There is various coating process such as water-soluble coating, hot melt coating, extrusion coating, emulsion coating etc. Starch coatings to paperboard, ethyl cellulose to plastics are some of the water-soluble coatings. PVDC and nitrocellulose applied to plastic come under organic-soluble resins. To increase toughness and strength of paper, films and foils Polyolefins can be coated [4].

In cases where the coating is applied as a liquid, one of the most important properties in achieving an efficient coating is the viscosity of the coating liquid. Application of coatings can done by different coaters depending upon the viscosity, such as air knife, Meyer rod, reverse roll, gravure, extrusion etc.

3.1 Co-extrusion process

Co-extrusion is the extrusion of multiple layers of material together [2]. Extrusion of multiple layers by utilizing two or more extruders to deliver steady flow from the head or extrusion die where the extrusion of material is done in the desired way [2]. Two or more extruders are used to melt and deliver a steady proportion throughout of different gelatinous plastics to a single extrusion head (die) which will extrude the materials in the desired form. Co-extrusion is generally less expensive compared to laminating here all layers are attached together in one-step. Depending on the task and design of packaging the layers scales from 5-10. Different types of polymers are extruded from a separate extruder and extrudate can be divided further in the die to acquire the required number of layers. The molten polymers are remained separate and are usually lead in contact in a feed block or just before the die exit. Extrusion coatings, cast films, blown films can be done by extrusion. The film extrusion dies can be designed to be fed from more than one extruder. Thermoplastic adhesives are generally used as a middle layer for materials like nylon and HDPE which helps sticking to each other.

Figure 3.1 Shows co-extrusion set up using two different extruders and a manifold die.

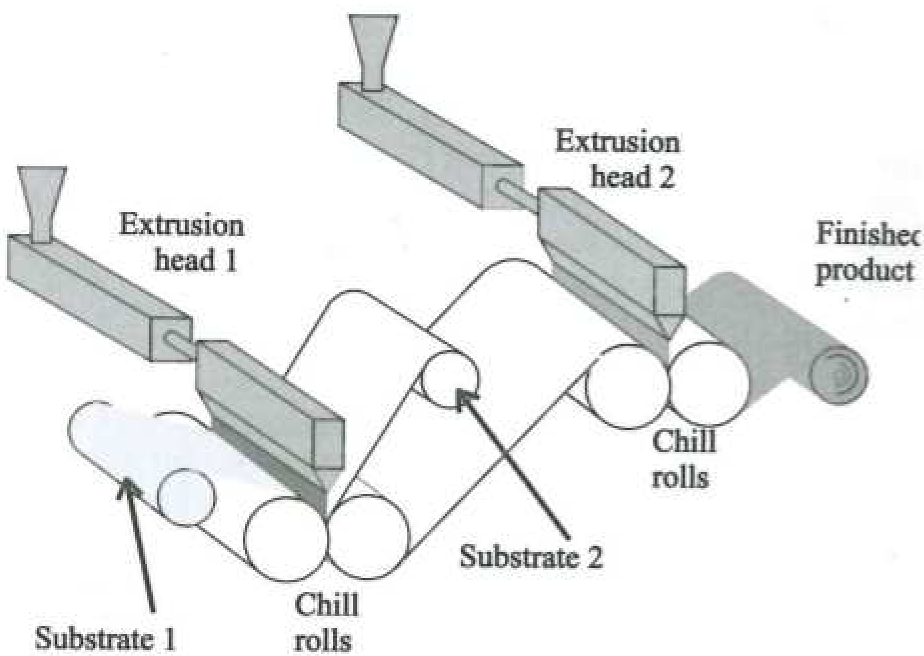


Figure 3. 1 Co-extrusion process [1]

3.2 Lamination and processes

Lamination operations results in the manufacture of multilayer flexible packaging and these are composite structures contains materials like plastic, metal, aluminum foil, and paper can also be referred as webs in laminating line are held by adhesives. Bonding of web and adhesive is done by applying pressure to adhesive layer, bonding is gained by heating and drying or chemically by curing agents. The method of bonding can be mechanical, chemical or combination of both [4].

3.2.1 Thermal lamination

In thermal lamination, the joining of two substrates is done by heat and one or more substrates are heated and cooled [1]**.** If the material is thermoplastic, there is no need for heating. If it is not, thermoplastic adhesive EVA is applied to webs and dried. The substrates are passed between two rollers and passed by heat nip rolls where the pressing is done against each other. By using heat seal coating film plastic, aluminum foils can be joined.

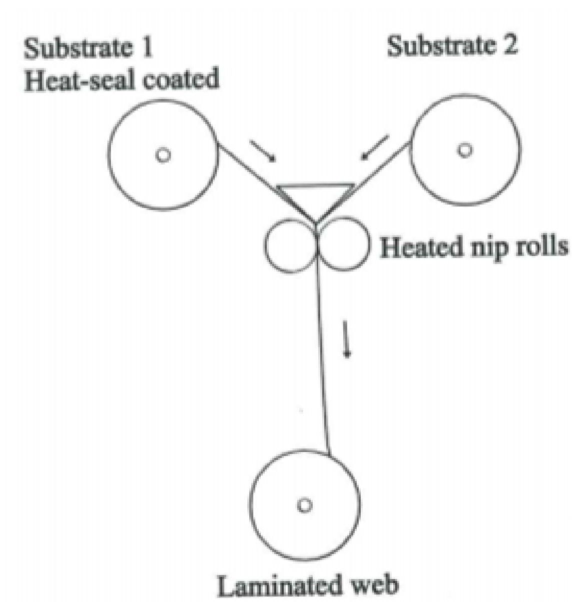


Figure 3. 2 Thermal lamination process [1]

3.2.2 Hot melt lamination

This method is narrowly related to extrusion laminating. It uses low viscosity adhesive polymer so there will no need of an extruder. It is normally used to join glassine, paper rather than plastics, molten wax is the adhesive used in hot melt lamination. Wax gives some amount of barrier to moisture and gases. These adhesives are applied to one of the webs and both webs are passed through heated nip rolls [1].

3.2.3 Extrusion Lamination

In method web of extruded polymer and heat is used where extruded polymer acts as adhesive. Compared to adhesive laminator is less expensive. For porous substrates, it is well suited. The main disadvantage of this method is only a few polymers are suited to each other due to this there will be less combination of substrates. In converting industry, extrusion coating is vastly used [1].

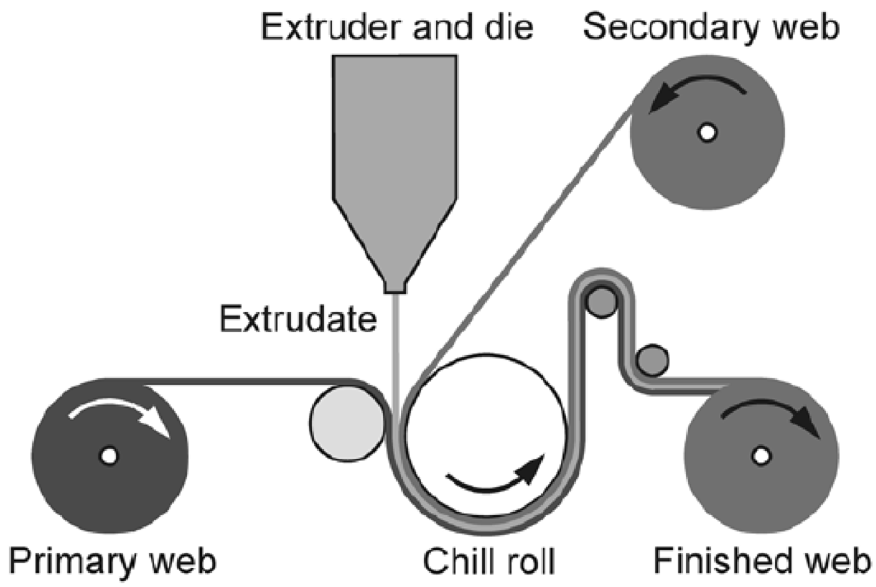


Figure 3. 3 Extrusion lamination [8]

3.2.4 Wet and Dry bond Lamination

When the adhesive is dry or wet during joining of webs the classification of this lamination is based. Methods like infrared, hot air, radio frequency, convection heating are commercial methods. The web is passed by the head tunnel in which hot air is passed on to the web for convection drying. In infrared heating, infrared is the source and air are passed onto the web for fast drying. In convection, there is a source of heat was the web is brought in contact [4].

One web must be permeable in wet lamination so that escape of solvent from adhesive and enough evaporation is done Here non permeable web is attached with the adhesive permeable web so one web must be paper or paperboard and another web be foil or plastic film. While passing through drying tunnel the solvent can be organic or water-based [3, 7].

Firstly, the solvent is dried before the webs are brought in contact with each other by this it is called as dry lamination. By using coating methods adhesive is applied to the web, the solvent is dried by passing substrate to drying tunnel, and now it is brought together with the second web near the nip rolls and after adhesive curing is done. The complete cure process can take up to one or two weeks [3, 7].

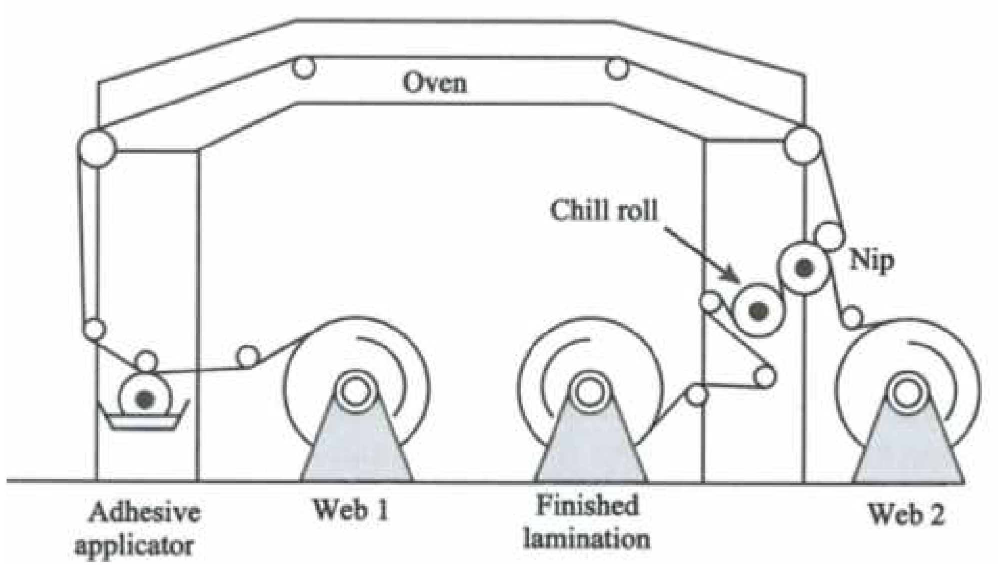


Figure 3. 4 Dry lamination process [9]

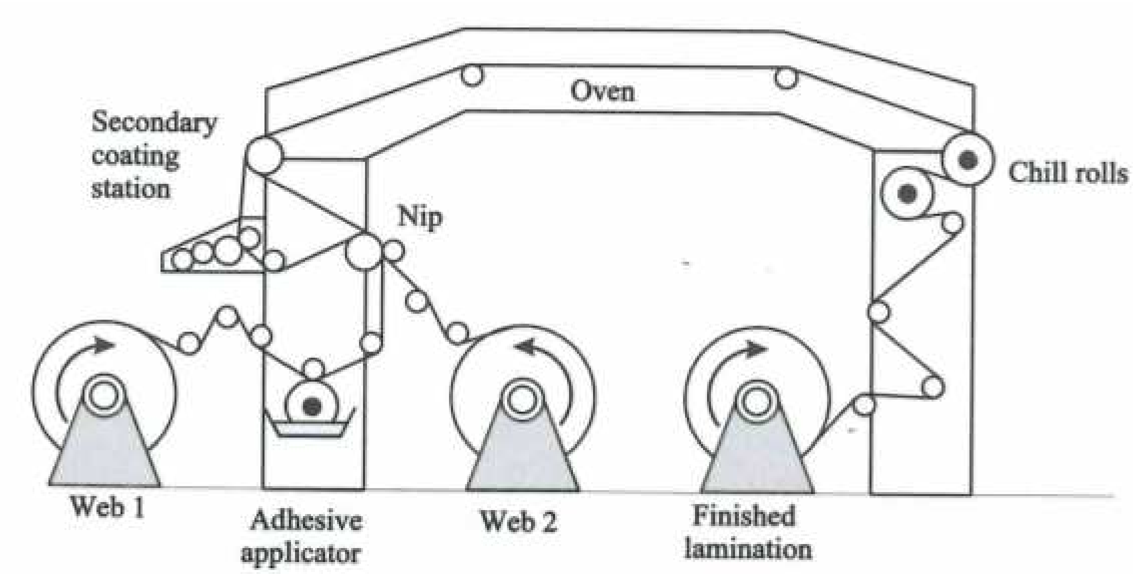


Figure 3. 5 Wet lamination process [9]

3.2.5 Solvent less Lamination

In converting industries, this is the fastest developing adhesive laminating technique. Advantages of this method are Volatile organic compounds (VOC) or any other solvents are not used and it is eco-friendly. The operating costs and capital cost are relatively lower than solvent-based or water-based lamination. Whether it is the single or two-component system the adhesive action is gained when components react and polymerize [4, 7].

4. ADHESION AND ADDITIVES

An adhesive is a substance, which holds two separate bodies such as adherent, or substrate and this process is called adhesion. An adhesive is generally referred as glue, which joins materials like glass plastic, metal and this substance mechanism works by means of hydrogen bonding, intermolecular bonding forces, chemical bonds etc. Places across the cross-section of a composite structure, adhesive bond separation may occur [1]. The adhesion process can be done by adherent heating and press them together. Cohesive bonds act with the adhesive holding it together and adhesive bonds act with adhesive and adherent interface [1].

The factors like viscosity, surface tension, solubility parameter effects adhesive bond strength. To meet certain adhesive to set of adherents these all factors are taken into consideration. Applying primer and other like plasma, corona discharge treatments to adherent's surface tension can be improved. For good adhesive bonds, the viscosity of adhesive plays a major role and for even spreading of adhesive is gained by low viscosity adhesive. According to the temperature and molecular weight, there will be decrease and increase in viscosity. There must be a similar solubility parameter for adherents and adhesives for prudent adhesive bond strength.

The strength of regarded physical and chemical nature of adherent and adhesive relates to cohesive bond strength. Cohesive bond strength should be preferably less or equal to its adhesive bond strength to achieve good performance of adhesive. Cohesive bond strength decrease with wettability and increases with high molecular weight. For achieving a prudent level of overall bond strength there should be a balance of these factors [1].

Organic based solvents, hot melt, water-based solvents are natural and synthetic adhesives. The base polymer is mixed in an organic solvent with other ingredients to form solvent-based adhesives. After the solvent evaporates, the strength of the adhesive is gained. Water is used as a solvent in water-based adhesives. Hot melt adhesives gain strength as it cools down and it is an essential molten polymer (like PE, EVA, and PP) which does not react chemically or discharge harmful solvents. Adhesives like cyanoacrylate, polyurethanes are reactive which composes low molecular weight, which on the application will begin to polymerize, eventually achieving the desired bond strength values, and they are flexible to a large variety of adherents [1].

4.1 Surface treatments for good adhesion

Primers, corona discharge, and plasma treatments are some the surface treatments done in order to make adhesives stick to the film surface. If the adherent surface tension is greater than wetting liquid surface tension wettability can be gained. The liability to decrease its surface area can be defined as surface tension. A high voltage current is applied close to the surface of the substrate thereby oxidizing the surface at an atmospheric pressure in air is known as corona treatment and it is done in an inert gas under vacuum is known plasma treatment [2,3].

4.2 Bond strength

It is important to supervise adhesives, which are used to bond flexible packaging such as films and foils, which requires the conscientious reliability of applying adhesives to substrates. Even though, the outcomes are major- product loss, packaging failure. Laminate bond fails will occur due to improper coat weight, delaminate from the base material, causing of blisters etc. [1].

Bond is the main property of a laminate structure. If the bond strength is calculated when the two substrates are brought it is known as green strength and which is measured after curing it is known as cured strength here the bond strength is calculated by (Friction/ peel tester model 225-1).

Depending upon strength of substrate and adhesive there are 3 types of failure

- Adhesive bond failure
- Cohesive bond failure
- Material / structural failure

When adhesive bond strength is lower than cohesive bond strength failure occurs at the adhesive-substrate interface and when cohesive bond strength is lower than adhesive bond strength cohesive bond failure happens. When substrate strength is lower than both bond strengths material failure occurs [7].

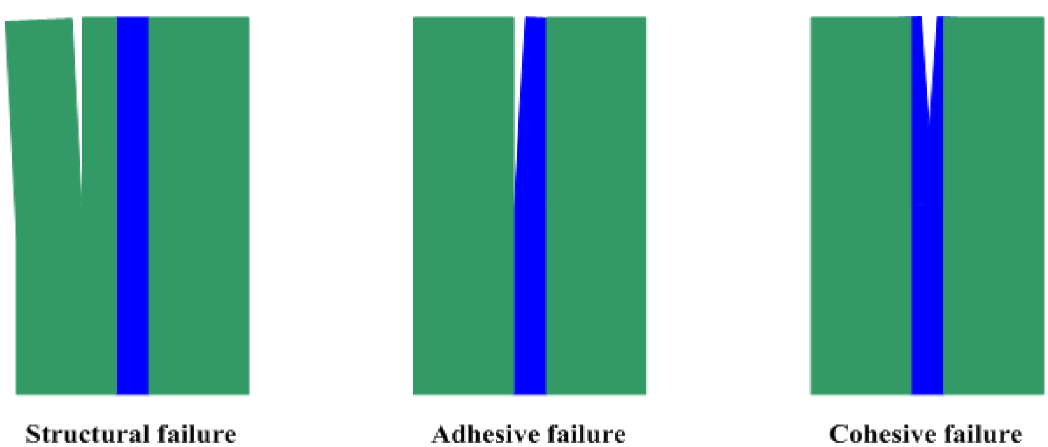


Figure 4. 1 Different failures of lamination structure [10]

5. RELEVANT METHODS USED FOR MEASURING GREASE RESISTANCE

There have been few research articles for measuring grease barrier properties in flexible packaging films. These abstracts are given below. All these tests involve setting of grease like oil, fat, fatty acids etc. on one side of the film was tested for a certain period of time under elevated temperatures (visual analysis such as staining).

METHOD 1: ASTM F119-82

Title: Standard method for rate of grease penetration of flexible barrier materials (Rapid Method) [12].

Abstract: The substance is placed between frosted glass and grease soaked cloth patch and weights are placed on cloth. The signs of grease are observed on the frosted plate by lifting up the assembly.

Measurable: Time taken by the grease to penetrate.

Drawbacks:

- Requires continues monitoring.
- Careful handling of equipment during the experiment.
- Elevated temperature up to 60°C is used and large error is possible.

Advantages: Simple experiment with minimal equipment.

METHOD 2: TAPPI T507 cm-09

Title: Grease Resistance of flexible packaging materials [14].

Abstract: For materials like glassine, vegetable parchment, greaseproof and other plastic coatings. The substrate is placed between oil statured blotter and clean blotter and stains are observed on clean blotter after 4 hours, at 60°C.

Measurable: Visual observation.

Drawbacks:

- Detection of stains potentially difficult.
- For PE films time is shorter.
- The significant error in quantifying.

Advantages: potential for quantification.

METHOD 3: CHENEY AND BREESE

Title: Improved technique for monitoring the grease penetration through substrates [13].

Abstract: Here monitoring is done with time elapsed photography and without a weight and the rest of the experiment is same as ASTM standard.

Measurable: Time taken by the grease to penetrate.

Drawbacks:

- Difficult to see failure.
- Larger error is possible.

Advantages:

- Due to little interface with the experiment, the error is minimum compared to ASTM standard.
- Photographic record.

METHOD 4: WYSER *ET AL*

Title: Novel method for testing the grease resistance of plastic based dry pet food packaging [15].

Abstract: The substrate with fat source and weight on the source are placed on TLC plate. After a certain period of time, the TLC plate is observed under Ultraviolet rays for an amount of grease penetration.

Measurable: Area of grease strain by U-V rays.

Drawbacks:

- Based on ranking given by the operator.
- The significant error in quantifying.

Advantages: Clear and simple detection system.

6. MATERIALS FOR EXPERIMENT

Material 1

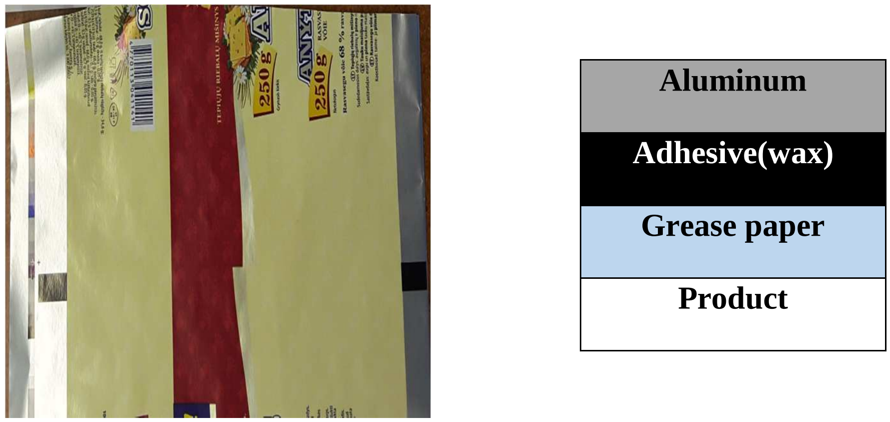


Figure 6. 1 Picture and structural view of foil laminated by paper and wax

Aluminum foil

Square weight: 24.3 ± 1.944 g/m^2.

Thickness: 0.009 ± 0.00072 mm.

Adhesive

Type: wax.

Square weight: 8.0 ± 2.0 g/m^2.

Paper

Type: grease proof.

Square weight: 40 ± 1.6 g/m^2.

Greaseproof paper is made by beating fibers more thoroughly during the manufacture of sulfite pulp. The smaller fibers make a denser surface which is more resistant to oils. However, this resistance is lost when the Paper becomes wet [16].

Fresh fish or meat, a liner for shipping containers for butter/cheese, a liner for packs of biscuits, fats and other greasy foods. Weight ranges from 40 – 60 g/m^2

Al foil acts a barrier against moisture, oxygen, gases, aroma and light Barrier is higher compared to any other plastic laminated material [6].

These foils are classified according to thickness

Rigid- >180 micrometer.

Semi- rigid- 50-180 micrometer.

Flexible- 9-50 micrometer.

Material 2

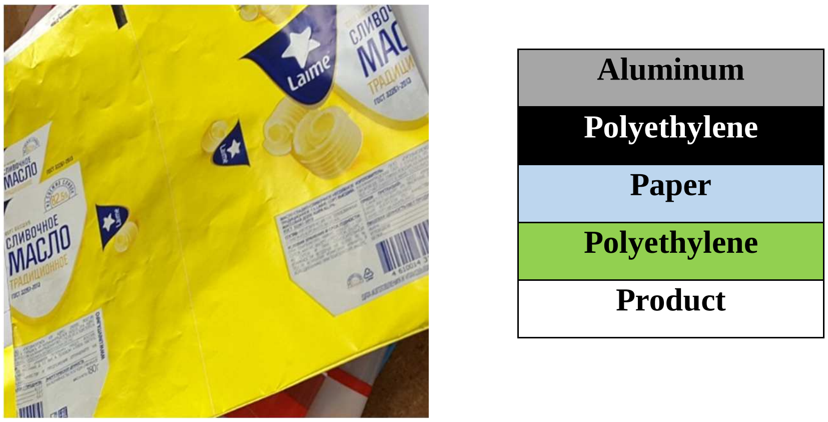


Figure 6. 2 Picture and structural view of foil laminated by paper and polyethylene

Structure: Al6/PE10/PAP35/PE15

Aluminum- 6 g/cm^2, Polyethylene-10 $g/\ cm^2$, Paper-35 $g/\ cm^2$, Polyethylene- 15 $g/\ cm^2$.

Polyethylene family is divided into

1) Branched- LDPE

2) Linear- LLDPE, HDPE

LDPE: 0.910 – 0.925 g/cm^3

HDPE: 0.940 – 0.965 g/cm^3

LLDPE: 0.916 – 0.940 g/cm^3

LDPE exhibits high clarity, flexibility, good oil resistant and heat sealability. Transparency is higher than HDPE. LDPE has better seal, clarity, and gloss than LLDPE [1].

LLDPE exhibits improved mechanical properties compared to LDPE. The Melting point is 10-15°C greater than LDPE. Major completion to LDPE is LLDPE provides greater strength at equal densities [1].

HDPE majors used for milk, detergents, shampoo, juices, water bottles its melting point is 128-138°C [1].

Material 3

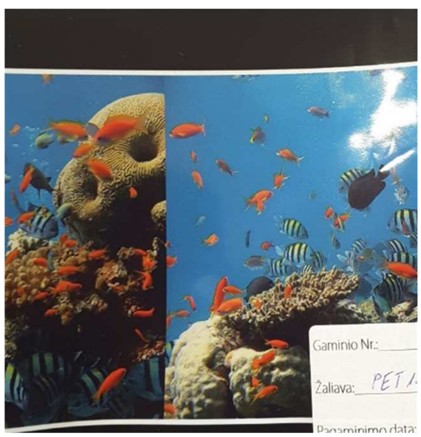
Paper
Polyethylene
Product

Figure 6. 3 Picture and structural view of paper laminated by polyethylene

Structure: PAP 60/PE 30

Polyethylene-30 g/ cm^2, Paper-60 g/ cm^2

Material 4



PET
Polyethylene
Product

Figure 6. 4 Picture and structural view of Polyethylene terephthalate (PET) laminated by polyethylene

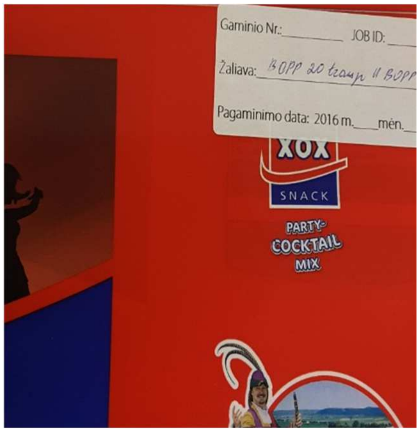
Structure: PET 60/PE 40

Polyethylene terephthalate 35 g/ cm^2, Polyethylene- 15 g/ cm^2

PET exhibits good barrier for Oxygen, CO_2 largely used in soft drink bottles. Density: 1.29 - 1.40 g/m^3

Typical food involves edible oil, syrups, spices, peanut butter, cocktail mixers. CPET is crystalized form it is used in basic materials for microwavable containers for frozen foods. PET coating on paperboard provides oven able board for use in applications such as frozen dinners. It has low melt strength and biaxially oriented PET film has excelled odor barrier properties [16].

Material 5



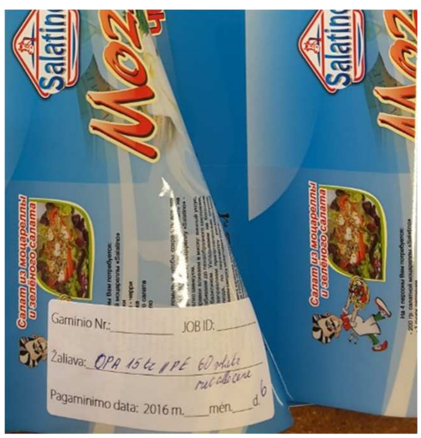
BOPP
BOPP
Product

Figure 6. 5 Picture and structural view of BOPP material

Structure: BOPP 20 /BOPP 30

BOPP has a low density of 0.9 g/m^3. The melting point is 169°C highly suitable to allow for sterilization. It shows good moisture barrier, toughness, clarity and low cost. There are some disadvantages such as low tear resistance, gas and aroma barrier, poor reliability and printability. Especially used in chocolate bars, biscuits, snacks, ice cream, sweets etc. [17]

Material 6



Oriented polyamide
Polyethylene
Product

Figure 6. 6 Picture and structural view of oriented polyamide and polyethylene

Structure: OPA 15/PE 60

Materials involved **OPA** (Oriented Polyamide) and polyethylene.

Due to its unique properties such as mechanical strength, high flexibility, high transparency, toughness polyamide is vastly used in flexible packaging. OPA film is characterized by very high stiffness, high strength, and good printability, compared to non- oriented polyamide film OPA has 30% higher O_2 barrier [17].

7. TESTING PROCEDURE

7.1 Grease penetration test

Significance

The rate of grease penetration of flexible barrier materials in standard conditions can be obtained by this method. This method is useful in innovation and development of a selection of flexible barrier materials used for grease barriers.

As compared to other tests, this is rapid because less equipment and amount of oil (grease) for detection is less. Depending upon the variation in and thickness of structure the failure time varies for each material [12].

Apparatus

- **Thin layer chromatography** (TLC) Plates (10cm × 10cm) with silica gel on one side only. It helps in viewing minute drops of reagent after the failure of material which fluoresces under UV light.
- **Cotton patches** of 20 mm diameter are used to hold the reagent the exact position on the material.
- **Olive oil** (reagent) exhibits low viscosity so there will be a free flow of reagents between layers after puncturing of material and it is cost effective.
- **SNOL oven**, it helps in the thermal treatment of material and to maintain test temperature of 40°C.
- **Ink filler** which helps to drop oil olive on to the cotton patches drop by drop without any excess oil.
- **Weights** circular stainless bars of diameter 20mm which gives pressure on the material. Below in table 7.1 shows weights used in the experiment.

50.1	49.8	51.1
50.7	51	50.4
51.1	50.5	52
51	47.8	50.9
50.9	51.1	50.8
49.6	50.1	50.5

50.7	50.4	50.9
49.8	51.2	50.6
50.7	50.6	51.4
50.9	50.3	50.5
50.6	50.4	51
50.3	50.5	49.6
50.6	50.5	50.8
50.9	51.2	50.6
51.2	50.3	50.6
50.9	50.8	50.5
50.6	50.4	

Table 7. 1 Values of weights used in experiment

Mean: 50.594 g.

Standard Deviation: 0.6011.

Confidence Level (95.0%): 0.170.

Confidence interval: 50±0.17 g.

- **Metal block** of weight 5 kg to give substrate a light crease.
- **Ultra violet lamp** use to examine TLC plates if any grease penetration is done.
- **Analytical balance**

Below table 7.2 shows apparatus used in experiment

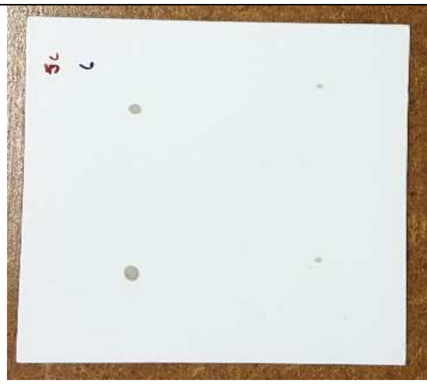
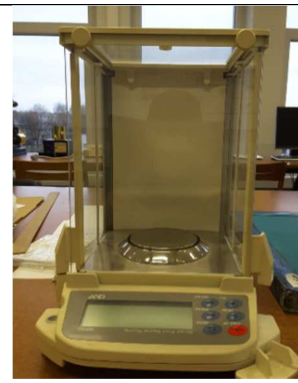
 **TLC plate**	 **Analytical Balance**
 **Oil olive**	 **Oven(thermal treatment)**
 **Ink filler**	 **Weights & cotton patches**

Table 7. 2 All apparatus used in the grease penetration test

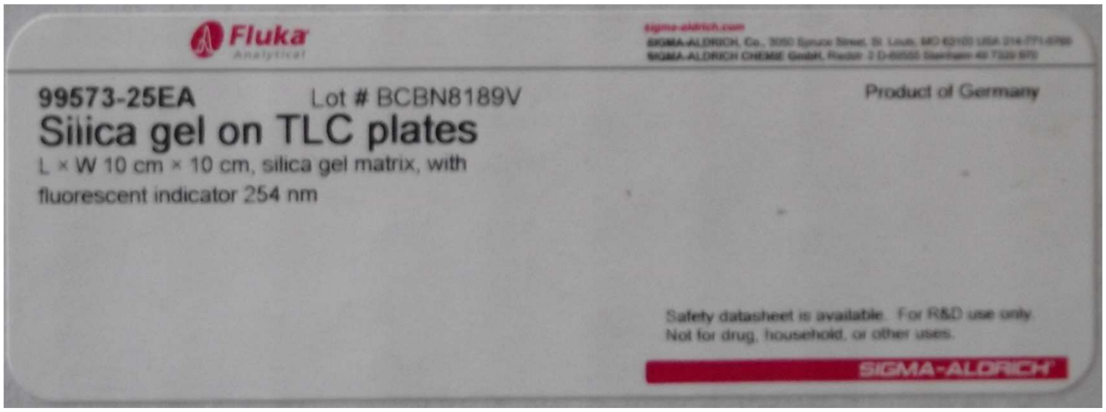


Figure 7. 1 TLC plates with silica gel produced by FLUKA analytical is used in experiment

Testing Procedure

Firstly, four specimens or samples of each material is taken or cut by 10.2 × 10.2 cm. Two sample of each material are creased of one time by folding the sample from both ends to the center of it and metal block weighing 5 kg is placed parallel to the edge of the fold.

Now the TLC plates are placed on oven tray and these plates are covered by the samples which covers the TLC plate which prevents premature edge creep failure.

Now cotton cloth is scissor into 20 mm diameter disks or patches just to fit under weights. These cotton patches are placed on top of substrate material or sample as shown in figure 10.2 with help of ink filler, 2-3 drops of the reagent (olive oil) are added to the cotton patches and place the weights on the patches. Periodic addition of oil is done if necessary.

The whole assembly of six specimens at one time are placed on the oven tray and heated to a desired temperature (40°C) for 20 min and time is noted down. By picking up the assembly monitoring for signs of wetting is checked.

At periodic intervals, the experiment is conducted that is, every 20 min for the (first hour every 1 hour for the next 3 h, then at convenient intervals) the test specimen, cotton disk, and weight are lifted from the TLC plates as a unit and the surface of the glass is observed under U-V light. The time is recorded at which the first trace of wetting is visible at the position of the weight. If no failure is visible, the assembly is replaced in the oven.

Figure 7. 2 View of experimental procedure

7.2 Delamination test

Delamination occurs when inadequate and failure of adhesion between two laminated takes place layers which result in mechanical failure of the material. For testing of delamination, material 1(foil laminated with paper) and adhesive (wax) is between there layers. This material sample is tested to observe when the delamination occurs. The sample or substrate is heated at a temperature from (25°C) and time is noted down.

At periodic intervals, the experiment is conducted for every 10 min the temperature is keep-on increasing about 5°C from (25-45°C) and time is noted.

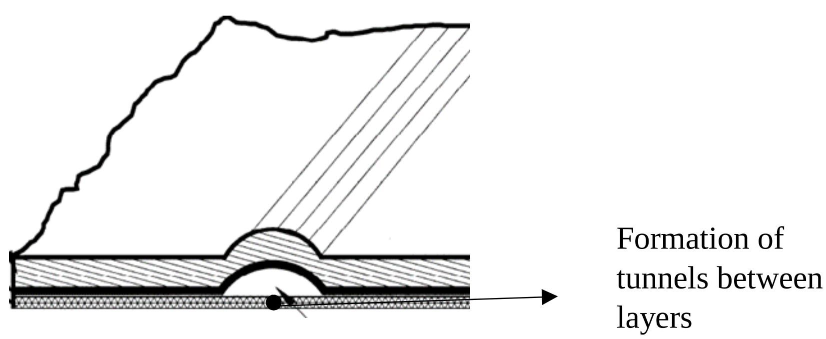


Figure 7. 3 Schematic view of tunnel between layers

7.3 Analysis of adhesive bond test of flexible packaging

7.3.1 Peel test

In peel test adhesive bond strength or called as adhesive fracture toughness between two flexible substrates, usually by tensile are tested. The average force required to fracture adhesive bond is measured when pulled at a specified angle. An Adhesive is a thin layer between two substrates and strength of the adhesive is also referred as the stickiness of a material. By peeling we can know about its properties like bonding strength, bond durability, sealing consistency, cohesive properties of the interface and adherence ability. Peel test is a tool which used for quality assurance control of a product and benefit of performing the task. Precise and repeatable peeling testing can improve product stability and determine interface adhesion [5].

- **Type:** 180° peel test

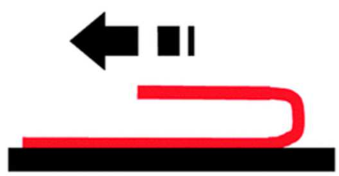


Figure 7. 4 Schematic view of 180° peel [18]

- **Test Material:** Aluminum foil laminated with paper.
- **Adhesive:** wax is used as an adhesive between these flexible layers.
- **Equipment:** Friction/peel tester model 225-1by Thwing-Albert Instrument Company shown in figure 7.5

Figure 7. 5 Peel equipment used in the experiment

7.3.2 Testing procedure

Significance

This test method provides the average force required for determining the adhesive bond of flexible barrier materials. This method is valuable in the development and stability of adhesive in flexible barrier materials used for grease barriers.

Apparatus

- Six sample rectangular strips of same material (Al foil laminated with paper) is cut with dimensions width 15 mm and length 80 mm is taken.
- Dino-lite microscopic camera used for a closer view of adhesion fracture between two layers.
- Friction/peel tester model 225-1 manufactured by Thwing-Albert Instrument Company.
- Sharp knife for initial separation of substrate layers.
- Tape for fixing substrate to 180° peel arm and spring clip.

Procedure

Firstly flexible multilayer layer material (foil laminated with paper) with adhesive bond (wax) is taken and cut down into 6 rectangular strips of dimension (80 mm × 15 mm).From one end of the strip is peeled or separate two layers carefully with a sharp knife of about 10 mm length. From this end, one substrate is attached to spring clip as shown in figure 10.4 and the other end attached to 180° peel arm these are attached with tape. The whole setup is placed on tester platform and Peel arm is fixed to the load cell of the tester which calculates load required to separate two substrates.

This procedure is conducted for all the 6 strips and with the help of Thwing-Albert FPDAS version 1.0.0.2 copyright (C) results are shown in the computer for each individual strip.

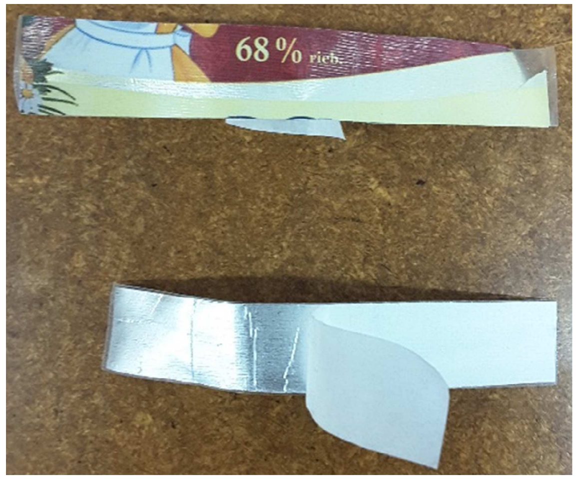


Figure 7. 6 Rectangular strips (80 mm × 15 mm) used in experiment

Now power on the peel tester and the settings are changed as followed

Load (LD): Newton (NT)

Prepeel: 5 sec

Time: 40 sec

Speed: 5 cm/min

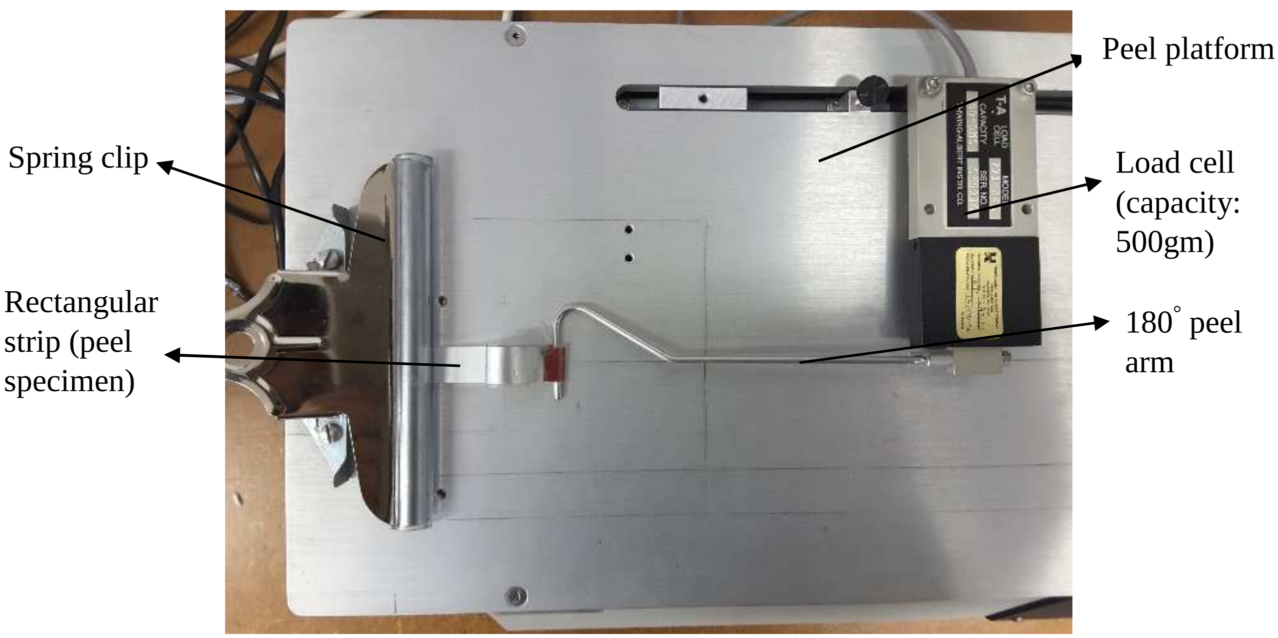


Figure 7. 7 View of peel test equipment and the apparatus

8. RESULTS & ANALYSIS OF RESULTS

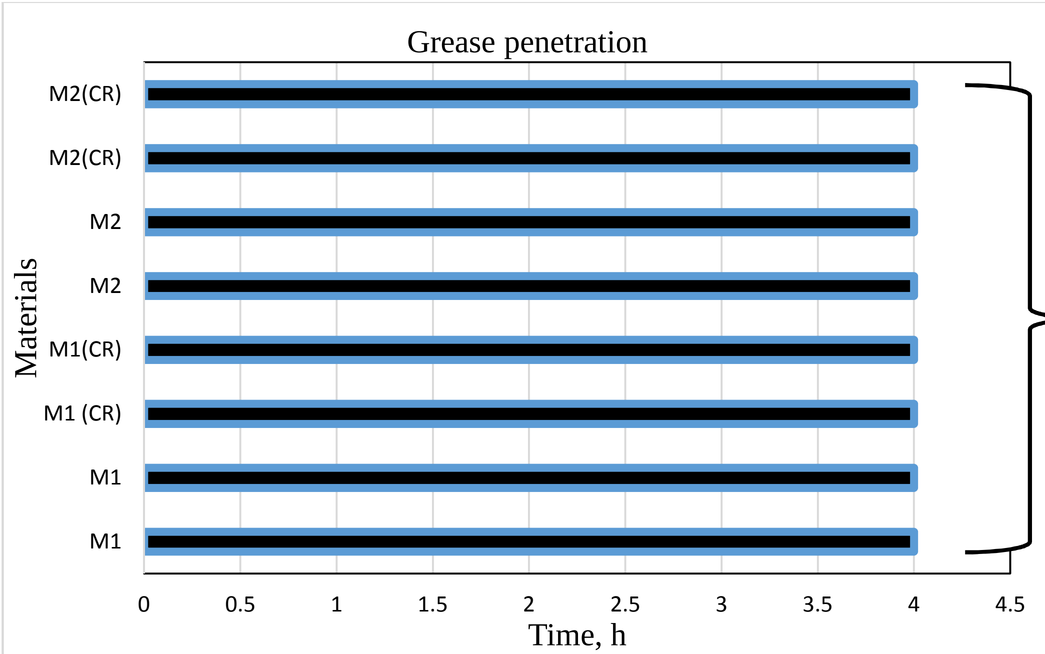


Figure 8. 1 Grease penetration result of materials (1&2)

Above figure 8.1 shows foil laminated by paper and wax (M1) & foil laminated with paper (M2). Where M1 (CR) & M2 (CR) represents substrates which are creased for 1 time. This test is conducted for 4 hours at regular intervals and a constant temperature of 40°C is maintained.

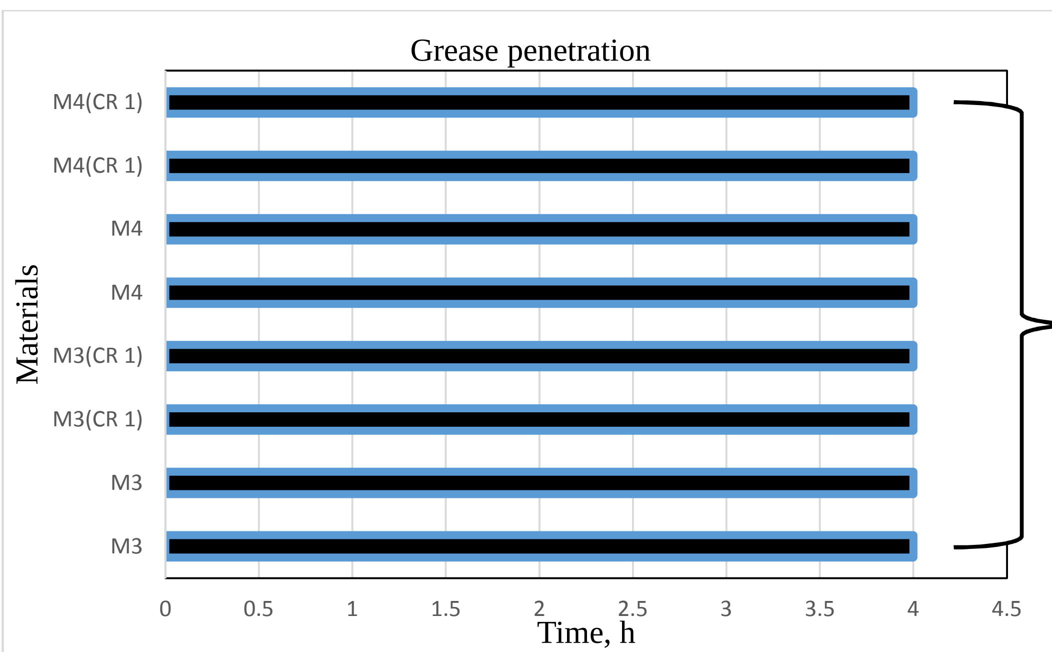


Figure 8. 2 Grease penetration result of materials (3&4)

Above figure 8.2 shows paper laminated with polyethylene (M3) & polyethylene terephthalate with polyethylene (M4). Where M3 (CR 1) & M4 (CR 1) represents substrates which are creased for 1 time. This test is conducted for 4 hours at regular intervals and a constant temperature of 40°C is maintained.

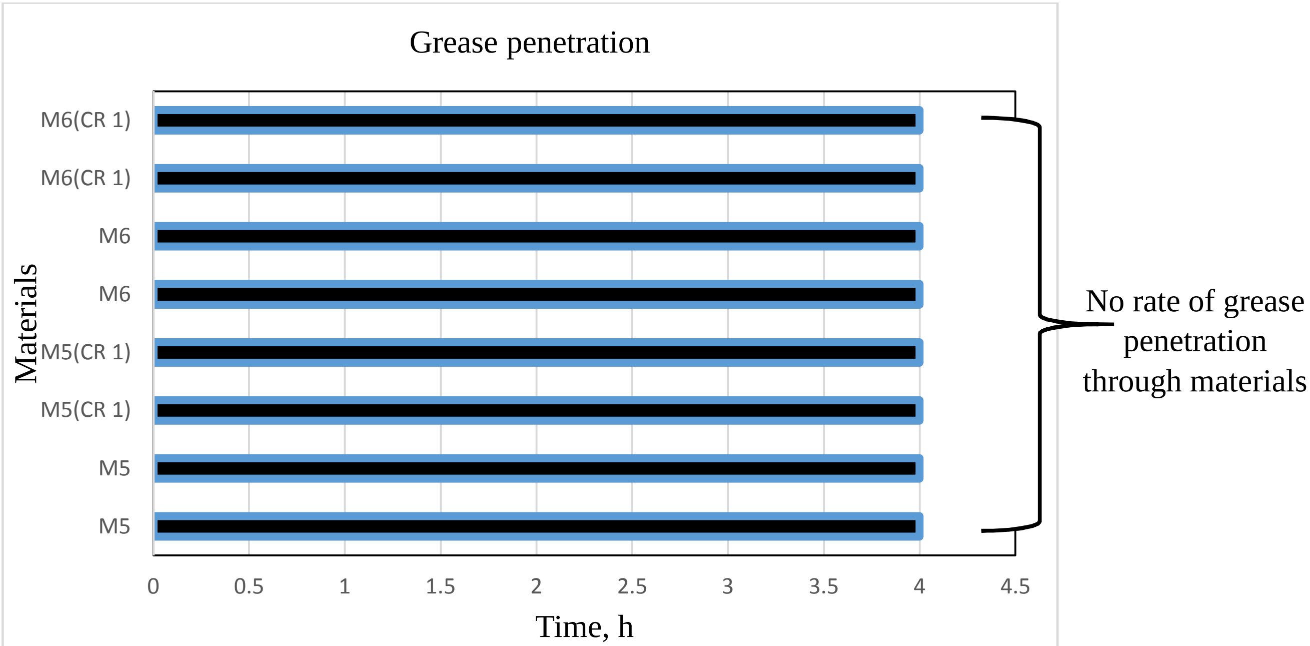


Figure 8. 3 Grease penetration result of materials (5&6)

Above figure 8.3 shows biaxial oriented polypropylene (M5) & oriented polyamide with polyethylene (M6). Where M5(CR 1) & M6(CR 1) represents substrates which are creased for 1 time. This test is conducted for 4 hours at regular intervals and a constant temperature of 40°C is maintained.

Result: As from the above figure 8.1, 8.2, 8.3, we can see that the substrates exhibits zero rate of grease penetration at 40°C. For further analysis, the test is conducted again by increasing number of creasing time of materials (1 & 2).

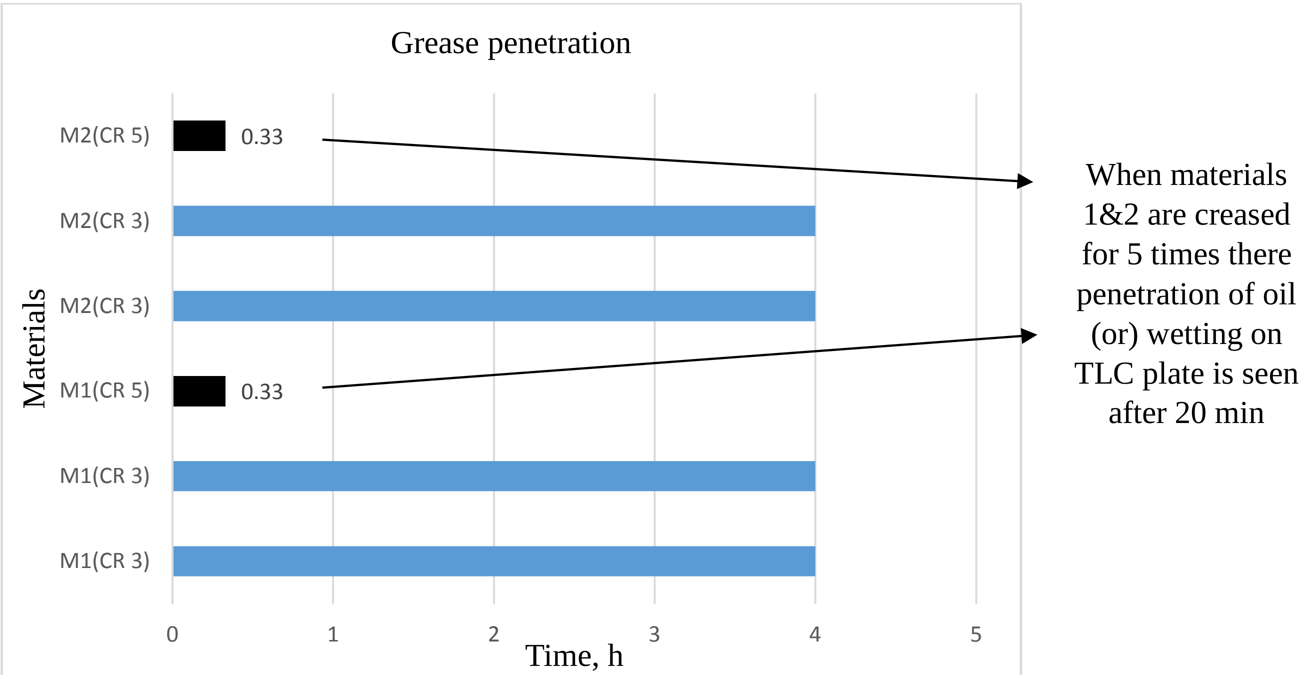


Figure 8. 4 Grease penetration result of materials (1&2)

Above figure shows 8.4 foil laminated by paper and wax (M1) & foil laminated with paper (M2). Where M 1(CR 3, 5) & M2 (CR 3, 5) represents substrates which are creased for 3 and 5 times. This test is conducted for 4 hours at regular intervals and a constant temperature of 40°C is maintained.

Result: As we can observe there is no failure of substrates or material (1&2) which are creased for 3 times and materials which are creased for 5-time failure occurred at 0.33 Hour (20 min) for Material 1 and material 2. By this it is clear that when aluminum foil with paper is subjected to more creasing it fails.

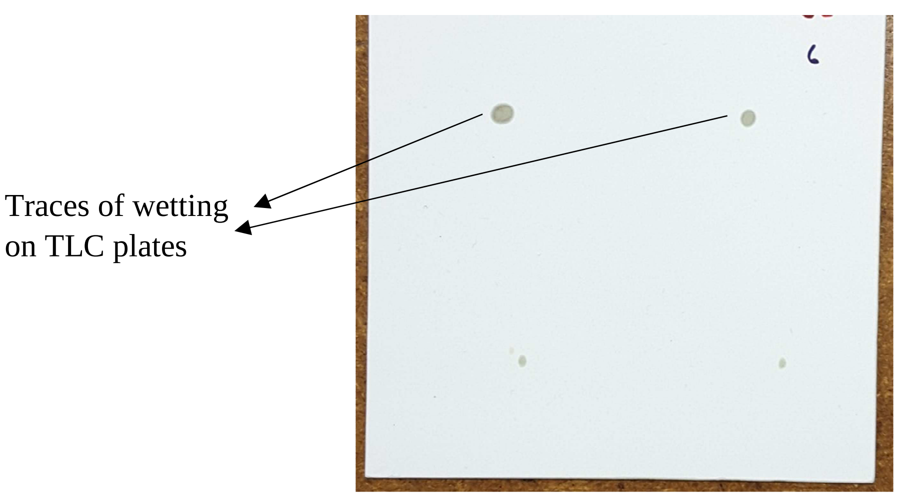


Figure 8. 5 Traces of grease penetration on TLC plate through material

Delamination Result

25°C	No failure
30°C	No failure
35°C	No failure
40°C	Failure occurred
45°C	Failure is more

Table 8. 1 Failure between layers at different temperatures by formation of tunnels

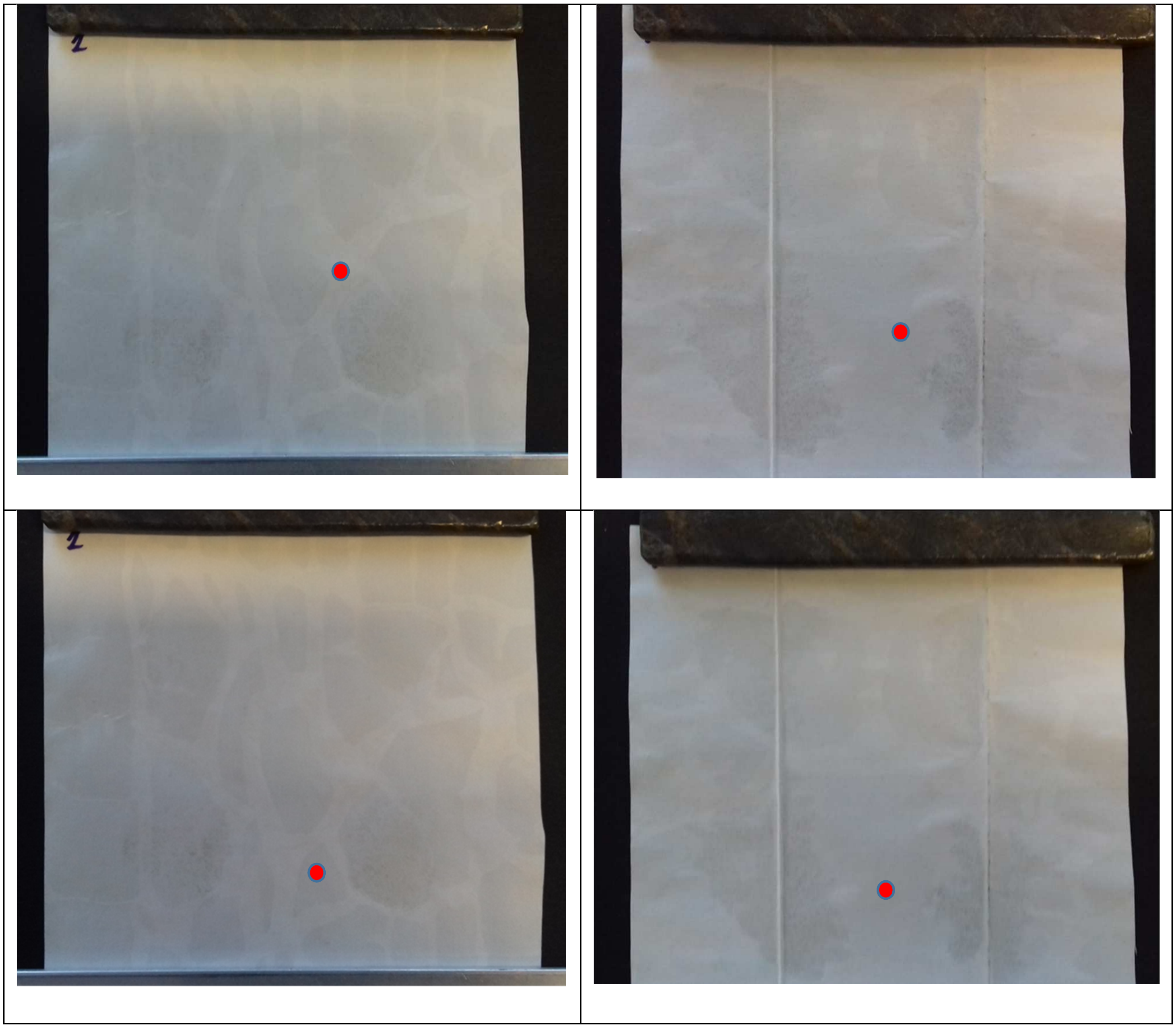


Figure 8. 6 Failure or delamination by formation of tunnels between layers of material

There is no failure or delamination at (25, 30, 35°C) for every 10 min respectively. There is failure of adhesion between substrates after 30 min at 40°C is shown in above Figure 8.6 and failure is maximum at 45°C.

Peel test

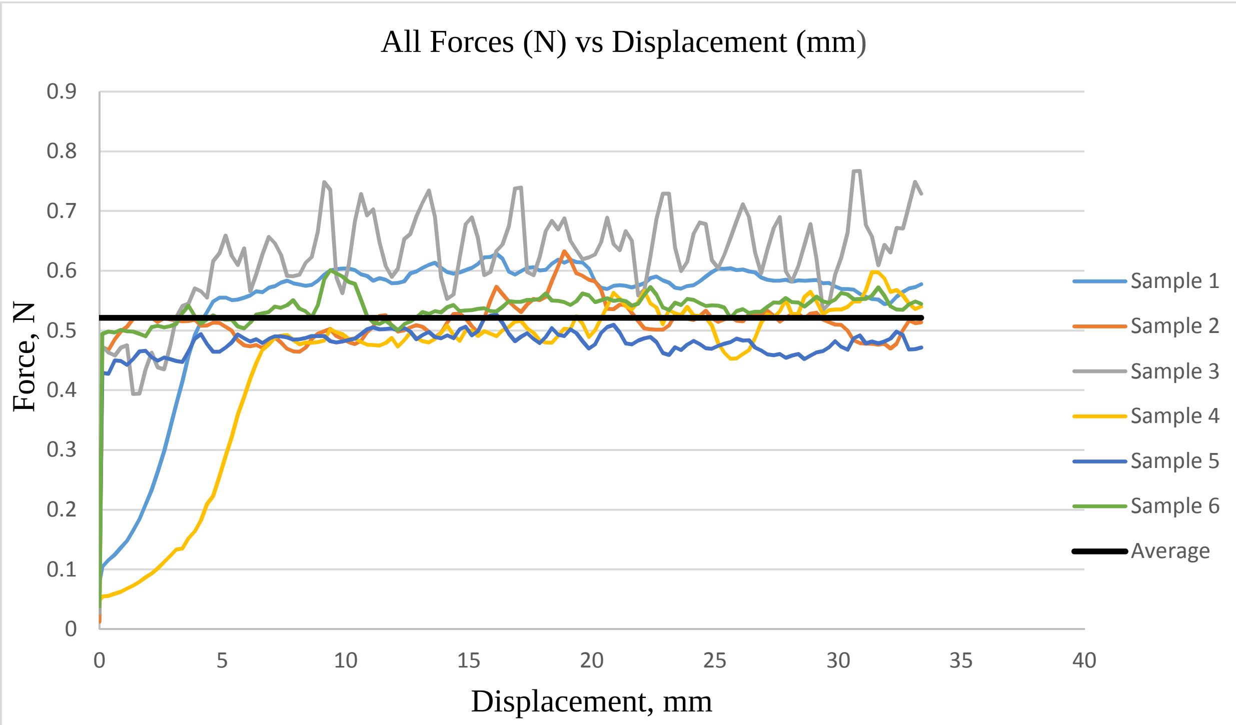


Figure 8. 7 Forces and displacement of all 6 samples used in experiment

From the above figure 8.7 shows forces required to fracture wax adhesive between aluminum foil and paper for all 6 rectangular strips (average force: 0.52 N) with displacement of 33.33 mm.

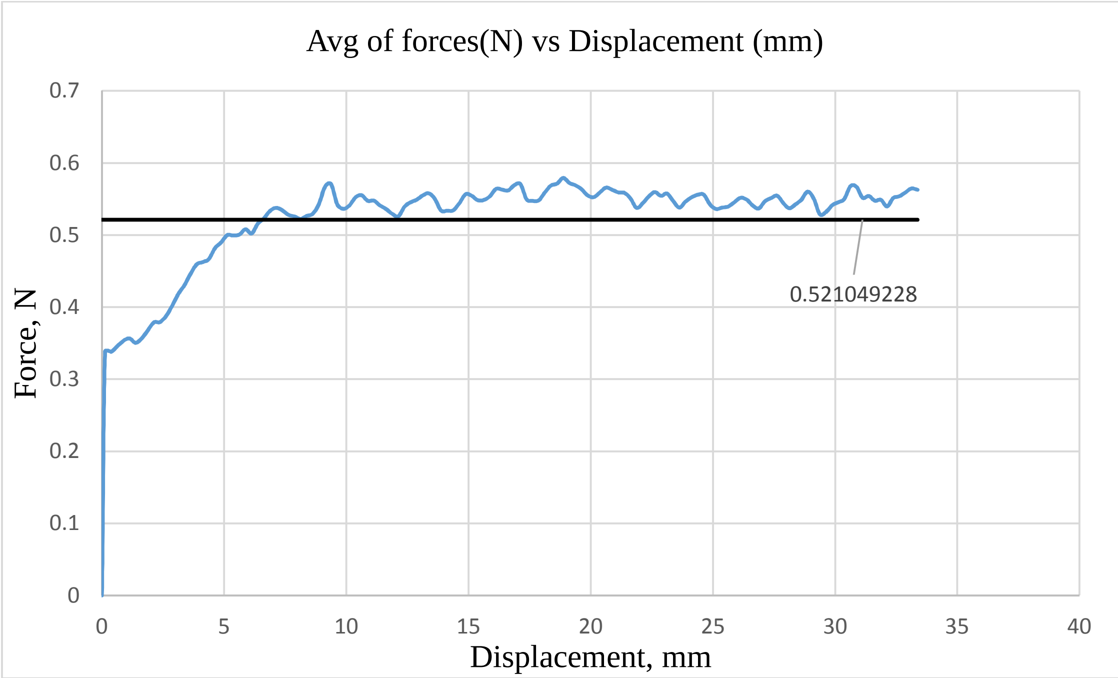


Figure 8. 8 Average force of all 6 samples

From the above figure 8.8 shows average of all forces required for the adhesive (wax) to puncture of six rectangular strips max load is: 0.579 N and the average load is 0.52 N.

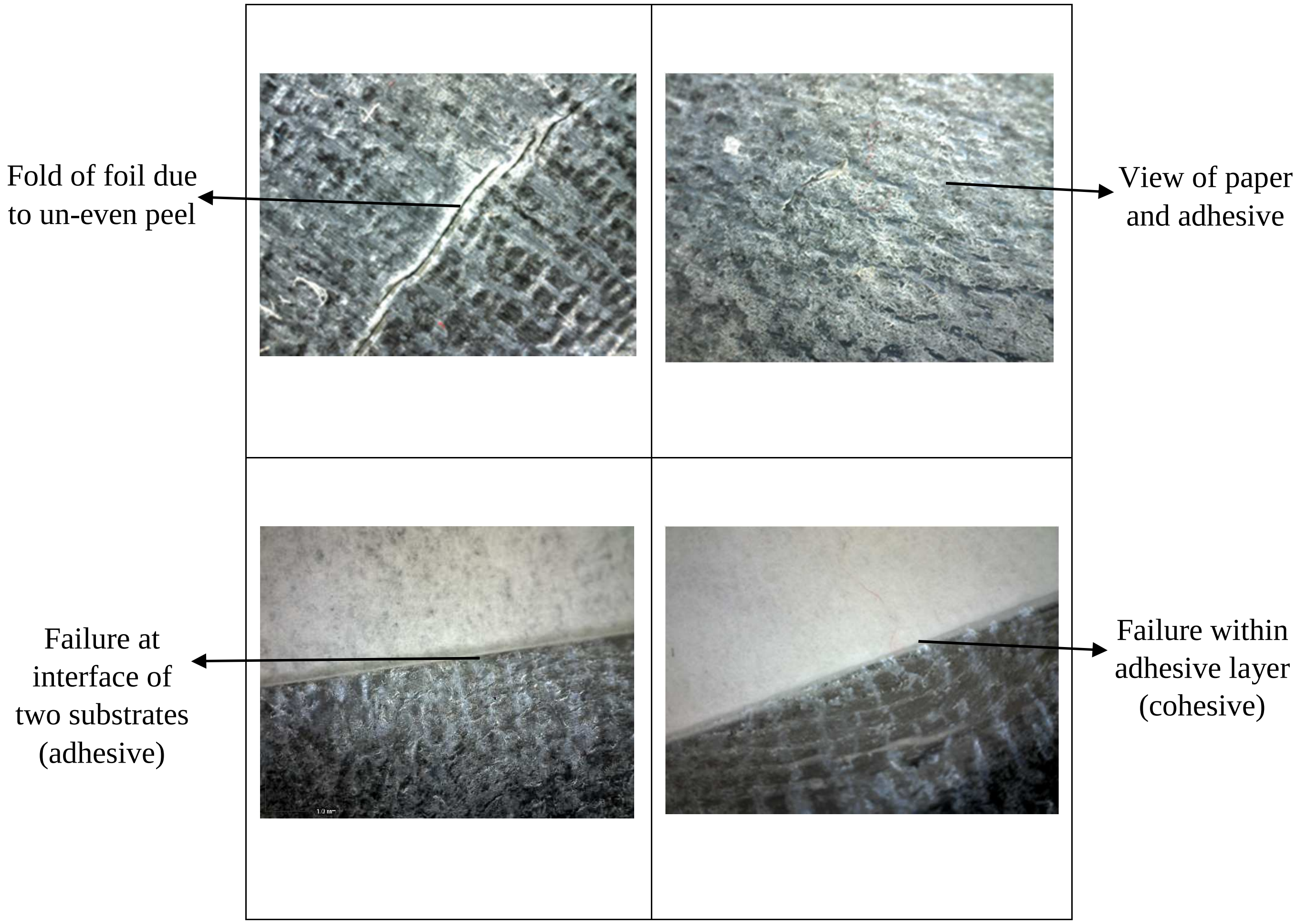


Table 8. 2 Microscopic images of material and adhesion after peel test

From the above Table 8.2 shows closer view of the material and adhesive (wax) fracture after the peel test. It is clear seen there is some un-even peel between the substrates and improper peel of paper most of paper fibers adhere to the aluminum foil so this shows poor adhesion between substrates. The failure is not within the adhesive layer this indicates adhesive failure.

CONCLUSIONS

1. Aluminum foil and films like PET, PE, OPA, BOPP exhibit good grease barrier properties when they undergo thermal treatment or heat conductivity at constant 40°C temperature. When aluminum foil is creased for 5 times there is penetration of grease after 20 min.
2. When aluminum foil is subjected to high pressure on the creased edge it tends to fail its grease barrier properties at constant 40°C temperature.
3. Wax adhesion at different temperatures could not control the bond strength between foil and paper layers and extreme relocation occurred when it is subjected to heat treatment ≥ 40°C.
4. The rapid method is a semi-quantitative method for assessing the grease resistance of multilayer packaging films was developed. It was seen that films containing PE or PET can provide exceptional grease-resistant barrier films. Foil laminated with paper exhibit delamination properties at elevated temperature 40°C.
5. The method used for grease penetration requires constant observation and cautious handling of equipment. This experiment requires minimal equipment and there is a possibility of large error.
6. If the failure is within adhesive layer it shows good adhesion between substrate and adhesive since the failure in the experiment is at the interface of two substrates shows adhesive failure, so this indicates bad adhesion between the substrates.

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