LAPPEENRANTA UNIVERSITY OF TECHNOLOGY
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THE EFFECT OF CREASING AND CALENDERING ON BARRIER PROPERTIES OF DISPERSION COATED PAPERBOARDS

Examiners: Professor Kaj Backfolk
D.Sc. Katriina Mielonen

Supervisors: D.Sc. Sami-Seppo Ovaska
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ABSTRACT

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Master's thesis

2017

61 pages, 35 figures 11 tables and 8 appendices

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Keywords: Creasability, Calendering, Paper and paperboard material properties, coatings material creasability.

Paper and paperboard based product are significantly used in our daily life. The most effective and efficient way to produce paperboard packages is by converting. This study analyses the key aspects of converting mechanism and the effect of various barrier properties of dispersion coated commercial paperboard material to enhance the end user quality of paperboard and paper-based products. The preliminary focus of this study is to determine the creasability of barrier properties of dispersion coated paperboards, and the results are complemented and validated with experimental techniques such as calendering, creasing, Cobb60 and OGR test. The coating materials used in this study are hydroxypropylated starch, kaolin clay, and styrene-butadiene latex and also their effects on creased and uncreased paperboard are compared with the aid of light microscopy. Furthermore, the effect of creasing and calendaring on barrier properties of dispersion coated board is investigated. The results revealed from these two approaches that light microscopy is not capable to thoroughly analyze the coating cracks. High-resolution microscopes such as SEM is required entirely to understand the individual fibre damage. Moreover, dispersion coated paperboard, and commercial material showed positive results on oil and grease resistance (OGR) test. The calendaring process can be used to improve the OGR of barrier properties of dispersion coated paperboard. Mechanical and physical properties like bulk, density, grammage, air permeability, roughness, strain break (% elongation) and tensile strength are measured to analyze and contemplate the study.

ACKNOWLEDGEMENTS

The thesis was conducted at the Lappeenranta University of Technology in the Department of Packaging. Kindly my great thanks to Professor Kaj Backfolk, D.Sc. Katriina Mielonen, D.Sc. Sami-Seppo Ovaska and D.Sc. Ville Leminen, for initiating the thesis topic and notable support, which guide me to do this study under their invaluable supervision, to ensure accomplishment of this thesis on appropriate time.

First and foremost, I would like to thank Almighty God, creator of heaven and earth, for the gift of life granted to me, and making everything possible for me since my birth. I will always adore him, glory to God.

Special gratitude goes to my parents, Mr and Mrs Ernest Ekaini Osima who are always in support of my academic career. The soft heart and love of Faith-Sari, Alice-Sara and Gabriel-Ethan Osima as my children motivated me to reach my goal on this study.

Besides, I appreciate my siblings Florah Osima, Teddyson Mauki, Anna Osima, Edaline Osima and Edina Osima for their unconditional support, love and prayer that strength me during my studies.

Special thanks go to my friends Peter Meta, Peter Mwakosya, Mawazo Mtafya, Dr. Binamu Augustino, Mr and Mrs Armatia Foya, Mr and Mrs Frank Kajula and Rehema Fank. Aslo, Gilbart Osima, Kelvin Mauki, Amani Meta, Felix John, Eliaman Foya, Adil Ponnurangam, Fahad Hamad Rashid, Vinod Mgidange and Elisante Kimambo for their continuous assistance in many aspects throughout my studies.

Furthermore, I would like to acknowledge my debt of gratitude to my colleagues Muhammad Awais, Sushil Kasala, Javier Hernandez, Yang Gao and Kristóf Szombati. I appreciate their assistance, cooperation, encouragement, and invaluable advice throughout the time at School.

Special warm thanks go to my lovely wife Esther Frank Kajula, I am genuinely grateful for her love, patience, compassion and support. For all these three years, she played a significant role, taking care of the family; it was indeed not easy, but she made it.

Osima Edson

Lappeenranta 13.12.2017

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LIST OF SYMBOLS AND ABBREVIATIONS

AKD	Alkenyl ketene dimers
ASA	Alkenyl succinic anhydride
CD	Cross direction
CTPM	Chemo thermomechanical pulp
EVOH	Ethylene vinyl alcohol
GCC	Ground Calcium Carbonate
GSM	Grams per square meter
HPS	Hydroxypropylated starch
LM	Light Microscopy
LUT	Lappeenranta University of Technology
LWP	Light - weight two-sided coated paper
NaOH	Sodium Hydroxide
ml/min	Bendtsen SI Unit; Number of milliliters of air escaping per minute
OGR	Oil and grease resistance
PCC	Precipitated Calcium Carbonate
PE	Polyethylene
pph	Parts per hundreds
PVAc	Poly-vinyl acetate
REF 1	Reference material 1
REF 2	Reference material 2
REF 3	Reference material 3
SA	Styrene n-butyl acrylate
SB	Styrene Butadiene
SBB	Solid bleach board
SEM	Scanning electron microscopy
SUB	Solid Unbleached board
T_g	Glass transition Temperature
WBBC	Water base barrier coatings

1 INTRODUCTION

1.1 Paper and paperboard overview

Paper and paperboard based product are significantly used in our daily life. The most effective and efficient way to produce paperboard packages is by converting process. This study analyses the key aspects of converting mechanism and the effect of various barrier properties of dispersion coated commercial paperboard material to enhance the end user quality of paperboard and paper-based products.

Globally, the manufacturing of paper and paperboard materials has been increasing gradually on a global scale. These materials possess various applications such as stationary, money, general printing, newspaper, wallpaper, tissues, books and packaging materials. Eventually, paper and paperboard packaging are available where commodity is manufactured, supplied and delivered to the end user (Kirwan, 2013, p.3-5.)

Packaging has a significant role in production industries that enhance product shelf life, storage, transportations and distributions of goods to the end users (Kuusipalo, 2008, p.286). Firms are putting in more efforts to ensure that goods produced get to the customers with desired conditions, while the packaging is considered as a key factor to spearhead this battle. Packaging itself is a growing sector considerably, due to its importance. However, when considering sustainable packaging, the use of paper and paperboard material has been increasing significantly (Tambe, et al., 2016, p. 270). Paper and paperboard material used in packaging are preferable over plastic films due to recyclability and environmental aspects (Petrie, 2006, p. 1). At least 50 % of the material used in packaging industries contains paper materials. Packaging material such as carton are more suitable for high quality, hygiene products, and they are manufactured from printed coated paperboard followed by converting process such as folding and creasing (Kim, et al., 2010, p. 842). Correspondingly, Holik (2012) estimates that the consumption of paper material will progressively increase from 400 million tons per year to 530 million tons per year in 2020 and reaching to 605 million tons/year in 2030.

Package manufacturing involves various processes after printing such as forming, varnishing, lamination, cutting, creasing and folding. These are just examples of converting processes. Paper and paperboards are coated for several reasons depending on final use; one of such uses could be improving surface properties. During converting process, such as creasing, barrier properties of coated paperboard may influence the effectiveness of package. Thus, this study is intended to investigate the effect of creasing and calendering on barrier properties of dispersion coated paperboards. Paperboard possesses high bending stiffness and tensile strength. Paperboard has only moderate cracks resistance where both creasing and folding may damage especially the coating. The type of fibre has great effects on cracking tendency (Sim, et al., 2012, p. 445). Packaging industry records, a successful material when converting properties, for instance, workability, creasability and foldability aspect is achieved (Calvin, 1988, p. 77.)

1.2 Research problem and objectives

Paperboard and paper material dominate a significant share in the packaging industry due to their flexibility, cost-effectiveness and environmentally friendly nature. However, they possess drawbacks when compared to other packaging material such as plastics, mainly when converted into different packing profiles. Paper and paperboard possess poor formability. Starch-based coatings are brittle and might contain defects in their convertibility. This study will focus on the effect of creasing and calendering on barrier coated paperboards, such as clay and latex, which have its advantages and disadvantages. For instance, the biggest, latex coating drawback is its price compared to other coatings such as extrusion Polyethylene (PE). Findings on how to improve the creasability of barrier coatings will be advantages of this thesis studies. The following questions will lead to determining creasability issues;

- What is the effect of latex in the coatings on substrate creasability?
- How does calendaring affects substrate convertability?
- What is the effect of bulk on substrate roughness and creasability?
- How does different drying strategy after coating affect substrate creasability?

1.3 Research methods

This thesis is the practical base, which investigates the effect of creasing, and calendering on barrier properties of dispersion coated paperboards. In addition, material creasability is studied. Basic material properties such as bulk, density, grammage, air permeability, roughness, strain break (% elongation) and tensile strength of selected paper were measured and analyzed accordingly. Creasing was carried out using Kongsberg XE 10 digital creasing and cutting table whereas barrier properties were determined using Oil and Grease resistance (OGR) and Cobb60 tests.

2 LITURATURE REVIEW

A review of the literature was carried out to find the link and approach required for determining the effect of creasing and calendering on barrier properties of dispersion coated paperboards. Paper and paperboard manufacturing process, properties, coatings, calendering, creasing and folding were studied through literature.

2.1 Paper and paperboard

Morris (2011) defined paperboard as a normal paper material with a thickness ranging around 0.25 mm to 0.30 mm based on local standards. Most of them are made by cylinder type or multiple-pass Fourdrinier machines. There are different grades of paperboards ranging from chipboard of recycled and low-grade paper fibers to lined paperboard, with one or two surfaces of high-grade paper fibers or clay coated sulphate boards with highly treated fibers. They provide good strength with excellent food contact surfaces while maintaining a high quality even after being bleached.

Paperboards are made with different materials, and their nature will influence their mechanical properties to the finished board. Correspondingly, bending of board stresses the surface layers more than the interior of the board. Therefore, the paperboard should possess good properties such as stiffness, strength, and surface printability. These properties are important for packaging design and graphic and end-use aspects. Mostly, top layers should be made from Kraft pulp to enhance stiffness. The middle layer is based on chemi-thermomechanical pulp (CTMP) that provides bulk, but is not as strong as kraft pulp (StoraEnso, 2015). Paperboards are required to retain its strength and other surface properties during converting process without propagating surface cracks (Morris, 2011, p.115-116).

There are different commercial paper products based on their manufacturing methods. There are papers made through layering of fibers which makes them possess increased fiber length through their cross section. This orientation dictates how the paper will appear when stressed or after application of moisture on its mechanical changes. Since the main components of kraft pulp are cellulose and hemicellulose, paperboard appears hygroscopic and viscoelastic. Then, the effects of stress and strain will depend on conditions of time, temperature, humidity and rate of loading which makes it difficult to mathematically predict its properties (see Fig.1 that illustrates the fiber orientation CD and MD directions in paper) (Morris, 2011, p. 113-114).

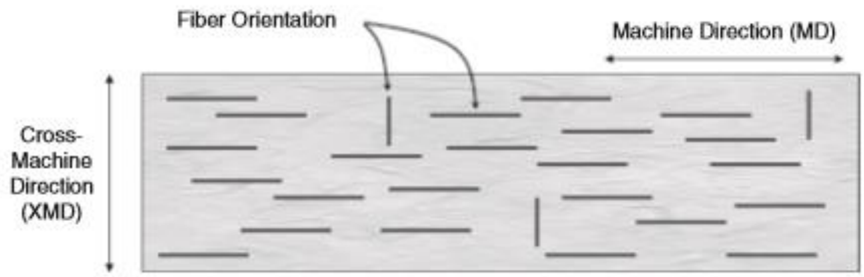


Figure 1. Fiber orientation MD-CD directions in paper making (Morris, 2011, p.114.)

Both paper and paperboard based packaging fulfil the needs of effective packaging, since they prolong the shelf life of products, provide information to users, and protect the goods from mechanical damages and spoilage. From a design perspective, customers are attracted by the graphical and structural aspects which aids the sale of the desired product. Additionally, paperboards possess considerable important features such as recyclability, renewability, biodegradability and outstanding printability. Similarly, these materials can be manufactured anywhere in the world using wood fibres at friendly cost. Despite these payback features, converting of paperboard materials is more complex compared to thermoplastics material which is much easier. The effectiveness of paperboard is reduced by moisture issues, barrier properties and is limited to adopt 3D profiles (Vishtal, 2015, p. 3)**.** However, flexible 3D packages can be made from paperboard with certain shape limitations with the aid of several processes including die-cutting, creasing, folding and forming (Kirwan, 2013, p.3-5.).

Paperboard is perceived as a heterogeneous composite material in which its structure together with its surface has huge impact on its final use properties. Additionally, process on paper surface such as calendering may be applied to improve surface gloss and smoothness, which are both important from the viewpoint of printing. Moreover, the calendering process affects the thickness of paperboard, where bulk also decreases. The structure of paperboard also has impact on printability and runnability issues (Vernhes et al., 2009, p.5204.) An extensive industrial paper and paperboard material needs to fulfil the market demands depending on fiber alternative. Pulps can be bleached or unbleached, mechanically-beaten or kraft-cooked, or made from recycled paper. Figure 2 represent the structure of (a) Solid bleached board (SBB) and (b) solid unbleached board (SUB) (Kirwan, 2005, pp. 23-24).

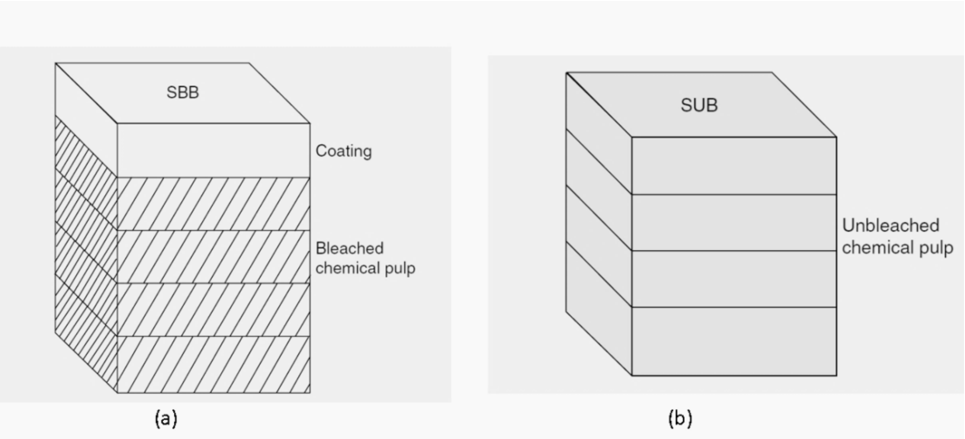


Figure 1. SSB and SUB (Kirwan, 2005, pp. 23-24).

2.2 Fracture properties of paper

Barbier et al., (2012.) analysed cracking behavior between folded and creased board. Their research took into account the defects of coated paper when creasing is applied for folding process. A clay pigment and ground calcium carbonate were used, followed by a binder which was SB-latex or a mixture of SB-latex and starch. In their approach to creating an analysis model, they found out that the pigment samples showed huge effects on cracks during creasing and folding process for thick paperboard. Meanwhile, clay coating revealed substantial crack zones compared to Ground Calcium Carbonated coating; this is due to binder pigment interaction. In this case, SB latex binder showed more promising potential when compared to pigment types. Based on the study stipulated by Barbier et al., (2012, p.5), there is a need to focus more on coated materials and determine its effects during converting process.

Correspondingly, according to Kim et al., (2010.) it was revealed that the key challenges in paperboard making process is the defects on creasing zone caused by huge pressure of around 100 N applied during creasing (Kongsberg XE 10). Coated paperboard manufactured with latex having less glass transition temperature, with small particle size, improves its foldability, lessens cracking and gives good printability. However, it is recommended that coated paper should be folded cross direction to avoid poor foldability, because of the fiber orientation which influences its properties (Kim et al., 2010. p.843). Creasability is one of the key processes to consider, since both paper and paperboard materials require special care during converting process.

Tensile strength of paperboard is influenced by manufacturing process either mechanical or chemical pulps. Inhomogeneous distribution of paperboard fibers can cause surface defects, voids, weak surface areas and lower tensile strength (Nazhad, et al., 2000, p. 1). Usually, the first failure occurs on defects zone and major defect account for risks of the shared load structure. For minor outer stresses, the defect is constant, which prevents the structure from cracking macroscopically. However, the outer stress tends to increase the defect, which will concentrate at the surrounding of the defect zone, causing cracks and thus affecting the efficiency of paper. Understanding the fracture mechanics of paperboard helps to eliminate cracks and defects during converting process. In addition, it increases efficiency during designing process like folding and creasing aspects, which are managed by eliminating possible defects. Figure 3 illustrates the fracture modes of paper (Niskanen, 2012, p. 177.)

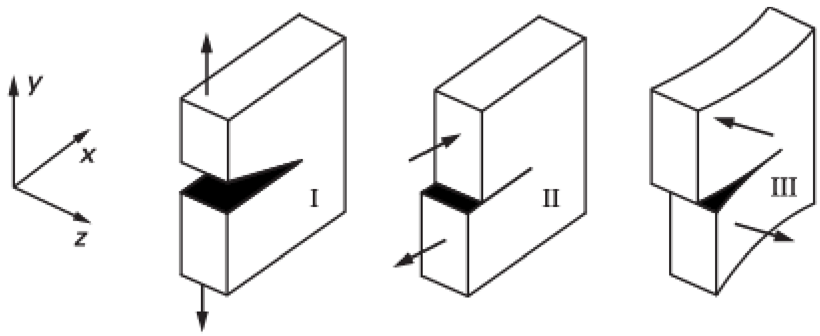


Figure 3. Major fracture modes as classified in 1. 11 and 111 respectively (Niskanen, 2012, p. 69).

2.3 The role of Barriers properties

Packaging materials such as paperboards require barrier coatings to prevent the products against unwanted stuffs either through or into paperboards packages. These coatings play a significant role in shielding packaging from substances such as water, oil, grease or oxygen. Additionally, they prevent mechanical deterioration that may develop when paperboard is saturated with liquid materials. Animal fat, plant oil, and grease substances are present in an extensive range of products posing a considerable challenge for packaging and makes OGR test a demanding aspect for paperboard in food packaging. Furthermore, some companies are developing new barrier coatings for paperboard. This new approach can be used to replace perfluorocarbon based coatings in paperboard. In high performance, OGR paper coatings where long-time experience, hot oil and folded packaging lead to more demand on the coating. The technology is compatible with common coating techniques and exhibit unique film formation properties. The solution prevents the surface of paper fiber from oil wicking through the paper. Hence, it covers a wider range of applications corresponding to performance target (McCulloch et al. 2017).

Previous research has revealed that, the designed starch based coating can be used in high-quality inkjet printing with dye-based ink, parallel with preventing the movement of foreign mineral oil into package mostly applicable for hybrid printing (Ovaska, et al., 2017, p. 1).

The use of packaging that contain outstanding barrier properties performance have been on the global increase, particularly for perishable foods such as vegetable preservation. Additionally, end user requirements accessibility together with food safety has stimulated the expansion of the use of barriers in packaging sector (Petrie, 2006, p. 1). Figure 4 presents how barrier works at different coating layers. Without the barrier, the penetration of foreign substances through the coating is easy. Whereas, with barrier, the penetration is more difficult through the coating, hence the product is protected

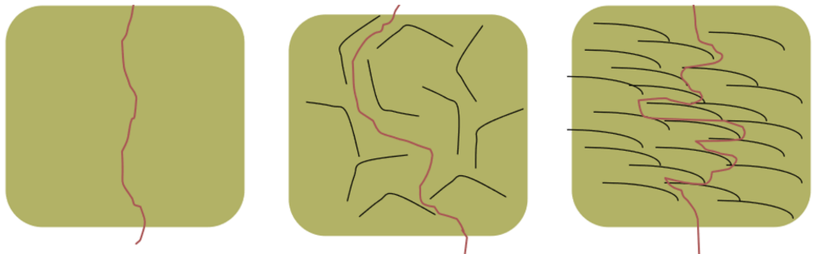


Figure 4. Shows how barrier works modified by author (Santos, 2010).

2.4 Dispersion coating

Conforming to definition, dispersion coating is expressed as a technique of applying metering and drying latex on paper and paperboard with the intention to produce nonporous film which is uniform with specified barrier possessions (Kuusipalo, 2008). Polymers used in barrier dispersion differ completely from the polymer used in extrusion coating, though they share the same term with barrier polymer coating. Examples include EVOH, latex dispersions and polyolefin dispersions. Latex has long been used in paperboard for coating colours as well as coating and laminating films. Latex is an emulsion, oil in water emulsion and polymeric compound. Paperboard grades coated with barrier-grade latex are pulpable in a conventional way, such that, broke may be slushed in a machine pulper and reprocessed without additional treatment. Commonly used polymers are polymetacrylates, polystyrene, polybutadiene, polyacrylates and polyvinylacetate together with polyolefins. Since packaging and wrapping sectors demand barrier against grease and water, dispersion coating sound promising to fulfil the demands. In addition to that, it is better for alternative use of inline or outline formations (Kuusipalo, 2008, p. 60-63.)

Previous studies in the field of paperboard materials converting have pointed out the convertibility and formability of various dispersion-coated biopolymers. The experiment focused on changing the creasing tools and creasing forces with different profiles. The base paperboard used was the same while barrier dispersion was altered. The pilot dispersion coated paperboard web was converted in the packaging line, manufacturing creasing blanks followed press forming to make trays. The objective of this study was to clarify demanding steps in the converting of biopolymer dispersion coated paperboard packaging substrates, focusing on die cutting and creasing operations. The forces applied on barrier coated paperboard during creasing and die cutting may initiate cracks in the coating, hence decrease barrier properties of the material. Starch based coatings have challenges with their convertibility as a result of the brittle structure of the coat (Tanninen et., al, 2015, p. 336,339).

There are different coating pigments binders and additives, which must be considered to accomplish excellent surface printability and durability, thus can withstand folding forces and another converting process such as creasing of paperboard material. The substance used for making latexes are chemically classified into three groups; which are styrene butadiene (SB), styrene n-butyl acrylate (SA) and poly-vinyl acetate (PVAc) (Sonmez et al., 2011, p. 1193). However, it has been stipulated by previous research that, currently there are also bio-lattices available which can be used for partial replacement of synthetic latex. These materials display exceptional, rheological, bounder properties and water retention that provide superior coater runnability (Bloembergen, et al., 2014, p. 69).

Dispersion coatings dry physically through ingestion of coating into substrate and the vanishing of water. This drying is typically accomplished through hot air and infrared. With the drying, water is removed from the material. The polymer particles approach each other and frame the covering film. Water-based coatings, because of their many favourable circumstances, have become the main innovation in the printing business with attributes like: ecologically inviting, scentless, dull, no yellowing and lessening of shower powder. Figure 5 illustrates the dispersion coating and drying techniques used in paper and paperboard (Actega,2017).

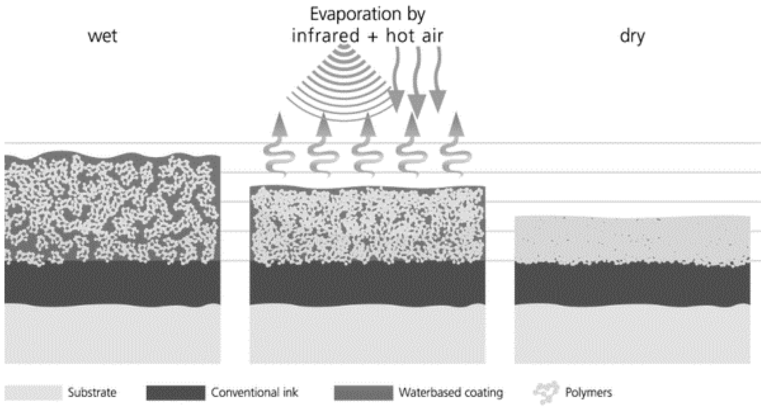


Figure 5. Illustration of dispersion coating (Actega, 2017).

2.5 Calendering

Calendering refers to as a technique of producing smooth surface of paper or paperboard sheet/substrate, by controlling the thickness and pressing the dry sheet between cylinders. Normally, calendering is utilised in different means based on design aspects of the product, and at least one of cylinders is heated such that it can be applied to the surface of substrate. Calendering involves at least two rolls example paperboard which need light calendering to regulate the thickness of substrate without squeezing the material excessively. Various paper grades require calendering to make the best use of it. The aim of utilizing calendaring is to improve surface properties of paper such as glossiness, smoothness, density, blackening, brightness and opacity (Kirwan, 2005, p. 18-19). Calendering of paper and paperboard is a manufacturing technique that is adopted to smoothen the external part, enhance glossiness and printability (Vernhes et al., 2009, p.5204).

Calendering of paper and paperboard: The topology properties of both papers and paperboard have significant benefits during converting process. For example, sizing or covering has an effect on the gloss appearance of the external part of paper and paperboard. Meanwhile roughness defines the surface height differences as well the paper and paperboard quality. Calendering lessens the surface deviations in general length scales and little alteration on the crossover length (Vernhes et al., 2009, p.5204). Figure 6 demonstrates cross section view of calendering process and how the thickness changes.

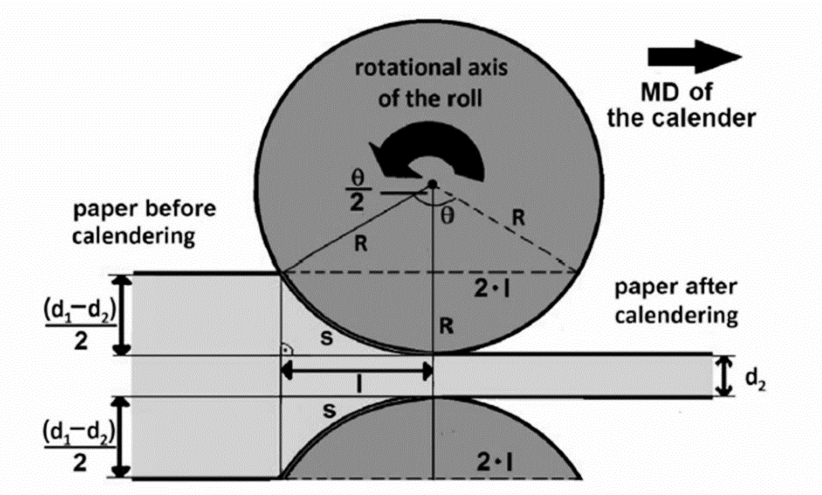


Figure 6. Cross section view of paper being calendered on two calender rolls machine (Schlordt, et al., 2014, p. 4921).

Paperboard have various applications in packaging sectors, thus having an impact on the surface of paperboard for a particular use, deemed to result in improvement so that the properties imposed on it are valid. One approach used to improve quality is the calendering process; this method helps to smoothen the surface fibers in the base of paper, or produce an even thickness, smooth surface and polished finish of paper or paperboard. In addition to that, Super-calendering in some cases is used to increase the smoothness of paper sheet or paperboard surface when required. In this method, the paper is pressed between fiber and steel rollers under huge pressure. Then, moisture is added and a lower roller is motorized, the other rollers offer slippage and thus iron the target paper. The outcomes are an even finished and glossy surface (Robertson, 2013, p.177). However, there is loss of bulk after calendering process, which is not desired in case of paperboard. Increased density makes converting more difficult and in some cases, it is impossible. Additionally, stiffness may suffer from calendering (Sim, et al., 2012, p. 447 and Sonmez, et al., 2011, p. 1193).

2.6 Creasing and folding

According to the definition, creases are expressed as lines formed on the surface of substrate or paper and paperboard surface that define the border of a folding line(s), hence it permits effectiveness of the folding process for the desired object (Amigo, 2012, p11). In correspondence to previous studies, it was revealed that the moisture content of the paperboard has a significant effect on tensile strength, stiffness, creasability, foldability, and when moisture is high, the deformation reduces (Rhim, 2010, p. 244). Additionally, paperboard must be creased if the material will be formed in a 3D shape using press-forming approaches. First, the creasing pattern is designed according to tray requirements, such as location of creases, width of creases and number of creases required (Tanninen, et al., 2015, p. 5191). With specified forces, the crease rule is pressed on board to create a groove which produces plastic deformation that permits the substrate to be folded as required without damage or cracks. During creasing process, substrate delaminates to thin layers, it is necessary to prevent it from bulging out from the profile. Bending and compression forces rise as the stresses rise in the creasing area. However, creasing settings of substrate characteristics, creases preparation and setting are considered as key factor that influence the effectiveness of creases. Apart from creasing, various adjusting settings can be applied rather than cutting. An efficient crease is obtained adequately deep along with narrow ones that offer excellent folding accordingly. Then, cracking issues on board is maximally eliminated. Thickness of substrate and grades are the key factors to take into account for creasing tool setting. The deepness and width of a groove, size of creasing rule and how much the rule should penetrate into a groove should be considered. Crease rule profile and edges are also variables in the creasing rule selections (Kuusipalo, 2008, p. 253.)

Creasing through machinery is made using creasing rules. The rules are to use thin strips of metal that have smooth rounded edges for pushing the surface of the paperboard to make a groove on board. Care must be taken to avoid cracking during creasing through the blade after pressing. Furthermore, in order to prevent tearing of layers outside the fold, the paperboard is partly delaminated to make a thin layer so as to control folding and deformation of the paperboard.

There are different types of paperboards with different properties; therefore, different adjustments must be applied to obtain good quality of the crease. Additionally, there are deep and shallow creases based on the specific pressure adjustments for the quality of the board. Also creasing is easier on bulky paperboard whereby the size of the crease increase with a certain base weight and stiffness degree. Therefore, the surface of the paperboard must have resistant surface layers. Those layers can be bent and the areas

for bending are coated with a pigment where creases are supposed to form (Sonmez, et al., 2011, p. 1193.)
Depending on creasing definition, creasability can be explained as the effectiveness of materials to undergo folding process after creasing is applied on it without fractures or damage based on design aspects. Similarly, creasability can be defined as the degree to which material can be creased without fracture or any failure based on design aspects. The aim of applying creasing on paper and paperboard is to allow smooth folding of object during converting process.

Creasing alongside folding are mutual converting processes of paper and paperboard to package. Folding depends on creasing during creasing process, blank holder and creasing rule utilised to facilitate the creasing. Depending on package design or requirement, some demand multiple creases and some require single creasing line per press. This means that the crease tool magazine can be with multiple rule or single. There are benefits of utilising creases before folding process, this helps to lessen the risk of package damage or cracks during converting process and hence provides efficient folding for package. Similarly, it helps to attain compression strength and good runnability (Tanninen, 2015, p.15). Nagasawa, (2003) described creases as a significant mechanical property in paper and paperboard converting process. However, the paper and paperboard deformability is not the same as those from isotropical elastoplastic. Crease line can be made parallel with cutting process of boards thus influence the productivity of converting process (Nagasawa et al., 2003 p.157).

The major objective of creasing is to eliminate folding failure and defects in the folding zone. The effectiveness of folding relies on excellent creasing process. The process is done when the creasing rule is applied into substrate surface placed against a die notch or when a groove is being formed. Huge compressive and shear on substrate stresses is released, but, delamination materialises on limited zone where by the bending stiffness is lessened (Niskanen, 2012, p.59).

In the construction of various package profile of different designs, for instance, designs of bags and sachets, cartons and solid fibre board cases, paperboard are frequently folded in the construction of pack shapes. The thinner materials are folded mechanically through 180°, where the resulting folds are rolled to give permanence. The thicker materials for cartons and cases require a crease to be made in the material prior to folding.

The material to be creased is supported on a thin sheet of material known as the make-ready or counter die put on a flat steel plate. Grooves are cut in the make-ready to match the position of the creasing rules in the die. When the die is closed, creases are pressed into the surface creating a groove in the surface of the paperboard and a bulge on the reverse side (Kirwan, 2013, p.42). Crease forming in this way subjects the material to several forms of stress, which are indicated in Figure 7.

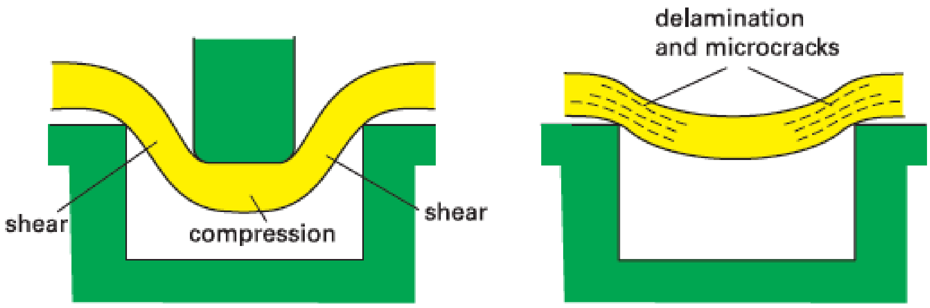


Figure 5. Shows the crease forming (Niskanen 2012, p.59)

Previous studies determined that barrier coatings may generate cracking during folding and creasing process (Barbier et al., 2012). Pigment coating is recommended to have sufficient elastic properties to prevent failure on the crease lines and hence effective folding (Sonmez, et al., 2011, p. 1194). Within the process of the folding of the crease, the topmost layers of fibre on the exterior of the resulting fold are extended in order to have a proportionate tensile strength and stretch. The core layers are compacted to make a confined delamination. As the folding continues, the reverse side bulge develops in the form of a bead to the required angle like a hinge. Figure 8 shows the behavior of creases in the folding process. Folding must guarantee that the bulge itself does not rupture or become distorted so that the layer of fibre on the rear side will appear as excellent strength properties.

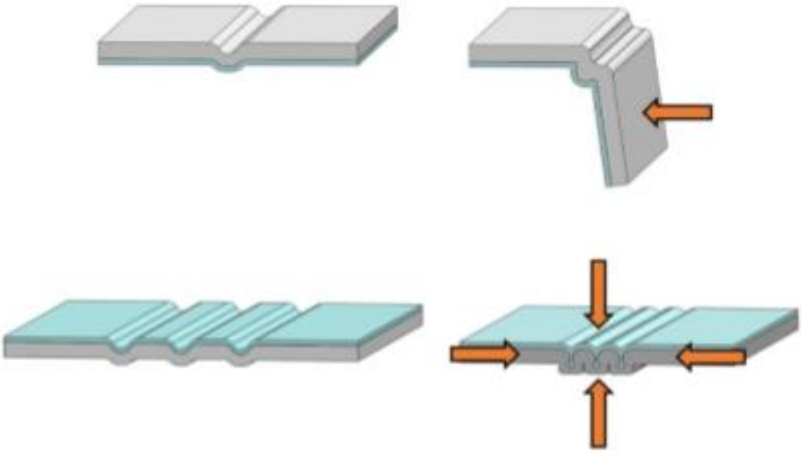


Figure 6. Demonstrates the difference between traditional folding and functionality in press forming (Tanninen, 2015, p.18).

Excellent quality of coated layer that enables effective printing is a key factor that makes coated paper to mostly used printing substrates. In addition to its outstanding benefits such as productivity and maximisation of various surface treatment, particularly varnishing, both paper and paperboard still have some drawbacks. One of such drawback is surface cracking during converting process (Pál, et al., 2017, p. 100.) Fig. 9 demonstrates the cracking on unprinted and printed sample latex coatings paperboard in machine direction.

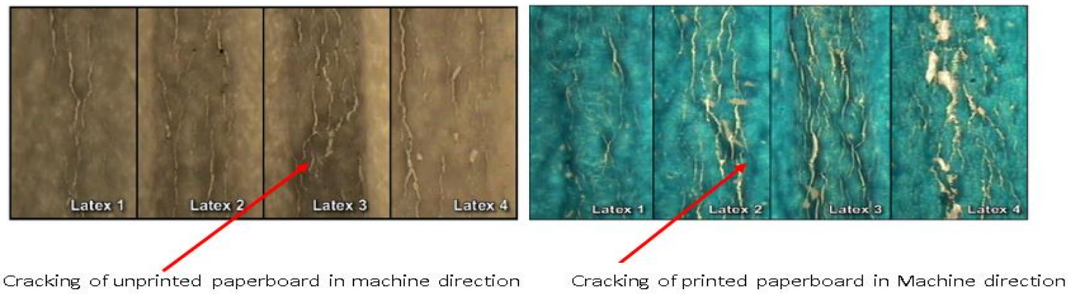


Figure 7. Cracks on unprinted and printed latex coated paperboard (Kim et al., 2010, p.846.)

Fibre orientation on paperboards

The orientation of paper fiber in the direction of wire can make them tear along the direction where most of the fibers are laid because of less energy to separate. This orientation has made most printed papers easy to tear in one direction from top to bottom and impossible to tear in straight line at right angles to that direction.

Similarly, the effects of moisture make most of cellulose fibers to swell or contract in their diameter than lengthwise. Correspondingly, in the machine direction, fiber orientation makes the paper to curl and buckle along its axis of orientation, and swell when one side become wet as illustrated in Figure 10 (Morris. 2011. p. 114-115.)

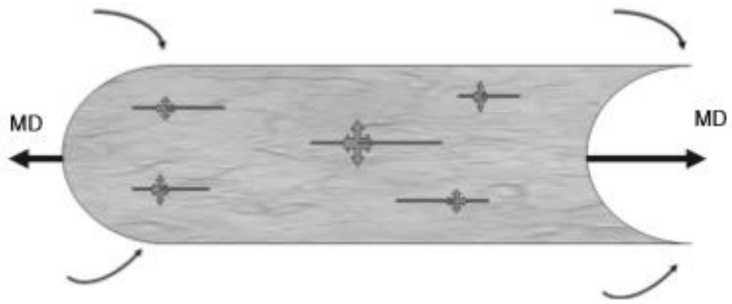


Figure 8. Effluence of moisture in paper based on CD and MD directions (Morris, 2011, p.115.)

2.7 Press forming of paperboard

Paper and paperboard as packaging materials are famous due to their flexibility; nearly all papers are converted when additional treatment is applied after basic production process is complete. Laminating, impregnating, saturating together with package such as bags, trays, boxes and pouches are few converting process of paperboard materials. Additional surface treatment that includes adhesives, calendering functional products and pigments, are frequently used based on the final applications of the particular paper (Robertson, 2013, p.177.)

In converting paperboard into packages that are accountable by users, creasing lines should be efficient and foldable. Effective creases result in successive folds; therefore, creasing and folding are twin converting processes. Excellent crease comprises delamination which is generated from creasing, and intended to lessen the bending stiffness and protects paperboard profile against cracks during converting process (Beex, et al., 2009, p.4192.)

Correspondingly, the main objectives of a paper converting process are to obtain final products with desired properties such as strength and stiffness. Package manufacturing stages consist of several procedures after the substrate has been printed. For instance, cutting and creasing are done with specific creasing machine, which performs both operations inline (Kuusipalo, 2008, p. 249). Figure 11 illustrates a paperboard tray, which has been made from paperboard material and has passed through various converting processes.

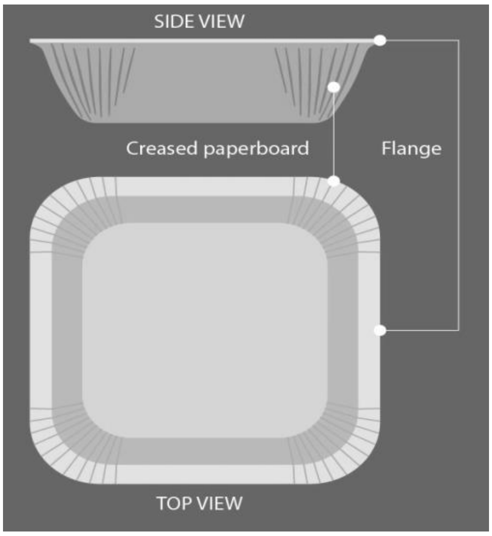


Figure 11. Paperboard tray (Iggessund, 2017).

In case of creasing and folding, the coating should remain crack-free and the loss of barrier properties should be minimal. Majorly, cracking of barrier coating propagates during creasing and folding processes. Particularly, when compressive or tensile strength is applied, it is favoured by coating layer and folding direction (Annushko, 2013, p. 39). Previous study determined that the convertibility of water base barrier coatings (WBBC) is very promising. These material properties play exceptional role of grease resistance particularly when measured in horizontal position. Furthermore, the study revealed that PE offer promising barrier properties after creasing. The new approach to elastic barrier dispersions possesses promising creasability as well suitability for disposable packages (Miettinen, et al., 2017, p. 1). Figure 12 presents the common defects over the coating layer: (a) propagation of cracks when substrate is folded (b) imperfections propagation when folded.

Typical example of fold-cracks over the coating layer (a) and base paper weakening during the folding process

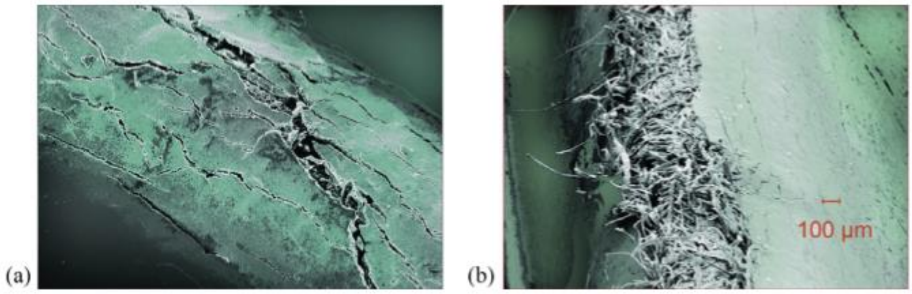


Figure 9. Illustrates the common defects on the folding zone behaviour over pigment-coated layer (a) and (b) defects propagation during folding (Magdolna, et al., 2016, p. 100).

EXPERIMENTAL PART

3 METHODOLOGY AND RESULTS

This chapter is divided into three major parts. The first part focuses on Experiment I and discusses physical properties of coated paperboards. Experiment II deals with creasing tests of paperboards and the third part illustrates experiment III. The Last part contains the Cobb 60 and OGR tests of selected samples. Figure 13 illustrates the experimental flow chart of this study.

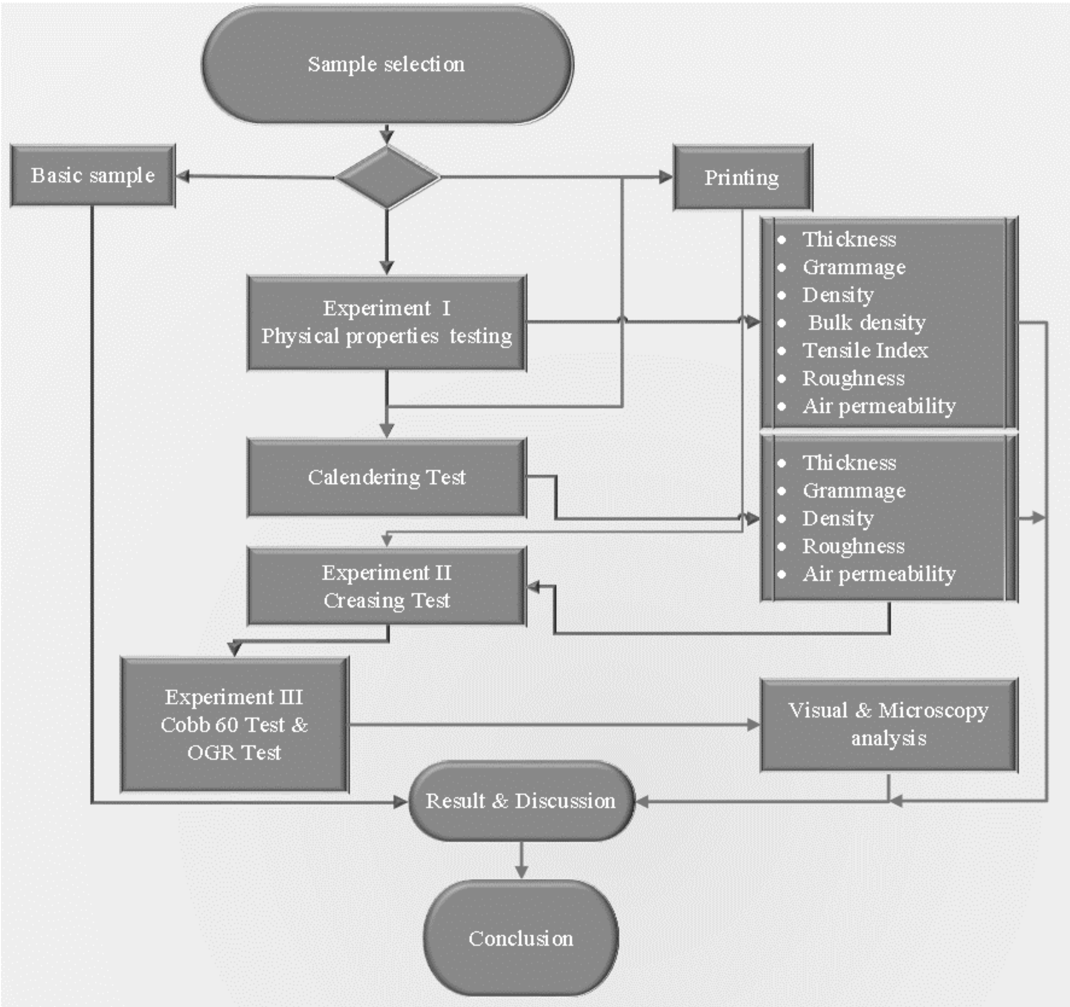


Figure 10. Experimental approach flow chart.

3.1 Experiment I

This experiment focuses on coated paperboard samples. Thickness, grammage, density, roughness, air permeability and tensile index are measured from the coated paperboards.

Materials and methods:

Eight different coated commercial paperboards were used in this study. Base material for the experimental coating was uncoated paperboard designed for tray applications and was used as a reference material and marked as REF 1 later in this study. REF 2 was a commercial dispersion-coated paperboard and REF 3 was a commercial PE-coated paperboard with pigment coating on the reverse side. The reference products were compared with the experimental samples 1, 2, 3, 4, 9, 10, 11 and 12. Their technical names are presented in Table 1.

Table 1, Technical names of selected samples.

Sample No.	Technical name
REF 1	SBS board, AKD-sized, designed for tray applications
REF 2	Dispersion-coated SBS board
REF 3	PE-coated SBB board with a mineral coating on reverse side
1	S100 F
2	S100 R
3	S45K55L30F
4	S45K55L30R
9	S60K40L30F
10	S60K40L30R
11	K70L30 F
12	K70L30 R

Where
S = Hydroxypropylated starch
F=Forward-intense drying (mostly IR-based)
R=Rear-intense drying (mostly air-dryer-based)
L= SB-latex
K= Kaolin

3.1.1 Coating

The dispersion coating was done on a base substrate of commercial solid bleach sulphate paperboard (300 gsm), with the operation of pilot coating machine. The blade coating unit and rollers are the main part of the machine. Machine speed of 100 m/min generated a smoother one-side layer coating on paperboard (backside) and the goal was to reach a weight of 8 gsm. The main constituents of dispersion coatings include:

- Starch – Solcoat P55
- Kaolin – Calypso BX GR
- Latex – Styron HPW-184

The latex had glass transition temperature (T_g) of -9°C and mean particle size of 160 nm. Kaolin clay was pre-dispersed using 0.1 parts per hundreds (pph) of dispersant with additive of 0.05 pph sodium hydroxide (NaOH) on dry solid ingredient of 45 %. A commercial scale dispersant was used for mixing the coating dispersions and the pH and Brookfield viscosity was determined right after the mixing of coating dispersion. The composition and values are depicted in Table 2.

The drying process of dispersion-coated paperboard was done in two ways: a) forward intense drying and b) rear intense drying. The forward intense drying is the exposure of coated paperboard to a fast heat shock produced by infrared dryers followed by a moderate air float drying, whereas, the rear intense drying is inverse to the stipulated above technique. First exposure to mild infrared drying is preceded with high temperature float web drying. These are the optimized drying solution top bio based dispersion barrier coatings. Normally, binder is calculated based on 100 pph pigment (Parts per Hundred) in coatings; though there are exceptions for test points 11 and 12 where kaolin and latex together are 100 pph.
All samples were cut from A3 to A4 size, followed by cutting to 0.02-meter square for measuring grammage, thickness, density and bulk calculations. Structural properties for chosen samples will be presented later in a table.

3.1.2 Reference material 1 specification

The physical properties of Ref 1 are presented in Table 2. The surface roughness was determined using Bendtsen roughness tester, and SI unit is presented as (ml/min) Number of milliliters of air escaping per minute. While the coating layout of Ref 1, paperboard, is presented in Figure 14.

Table 2, Ref. 1 material

Ref 1	
Thickness	410 µm
Grammage	300 g/m^2
Top side smoothness Parker print surf (PPS)	400 ml/min
Reverse side smoothness (PPS)	500 ml/min

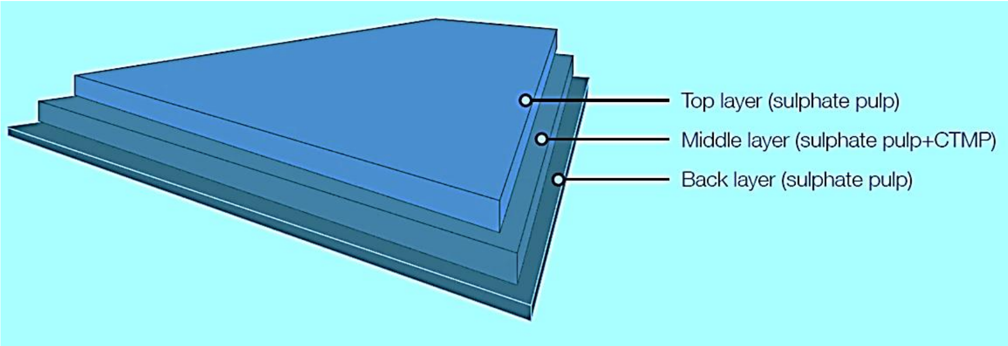


Figure 11. Illustrations of Ref 1 layout layers.

3.1.3 Reference material 2 specification

Ref 3 paperboard is made from sustainable wood fibre as base material. It is a dispersion barrier coated which is suitable for food packaging. These paperboards offer a potential resistance against grease, water vapour and water. Furthermore, the coatings used in REF 3 are appropriate for heat-sealing and water-base gluing/hot-melt gluing. Table 2 represents the basic properties of Ref 3 paperboard material.

Table 3, display the basic properties of Ref. 2 material.

Ref. 2	
Thickness	450 µm
Grammage	290 g/m^2
Cobb test- up side	40 g/ m^2 (60 s)
Cobb test – rear side	7 g/m^2 (30 s)
Smoothness Parker print surf. (PPS)	1.3 µm

Figure 15, illustrates the barrier dispersion coated layer of Ref 3 paperboard

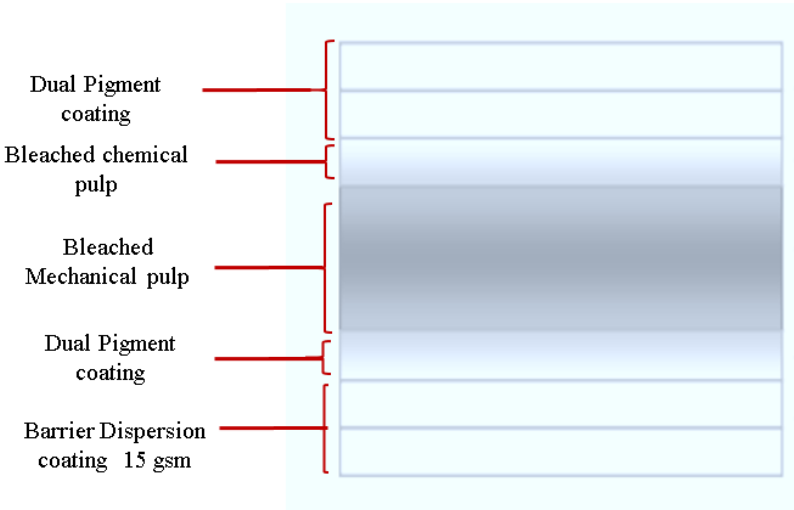


Figure 12. Illustrations of Ref. 2 materials.

3.1.4 Reference material 3 specification

Ref. 3 is a polyethylene-coated paperboard. The material is applicable for packaging usage in which moisture barrier properties is important. Furthermore, the PE coatings possess excellent means of package sealing. The structure of the material is shown in Figure 16.

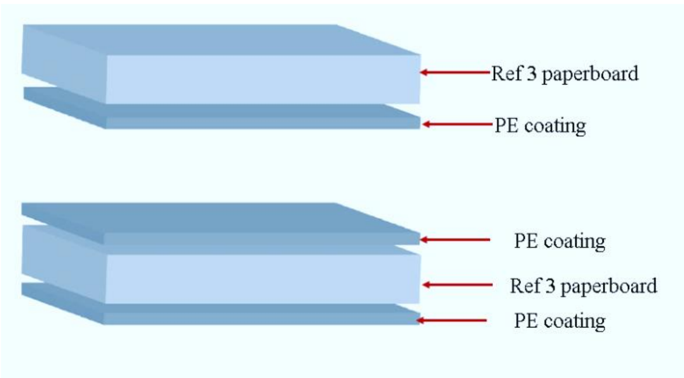


Figure16. Structure of Ref. 3 materials

3.1.5 Methods

The key objectives were to test the significant parameters for barrier properties of dispersion-coated paperboards. Thickness, grammage, apparent density and bulk density were tested based on the following standards: SCAN-P 7:75 for density and thickness and ISO-536 for grammage. Furthermore, the roughness and air permeability was measured accordingly.

3.1.6 Grammage

The area of trimmed sheet is 0.02-meters square (0.02 m^2) which is used to determine the grammage of the sheet. The equation is described as follows;

$$Grammage\ (W) = \frac{(m)\ Mass\ of\ sheets\ in\ gram\ (g)}{Area\ of\ one\ sheet.m^2(A) * n\ (number\ of\ sheet)} \qquad (1)$$

3.1.7 Thickness, density, bulk, roughness and air permeability

The thickness of paper is usually measured in micrometer (µm). Bulking thickness is the inverse thickness of one sheet calculated from the thickness of a pad of sheets. The thickness was measured by using a motor driven micrometer (Lorentzen &Wettre) and data was measured from one point per each paper sample. The average was calculated to obtain the desired thickness of each sample. The basic properties of the paperboard sample materials used in this study are presented in Tables 3 and 4, and were measured under standard conditions (23°C and RH 50 %).

$$Density = \frac{grammage(g)}{thickness\ (m)(t)m^2} \qquad (2)$$

The SI- unit is reported in (Kg/m^3). Bulk is inverse of density value (cm^3/g)

The grammage of reference materials varied between 285 and 301 gsm, whereas the experimental samples grammage were between 300 and 307. The coat weight of experimental samples was in the range of -1 to 6 gsm, but the result cannot be considered reliable due to high variance of basis weight in the case of Ref. 1. The roughness surface roughness was determined using Bendtsen roughness tester, and SI unit displayed as (ml/min) Number of millilitres of air escaping per minute. The values varied between 153 and 401 ml/min meanwhile the air permeability ranged from the least value of 0.69 to the highest value of 5.79 (µm/Pa s). The roughness values decreased with respect to change in line load. The bulk of paperboard materials decreased concerning increasing line load. Materials 1, 3, 9 and 11 were dried by using infrared, full-powered, and cooling was moderate controlled. The results of these drying methods showed decreased roughness and air permeability value significantly. Materials 2, 4, 10 and 12 were dried using moderate infrared power and cooling was increased gradually; the roughness and air permeability were a bit higher when compared with materials 1, 3, 9 and 11.

Table 3, Grammage, bulk, density roughness and air permeability values obtained from samples.

Samples No.	Grammage (g/m^2)	Thickness (µm)	Bulk (cm^3/g)	Density (kg/m^2)	Roughness (ml/min)-mean	Air permeability (µm/Pa s)-mean
REF1	301	378	1.256	796	226	5.79
REF2	298	298	1.429	700	153	3.43
REF3	285	326	1.144	874	462	1.55
1	301	384	1.256	784	298	0.69
2	300	384	1.280	781	401	1.92
3	302	377	1.248	801	269	0.73
4	305	384	1.536	651	374	3.88
9	307	383	1.247	802	228	1.80
10	307	369	1.202	832	223	3.89
11	305	327	1.072	933	294	2.33
12	307	379	1.235	810	269	1.93

Table 5 illustrates the mechanical properties of selected samples as obtained from experiment test. 10 MD and 10 CD represent the number of sampling tests. The tensile index of reference material was between 44 and 76 Nm/g in MD whereas in CD the tensile varied between 21 and 38 Nm/g.

Table 4, Tensile index and strain break of coated paperboards.

	Tensile Index (Nm/g)		Strain Break %(Elongation)	
Sample No.	MD	CD	MD	CD
REF 1	76	35	1.78	4.73
REF 2	44	21	1.35	3.59
REF 3	69	38	1.99	5.46
1	81	48	1.88	4.09
2	77	37	1.69	4.58
3	76	36	1.76	4.34
4	93	45	1.73	4.85
9	78	37	1.82	4.98
10	77	35	1.77	4.66
11	81	37	1.94	5.15
12	81	36	1,90	5.17

3.1.8 Calendering experiment

Calendering was carried out to smoothen the samples surfaces in a bid to discover its influence on the thickness, bulk, roughness, air permeability and density. Figure 17 shows the LUT laboratory calendering machine used for this study.

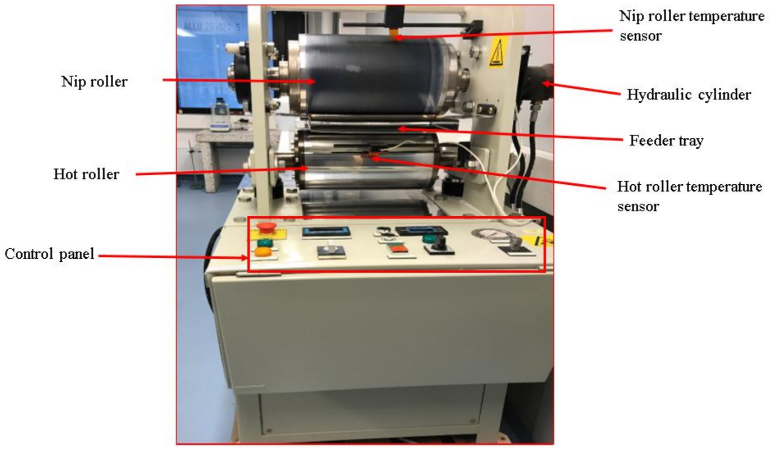


Figure 17. LUT Laboratory Calendering machine.

Experimental set up:

The coated side of reference and experimental sampling were positioned against hot roller (metal cylinder), while the uncoated side was positioned on polymer roller (nip roller). The target calendering temperature was set between 80 ± 10 ^{0}C and varying pressures were set to 27 kN/m, 75 kN/m, 99kN /m and 123 kN/m, respectively. At first, the width ratio of samples was calculated by measuring the width of samples and divided by a constant number provided in the lab, which was 0.3 m. Sample width was about 21cm since all samples were cut to A4 size.

With the aid of Equation 3, the width value ratio of 0.7 was obtained. Meanwhile Equation 4 guided the calculation of the line load values with relevance to pressure bar.

$$Width\ ratio = \frac{Sample\ paper\ width\ (cm)}{Standard\ paper\ width\ (\ cm)} \tag{3}$$

$$Line\ load = \frac{Nip\ load\ (\frac{kN}{m})}{width\ Ratio} \tag{4}$$

Table 6 shows the calculated line load values based on pressure bar setup. The ratio obtained will be used to calculate line load for calendering setup. Since target values 25, 50, 75 and 100 kN/m were not obtained, a close value to that was used in this study.

Table 5, shows the line load calculation data for calendering.

Pressure bar	Nip load (kN/m)	Paper width ratio	Line load (kN/m)
10	18.7	0.7	27
20	52.3	0.7	75
25	69.1	0.7	99
30	85.8	0.7	123

Figure 18 displays the comparison of basic and calendered sample´s thickness. The thickness of each sample was measured with six different points and the average was calculated. Thickness effect on each sample decreased considerably as it was influenced by variation of pressure applied on paper during calendering processes. The experiment revealed that basic sample possesses higher thickness values compared to calendered samples. However, since grammage and density depend on thickness, potential effects were also noted. A significant observation within this experiment is that there was practically no major difference between 99 and 123 kN/m whereas the 27 kN/m value had the greatest proportional impact.

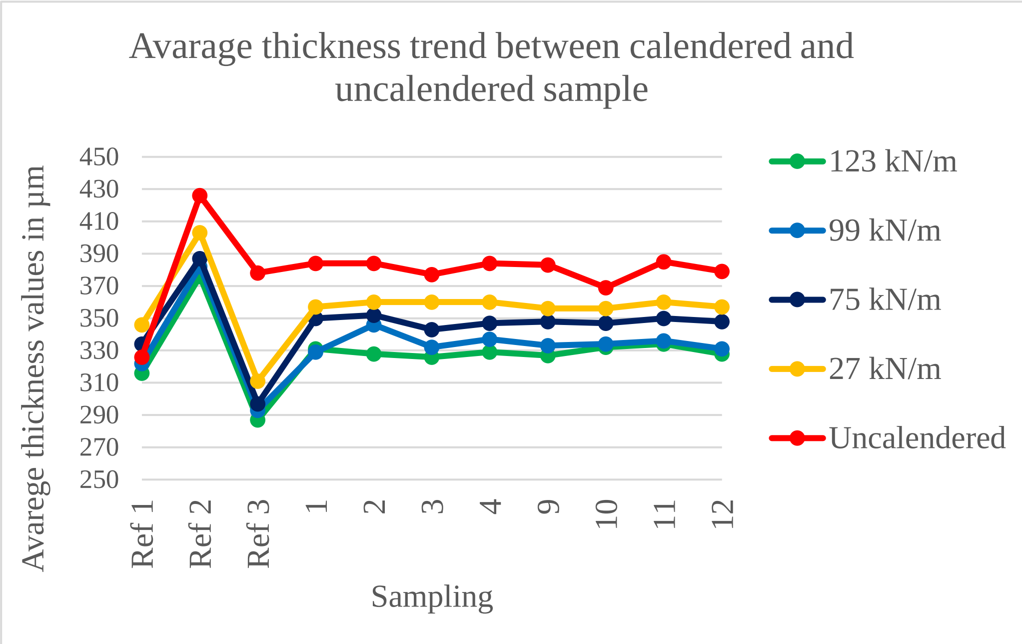


Figure 18. The thickness comparison between uncalendered and calendered samples.

3.1.9 Roughness

Roughness is defined as the quantity of air that flows through the paper surface and metal ring that is set to the paper when the differences are set toward unit time. The roughness values of selected samples were measured and observed on Bendtsen instrument accordingly. The Bendtsen instrument is equipped with the dimension of the measuring head and bulb with contact pressure of that area of the paper and the metal ring of 98 kN /m^2. The roughness values are presented in Figure 18, based on SCAN-P 21:67

Based on the data obtained from these experiments, the roughness values of uncalendered and calendered samples were compared. Uncalendered roughness values were huge compared to the calendered samples. The objective of calendering of paper is to improve the surface smoothness, glossiness and maintaining the general thickness of paper (Vernhes et al., 2009). The effect of calendering was noticed as the roughness values became less when compared with uncalendered samples. For instance, uncalendered sample (Basic sample) 1, roughness value was 298 ml/min and for calendered sample 1 it was 158 ml/min. With these two values, the study revealed that there is a significant influence of calendering on surface of sample papers. However, the change in calendering pressure effect was observed as moderate. Mostly, the 27 kN/m had the greatest proportional effect on material. Meanwhile 99 and 123 kN/m did not provide major difference on sampling as Figure 19 illustrates.

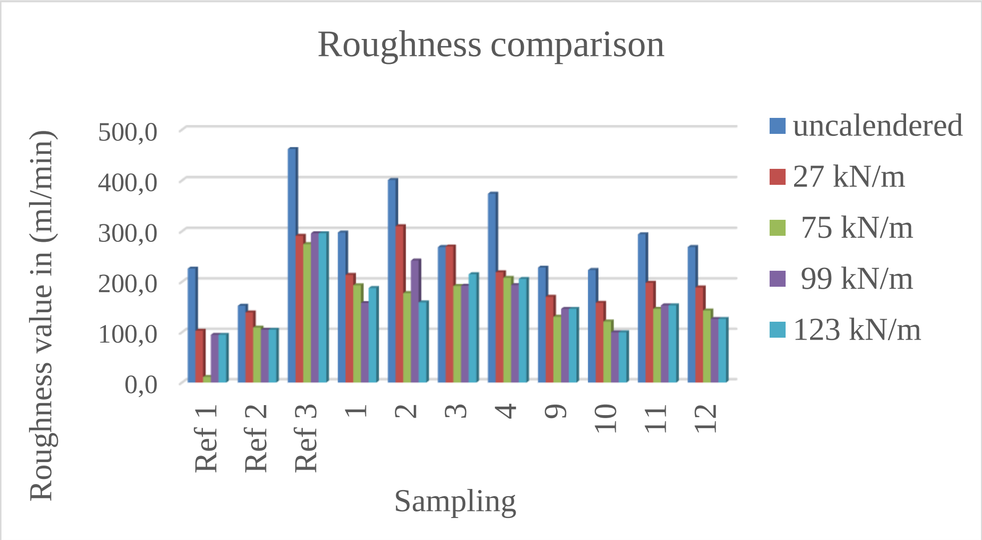


Figure 19. Roughness values trend for calendered and uncalendered sample.

3.1.10 Air Permeability

The air permeability is considered as one of the essential parameters, which describe the ability of a material to resist the gases that flow between package and product. This data can be used as an indicator of the barrier properties of packaging material.

The air permeance of selected samples were measured using air leakage instrument known as Bendtsen tester. The flow of air was measured through a defined area of sample papers which was defined by the difference in pressure of 1.47 kilopascal (kPa) between the different sides, 10 cm^2, of samples; this measurement is based on recommended standards of SCAN-P 60:87, for air permeability. An average of six consecutive measurements for each test points was automatically calculated with the Bendtsen tester. Based on observations, it was clear that uncalendered sample possesses huge air permeability values, when compared with calendered samples. The trend behavior of air permeability values for both calendered and uncalendered are displayed in Figure 19. Generally, observations indicate that the calendered sample test possess least value of air permeability. However, the air permeability values for calendered sample have moderate effects over each other. The smaller air permeability values indicate better air permeability resistance. The decrease in air permeability was huge in many cases as displayed in Figure 20, it can be recommended to use only small line load.

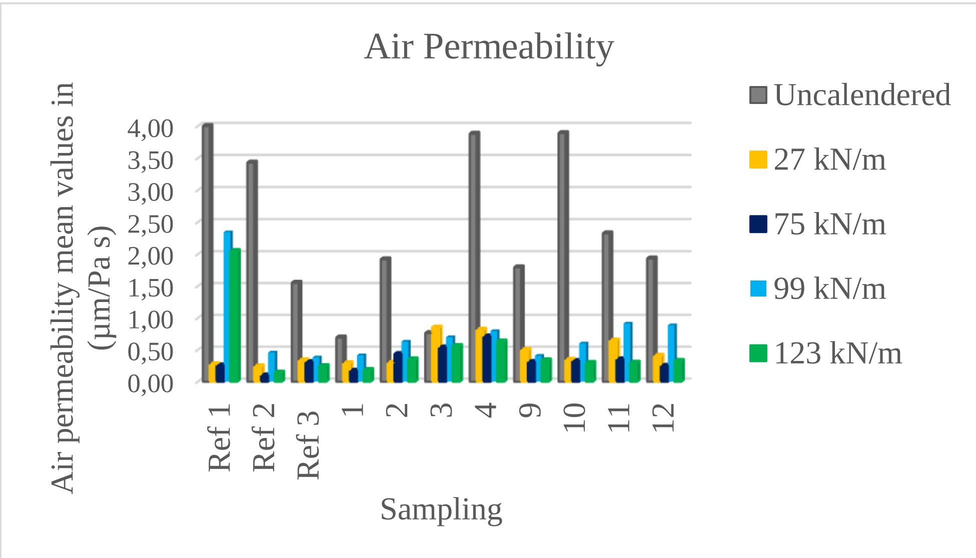


Figure 20. Air permeability trend

3.1.11 Tensile properties testing

The tensile strength and elongation of selected samples was measured with horizontal tensile machine test and setup load cell of 1000 N, clamping force of 1000 N, and consisted of test work and measuring programme based on SCAN-P 38:80. Data obtained was printed instantly from machine and tensile index, and strain break was recorded. Important parameters in this test were Machine direction, cross direction speed (mm/minutes), grammage of each sample (g/m^2), thickness in (µm) span and width in this case which was 15 mm. 10 pieces from each sample test were tested based on machine direction and cross-sectional direction, respectively.

The breaking strain compared to the tensile strength is presented in Figure 21. As is illustrated in the graph, the REF 1 paperboard has tensile strength index of 76 Nm/g against breaking strain of 1.78 in MD. The maximum tensile strength of paperboard sample 4 was 93 Nm/g value whereas the breaking strength was 1.76. This may be due to the drying strategy because drying of materials 2,4,10 and 12 was moderate while the cooling was gradually increased. Furthermore, the minimum value of tensile stregth is 44 Nm/g with the braking strain of 1.35 (REF2). The graphical illustration presents the mechanical behaviour of 11 different paperboard materials. However, comparison of all materials based on its breaking strain and tensile strength were done in machine direction.

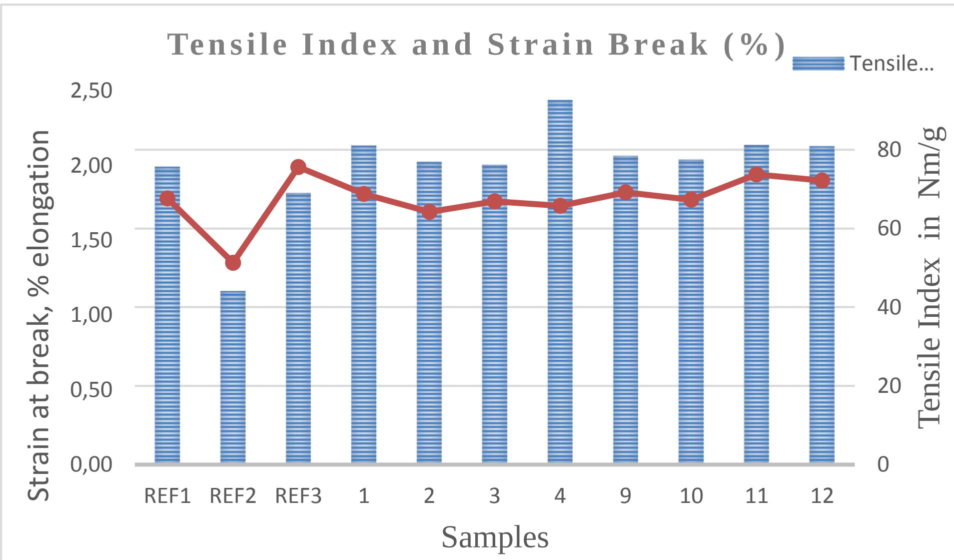


Figure 21. Tensile Strength (Nm/g) and Breaking strain (% elongation) in machine direction (MD) of different samples.

The tensile properties and breaking strain (% elongation) in cross direction (y axis) is presented in Figure 22, the mechanical properties of allocated samples were calculated and compared with breaking strain. The sample 1 paperboard has the highest value of tensile index, meanwhile the REF 3 possess high breaking strain, whereas the lowest values belong to REF 2 paperboard. The trend of mechanical properties of different materials is discussed in this graphical depiction with material's fiber orientation in cross direction. The difference in drying strategy for materials 1,3, 9 and 11 if compared with materials 2,4,10 and 12 is very minor in this case.

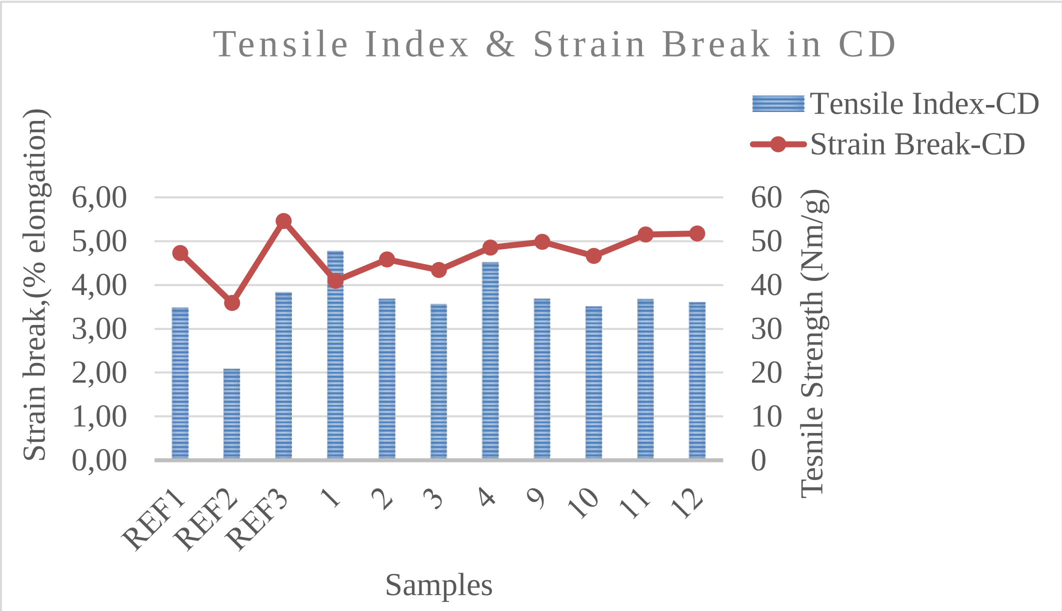


Figure 22. Tensile Strength (Nm/g) and Breaking strain (% elongation) in cross direction (CD) of different samples.

3.1.12 Summary of Experiment I

The calendering tests were successful, based on calendering experiment results. It was revealed that calendering process has huge effects on sample properties. Uncalendered and calendered thickness values were calculated and compared. In calendered samplings the thickness decreased gradually with respect to change in line load [kN/m], while the operating temperature was kept constant. Additionally, smoothness and glossiness were investigated visually, and the results were promising. Considering that the density and bulk of samples depend on thickness, the influence of calendering was clear. Based on calendering objectives, barrier surface properties of dispersion-coated material can be improved by calendering process, but a loss of bulk can be expected.

Based on values obtained after roughness test of selected samples, the study reveals a significant effect on each sample when compared. Uncalendered sample showed high roughness (Figure 17). Additionally, calendered samples reveal moderate effects on each other. For instance, the calendered sample for 123 kN/m test possess least roughness values compared to other tests. Thus, as it was expected, the roughness changes depending on the pressure applied on calendering process. The barrier properties of dispersion coated paperboards and the roughness values were influenced by calendering process.

As expected, air permeance decreased after calendering which was ascribed to densification of the paperboard. The effect of coating composition, however, on post-calendering air permeance was only minor. This might be due to relatively small coat weight or similar compressibility properties. The roughness and Air permeability values can be linked with calendering outcomes and it is concluded that calendering process can be used to modify the permeability of dispersion-coated paperboards.

The breaking strain (percentage) and tensile strength both in CD and MD is presented in the Figure 23. The bars in red and blue illustrate tensile strength index of individual sample, whereas lines with pointer in grey and yellow show the tensile strains of allocated samples. The comparison of tensile strength is complemented with its breaking strain in percentage. Based on value abstained for % elongation it seemed there is minor effect of drying methods for materials 1,3,9 and 11, the average was 1.85 whereas materials 2,4,10 and 12 averages was 1.775.

The maximum Tensile strength in the machine direction is 93 Nm/g of S45K55L30R. The minimum tensile value in MD is 44.13 Nm/g as seen in Ref. 2. The highest value of tensile strength could be related to starch, which has been shown to increase tensile strength in both directions (Biricik et al., 2011, p. 3154). Moreover, the S100F has higher values in CD 47.72 Nm/g. The minimum value achieved during a tensile test in CD was 20.82 Nm/ and is depicted in Ref. 2. Furthermore, breaking strain was also calculated. The highest value of Ref 3 is 5.6% in CD which represents the fiber strength before breaking. The highest value in MD is 1.99 % as depicted in Ref 3. Based on these mechanical properties, the criteria of material selection can be defined for specific applications.

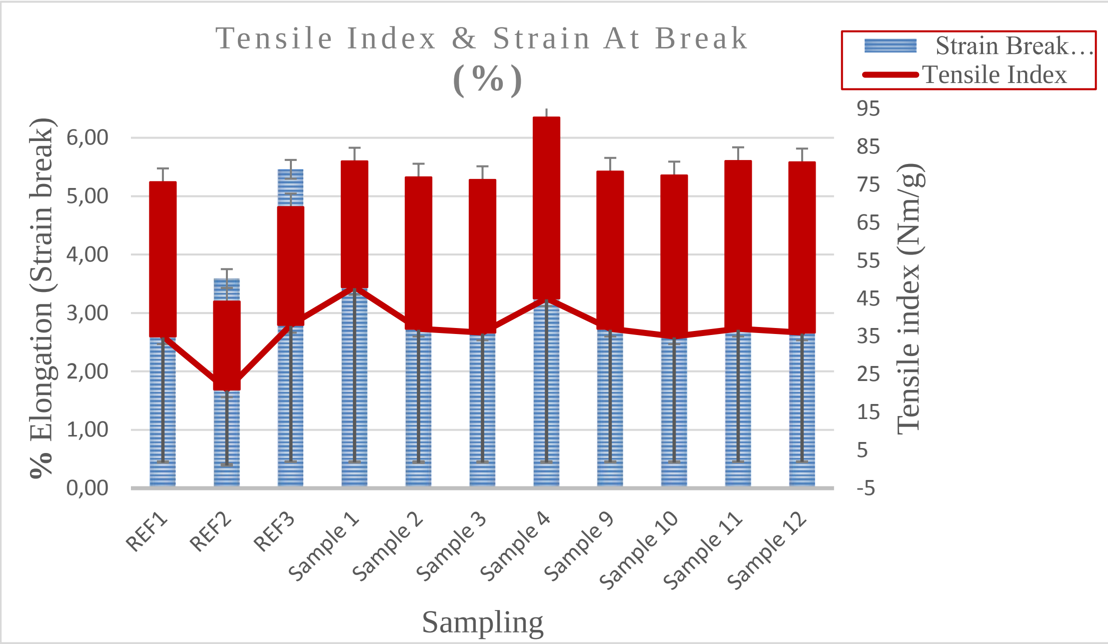


Figure 23. Tensile Index and % Elongation (Strain break) behaviour.

Generally, based on experiment 1 tests conducted for barrier properties of dispersion coated paperboard, the properties can be improved by calendering processes. The thickness and roughness were improved and led to promising convertibility process such as creasing. However, a loss of bulk will occur, which is detrimental to paperboard convertability.

3.2 Experiment II

In the second part of this thesis, creasing experiments are conducted. This section will deal with crease test for coated paperboard samples and further microscope image analysis will be discussed. A creasing experiment is designed on a single tray corner and machine direction or cross direction of sample boards.

3.2.1 Creasing Set up

Calendered, uncalendered and printed coated paperboard samples were creased with different Kongsberg creasing tools of 2 and 6 points. 2-point crease tool wheel diameter is 26 mm and creasing width is up to 0.7-1.0 mm as well as creasing depth, whereas 6-point creasing tool wheel is 26 mm, creasing width is up to 1.5 mm as well as creasing depth. These tools are suitable for creasing paperboard of 250-400 gsm. Two paperboards of A4 size from each sample were creased on single tray corner and machine direction or cross direction of sample boards. The creasing operation was done on allocated samples using Kongsberg XE 10 Machine which is an automatic cutting and creasing tool with computer aided setup (STL file compatible), and suitable for sample making and short run production of tray blank, boxes and similar products. The specifications of the machine are presented in Table 7. The creasing tool and maximum pressure were engaged to make sure all creasing pressure is absorbed; samples were taped on machine table during the creasing process. Starch base coating which are brittle defects were expected with their convertibility. Figure 24 denotes the creasing pattern used for creasing test.

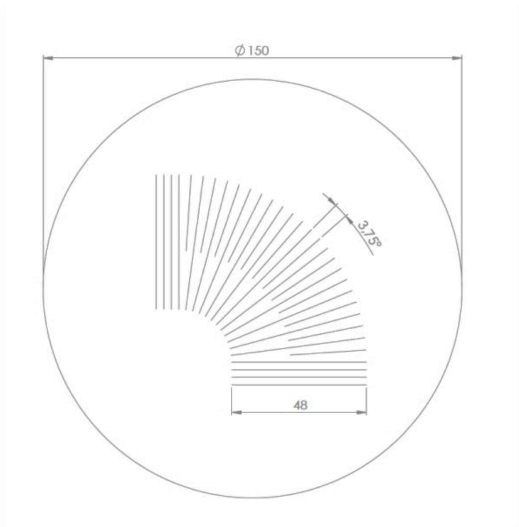


Figure 134. Creasing pattern used

Table 6, Kongsberg XE10 machine specifications

Parameters	Values
Total dimension	1630 *1580 mm
Work area	800*1100 mm
Weight	175 kg
Speed	64 m/min
Acceleration	12 m/s^2
Vertical creasing force	100 N
Horizontal cutting force	200 N
Repeatability	±20 μm

Kongsberg X10 machine is presented on Figure 25.

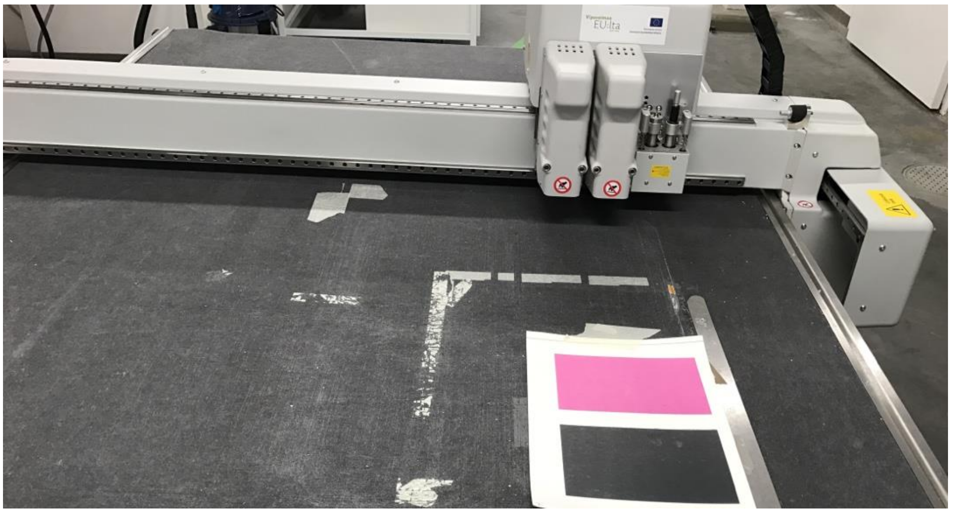


Figure 25 Sample layout on Kongsberg XE Creasing machine creasing preparation.

3.2.2 Visual analysis of calendered and creased samples

After basic and calendered sampling were creased, the next task was to inspect the sample visually to determine if there was any effect on creased lines. With visual inspection, no significant differences between sampling were observed. Figure 26 shows the outlook image of creased sample 4.

Figure 14. Creased sample 4 with 2 points creasing tool.

3.2.3 Microscopy image analysis

Light microscope Wild- M400, Xcam1080P equipped with top view software was used to inspect the defects on sample surfaces. The LM magnification was set to 1mm which was the minimum size.

The objectives of conducting the light microscopy images analysis was to find out possible cracks on creasing lines. However, the creasing tool width could influence the cracks size. After visual inspection, samples 1, 11, REF1, REF2 and REF3 were selected for microscope image analysis. The sample images were analyzed with light Microscope. As displayed in Figure 27, there were no significant differences between the creases in microscope images.

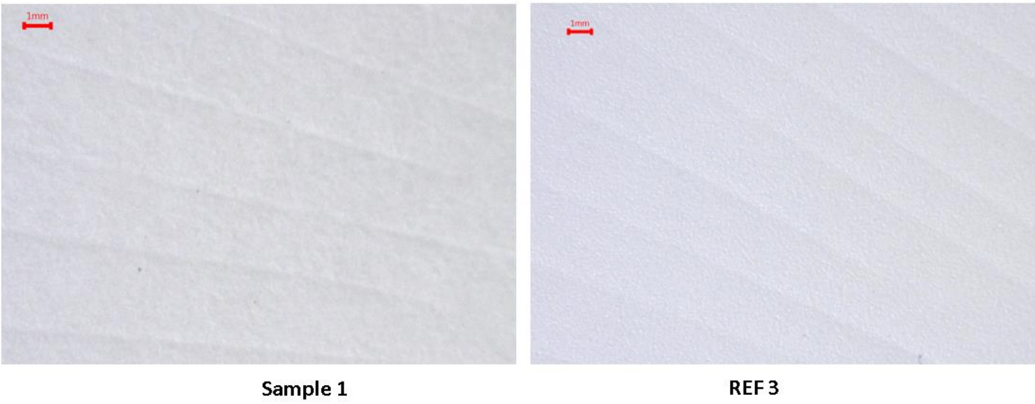


Figure 15. LM images of sample 1 and REF 3 creased with 6-point tool, no significant differences between creases

The microscopy images of selected samples were investigated. The light microscopy images of coated and creased samples are presented in Figure 28. Visually, the crease line width was noticed.

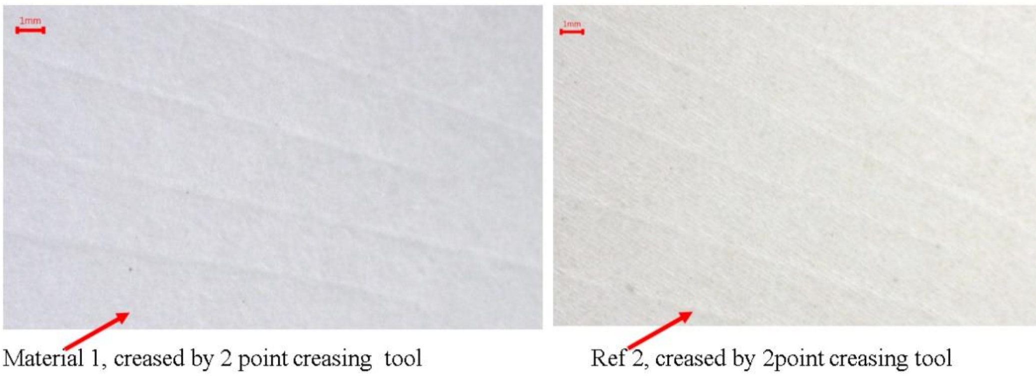
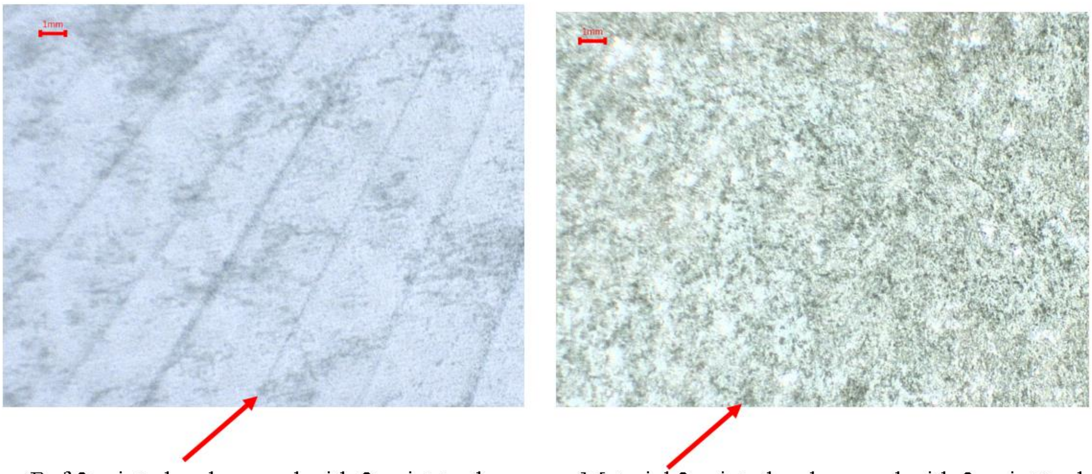


Figure 16. Creased sample 1 and REF2

The microscopy images of sample 11 and REF2 (printed and creased) are presented in Figure 29, Ref 2. Crease lines are visible compared to sample 11 where the crease lines are not visible though both were creased by 2-point tool. This is due to poor printing quality and probably is the effect of drying methods.



Ref 2 printed and creased with 2 point tool

Material 2 printed and creased with 2 point tool

Figure 29. Microscopy images of sample 11 and REF2 (printed and creased)

The microscopy images were taken from selected samples accordingly. The cracks were suspected to occur on crease lines. The creasing tool was oriented equally on sample surfaces. Only the tool width was changed to see the effects of creased groove and cracks propagations. Light microscopy scale of 1 mm seemed to be large and not suitable to detect crack surface on sample. However, the poor printing quality probably caused by coatings and adopted techniques influenced the expected results. The propagation of cracks could supposedly be influenced by creasing tool width. The method is not a sufficient/suitable way to analyse creased samples because no major difference was observed between samples and cracks noticed. Further advanced method such as SEM could be used for better results.

3.3 Experiment III

Water permeability of selected samples were investigated accordingly using the Cobb method. The objectives were to investigate the capability of selected samples to repel water.

Materials Standard blotting paper of 200 m^2, weighing 250 g, and deionized water was used; creased and calendered samples were cut to size to meet standard area of 100 cm^2 for testing water absorbency.

3.3.1 Cobb60 test

The defined cobb value is described as mass of water absorbed in specific time by 1 square meter of paperboard, corrugated fiberboard under 1 cm of water according (ISO 16532-1). The following apparatus was used in this experiment test.

- Stainless steel metal roller with smooth face of 20 cm width and 10 kg.
- Water absorption device for allowing one side of sample to be wetted consistently from the time soaking starts and equally helps to manage the removal of water after test ends.
- Graduated cylinder,100 mL.
- Stopwatch for controlled soaking time during test.
- Weight with an accuracy of 0.001 g.

3.3.2 Sequence of testing

All samples were kept at room temperature. Each sample was weighed before and after Cobb test. A 100 ml of deionized water with room temperature 23^0C was poured into ring as quickly as possible followed by a start of the stopwatch for two minutes per each test, after which the water was poured away rapidly. In case there was excess water during testing, the difference in time of removing unwanted water and blotting shall be 10±2 seconds. Since samples were coated with a barrier layer it is significant that the water film does not remain on the surface of sample test. It is possible that rollers and blotter may not be good enough to remove all the surface water from substrates, if this happens hand rubbing of the blotter paper should applied. Consequently, sample was reweighed instantly to note the weight change. With the aid of provided equation (6), the amount of water absorbed in grams per meter square [g/m^2] was calculated.

$$\textit{Weight of water, } (g/m^2) = [\textit{weight } (g) - \textit{Conditioned weight } (g)]x100 \tag{6}$$

Tables 8 and 9 present the Cobb test values for calendered and uncalendered sample, respectively. The values obtained by Cobb Test of different samples revealed a significant difference between the basic and creased calendering sample. However, the initial weight of each sample was less compared to final weight. This indicates that the samples absorb water considerably. The highest value of 41.45 g belongs to sample 1, whereas the lowest value of 0.85 was for REF2. When comparing experimental samples

1 and 11, the obvious reason for the different Cobb60 values is the coating composition. Sample 1 was coated with plain hydroxypropylated starch, which is a hygroscopic material. Sample 11 had a coating that comprised latex and kaolin, thus being biopolymer-free, and having a slightly more hydrophobic character due to high latex content (30 pph). Commercial samples REF1, REF2 and REF3 (calendered and creased) represent excellent barrier properties for water absorbency. Furthermore, samples 9, 10, 11 and 12 possess moderate absorbency values, compared to samples 1, 2, 3 and 4. The table illustrates the absorbency scale used in this study. Table 9 displays the Cobb60 test values for calendered sample. The smaller the values the better the results. The results of Cobb60 test values for basic sample (Uncalendered) are displayed in Table 8.

Table 8 Cobb60 test for uncalendered samples

Uncalendered Sampling	1		11		REF 1		REF 2		REF 3	
Absorption values	37.7	31.0	36.6	34.6	30.2	37.2	0.9	1.0	0.6	5.7
Average	32.35		35.6		33.7		0.95		3.15	

Table 9 displays the Cobb test values for calendered sample. The smaller value indicates better results.

Table 9 Cobb60 test values for calendered samples.

calendered sampling	1		11		REF 1		REF 2		REF 3	
Absorption values	33.1	29.1	32	31.3	30.5	27.9	0.8	0.9	3.3	2.4
Average	31.1		31.65		29.2		0.85		2.85	

The grading scale used in this study to evaluate the Cobb60 test value for calendered and basic sample is presented in Table 10.

Table 10. Absorbency grading scale.

Weight of water (g/m^2)	Absorption acceptance criteria
0-10	Excellent
11-20	Very good
21-30	Good
31-40	Satisfactory
>40	Poor

3.3.3 Summary of Experiment III

The values obtained by Cobb60 test of different samples revealed a significant difference between the basic and creased calendering sample. However, the initial weight of each sample was less compared to the final weight. This indicates that the samples absorbed water significantly. The study reveals that water absorbency for calendered samples are slightly less compared to uncalendered (basic) samples. For instance, the water absorbency values for calendered sample 1 was 41.1 whereas the basic was 41.5. Considering previous studies, it has been revealed that for different types of paperboard, Cobb test values decrease with respect to quantity of starch contents. This implies an increase in water resistance values (Biricik, et al., 2011, p. 3154).

3.4 OGR-measurements set up

The preliminary purpose of OGR is to investigate the capability of barrier coated paperboard to prevent oil penetration. Two commercial materials were selected for this study. Both Ref. 2 and Ref. 3 (without creasing and calendering) were tested in parallel with calendered and creased materials.

Palm kernel oil dyed with sudan red which followed the ISO 16532-1 standard with the setting temperature of 60 ^{0}C regulated in the oven. The tested specimens were cut to sizes of 5x5 cm. The pipetted volume of oil was set to 100 µl and the time consumed for parallel test was 1440 minutes. The coated sides of the samples were also tested according to the standards. Six parallel samples without the influence of weight addition were tested in order to analyze the penetration of oil with respect to time. Two samples were manually tested under room temperature to determine the oil penetration capability. The experimental setup of OGR is shown in Figure 30.

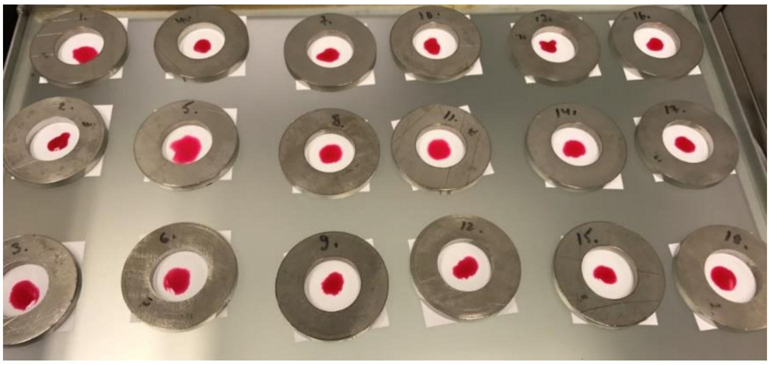


Figure 17. layout of sampling for OGR Test.

4 DISCUSSION AND ANALYSIS

This section will present the main results achieved during experimental tests as it was carried out at LUT packaging laboratory. The key barrier properties of dispersion-coated paperboards are discussed based on their influence on convertibility. Furthermore, the values are presented in Appendices I-IX.

4.1 Effect of calendering

Surface properties of paperboards are considered as a potential phenomenon in converting process, for instance the coating and printability of paperboard. The physical structure of paper and paperboard together with its surface possess potential properties to its end use. Calendering is considered as the finishing procedure employed to paper and paperboard material base to achieve smoothness, a glossy and shiny surface. The objectives are to manage the effectiveness of calendering and the performance of the finished product. It has been discovered by previous studies that paper and paperboard surface and structural conversions occurring during calendering process are essentially enduring (Vernhes et al., 2009, p.5204). This study reveals a significant effect of substrate thickness, and bulk decreases while the gloss and smoothness tend to increase compared to uncalendered sampling. The roughness and air permeability values of barrier properties of dispersion coated paperboards were improved after the calendering process. This indicates that to achieve better surface properties of paperboard there should be effective convertibility.

Considering the influence of calendering and creasing on barrier properties of dispersion coated paperboard, most of the results achieved have been linked with previous studies. (Effect of calendering on paper surface micro structure, image processing based on quality control of coated paper folding, studies on foldability of coated paperboard - influence of latex on foldability during creasing/folding coated paperboard, large bending behaviour of creased paperboard, effect of creases depth and crease deviation on folding deformation characteristics of coated paperboard, binder effects on the creasability of pigment coated paperboard and the unique convertibility of paperboard).

4.2 Creases and light Microscopy analysis

The dispersion coated paperboards were calendered and creased. The physical effect of creases and calendering operations on barrier coating were also analysed. For this purpose, two crease tools with 2 and 6 points width were selected to make the impression on the paperboard surface. The cracks around the creased surface of coated and uncoated paperboard were investigated with the aid of simple light microscope.

Visually, the crease lines were visible with good profiles extent, revealing no cracks on the surface. The crease depth can be differentiated based on its surface impression. To enhance the magnification and analyse the nature of cracks along the crease lines, a simple microscope with the magnification power of 1 mm was used. Microscope images indicate the position of paperboard crack lines. A higher power microscopy with magnification of 1 µm or less is required to analyse the nature of the rupture, crack length and propagation direction. Scanning electron microscopy (SEM) is suggested to further characterize the topology of paperboard.

In conclusion, light microscope can be used to identify the location of surface cracks and crease depth only at very rough levels. Starch based coatings usually with show brittle these materials can be investigated with other advanced methods to reveal the cracks since LM seemed not suitable to determine cracks. Moreover, it was also investigated that the crack propagation can be influenced by width and force of a crease tool. It is problems with their convertibility, which was expected. The suggested for clearer understanding of the above-mentioned studies to use high end microscope. For instance, Figure 31 illustrates the SEM and LM images comparison. (a) is SEM images of barrier coating and (b) is LM images of barrier coated creasability of paperboard, the significant details can be distinguished between the two. With SEM, the surface of sampling image is clearer; detailed and every single defect can be seen.

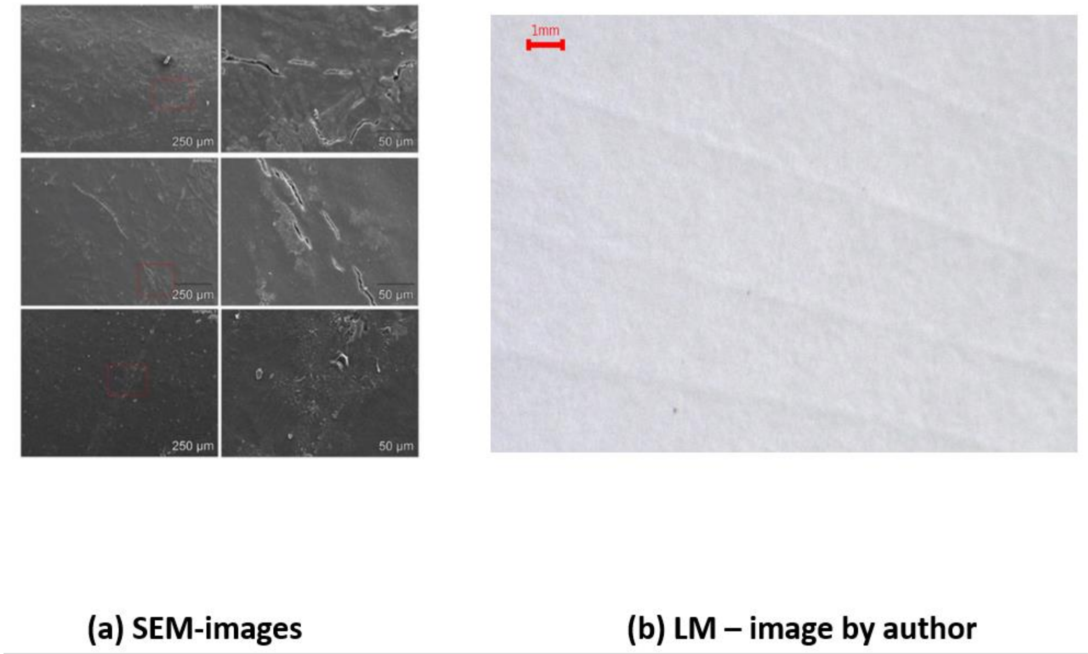


Figure 18. Illustration a SEM Images (Tanninen, et al., 2015, p. 340.) (a) and LM images (b) obtained from tests.

4.3 Oil and grease resistance tests results

The capability of barrier properties of REF 2 and REF 3 to prevent oil penetration with respect to time was investigated using ISO16532-1 at 60^0C. Samples were inspected based on elapsed time.

First, the oil and grease resistance for experimental samples 1 and 11 was tested manually to reveal the capability of oil penetration prevention into the material under the influence of time. In the case of the uncoated sample, significant penetration was found within a minute. Other uncalendered and uncreased experimental samples resisted grease by approx. 5-10 minutes, depending on coating composition. Furthermore, sample 11 penetration characteristics was same as sample 1. It can thus be concluded that barrier properties of dispersion coated paperboard for materials 1 and 11 OGR was not successful, it was unnecessary to proceed with further testing. However, it seemed that calendered sample 1 possessed good sign for OGR, but creasing is obviously detrimental (Figure 32).

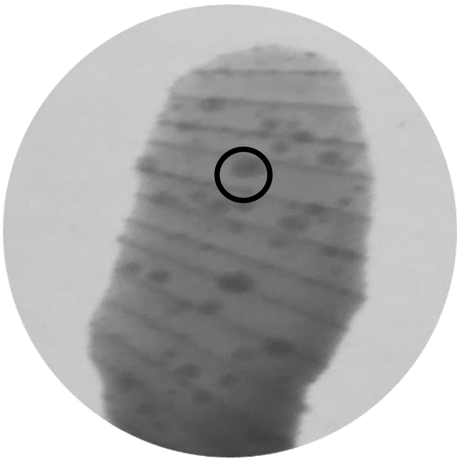


Figure 19. Sample 1 shows oil penetration on creased lines as marked on circle

The REF 2 possess promising oil barrier when compared to samples 1 and 11. This is due to the notion that Ref 2 had a synthetic dispersion coating; in this case, the coating weight was higher than Samples 1 and 11. Generally, the only few specimen was unsuccessful since minor penetration was found during the first six hours (360 min). Calendered and creased sampling OGR were successful as no penetration was revealed within 1440 minutes, and this indicated that the penetration resistance is greater than 1440 minutes. REF3 possess excellent OGR compared to previous material tested under similar conditions. The tests were successful: manor penetration was found after 1440 min, on basic Ref. 3. However, a minor leakage was found through a pinhole present in the plastic coating as shown in Figure 33.

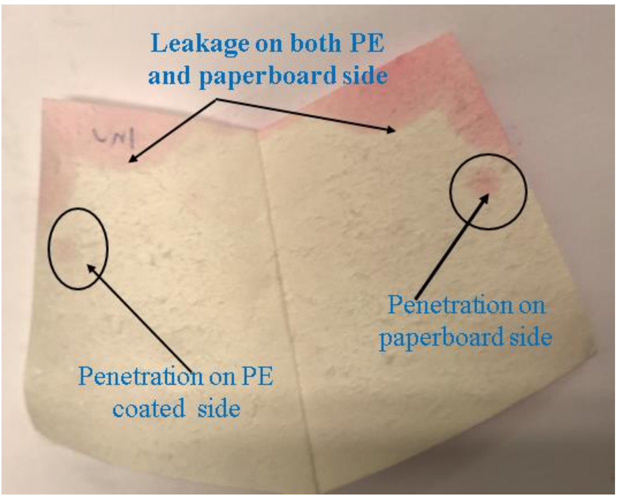


Figure 20. A pealed Ref. 3 showing oil penetration on PE coated and paperboard side.

Figure 34 illustrates the leakage on one side of sample material.

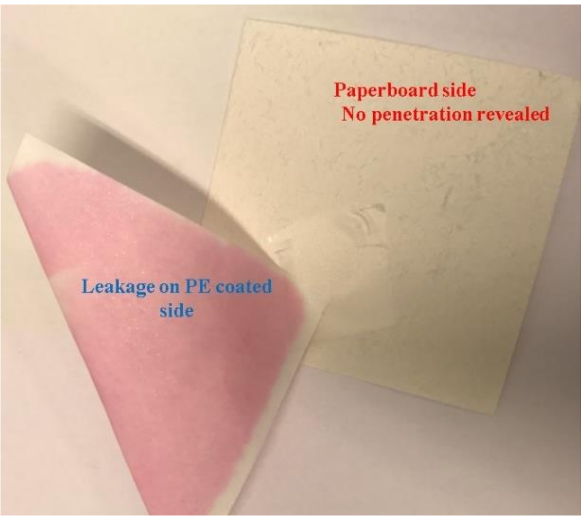


Figure 21. Illustration of REF 3 leakage found only on coated side

The penetration of oil into the coated surface is influenced by material roughness. The tests were carried out at 60°C, this implies that the temperature of palm Kernel oil is not solid anymore as it turns to liquid form. Usually, oil temperature and ratio of coating substance of materials influence the OGR (Ovaska, 2016, p. 51-52). (Prabhu, et al., 2009) studied the wetting kinetics of several different types of oils and observed that the low viscosity oils have higher spread rate, whereas the higher viscosity oils show lower spreading rate. Furthermore, the relaxation time for oil increases with increase in surface roughness. The performed experiment of OGR reveals that the penetration of oil can be improved by calendaring process: the penetration was greater at 1440 minutes. Furthermore, the velocity of the oil penetration was also high in dispersion coated paperboard (Ref. 2) samples compared to the commercial Ref. 3. A few of the samples were not following the general trend and as such were neglected. Creases can be another reason for higher penetration. The localized defects caused by creases enhanced the area to penetrate within the material. The two different paperboards were compared with respect to time and results are presented in Figure 35.

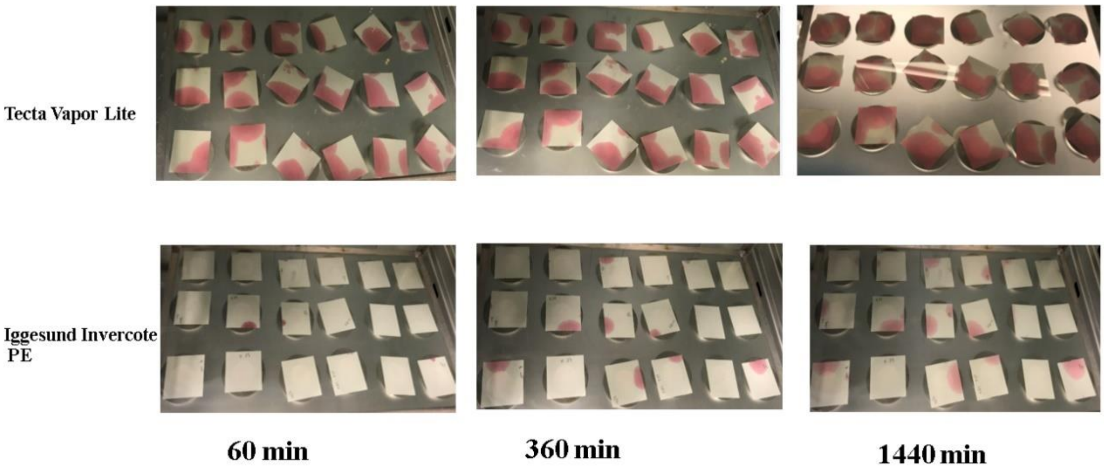


Figure 35. Comparison of Ref. 2 and Ref.3

5 CONCLUSION

The main goal of this study was to investigate the effect of creasing and calendering on barrier properties of dispersion coated paperboards. Starch based coatings are brittle, due to the convertibility defects which was expected. Significant attention was paid to the material creasability. General properties of selected materials were tested accordingly. Air permeability, roughness, tensile strength, creasing, calendering, Cobb60 and OGR tests were the key focus of this study.

This study revealed that it is impossible to see the cracks and propagation of cracks with light microscopy (LM). The LM images were not clear enough to recommend for further studies, since LM magnification scale seemed to be large. SEM scale of 0.5 – 1 μm magnification could provide clearer image for deep analysis. Images from LM were the same since no major significant differences between the samples were observed. Though starch based coating is brittle and cracks were expected with their convertibility, it is hard to say no defects were observed at all. In addition, the cross-sectional images could be a good option as well.

Bio-based dispersion coatings are very often highly hydrophilic, which means that the coatings are sensitive to moisture (Ovaska 2016). Therefore, the coating-water interactions were studied by carrying out Cobb60 tests, which showed moderate differences between the different coatings. However, special attention is required on laboratory sampling, since most of them possess high values of water absorbency compared to commercial sampling. The experimental results of tested samples which are above 40 can be neglected or exempted. Sampling which has less than 40 values can be considered and re-evaluated. These values can be revised by performing Cobb60 test of certain samples again with more suitable conditions.

The effect of calendering on barrier properties of dispersion coated paperboard and commercial material (REF2 and REF3) showed positive results on the OGR test. Calendered samples possess a promising oil penetration resistance compared to uncalendered samplings. According to results of air permeability, Cobb60 and OGR tests, the barrier properties of dispersion coated paperboards were significantly improved by the calendering process. In addition to that, a reduction in roughness was observed. Further studies with suitable conditions can be conducted to obtain results that are more appropriate.

Table 11. Effects of creasing and calendering on the properties of commercial reference samples and experimental samples.

Table 11, the effects of creasing and calendering

Material	Calendering		Creasing and calendering	
Ref 1	Increased glossiness and smoothness	Decreased air permeance and bulk, Cobb Thickness	No significant differences were observed between sampling (after creasing process)	Decreased Cobb OGR was improved
Ref 2	Gloss and smoothness was improved	Decreased air permeance and bulk, Cobb Thickness	No significant differences were observed between sampling (after creasing process)	OGR was improved
Ref 3	Glossiness and smoothness was improved	Decreased air permeance and bulk, Cobb Thickness	Cobb test was as expected	Decreased OGR
Experimental samples	Glossiness and smoothness was improved	Decreased air permeance, bulk, thickness	Increased Cobb test values (poor)	Poor OGR
Summary of all samples	Calendering result was as expected Glossiness and smoothness was achieved. Calendering has positive effect on barrier properties of dispersion coated material.	Effect of calendering on material was determined as thickness and bulk values decreases	Mixed effects observed depending on material. Ref. 3 has revealed better Cobb test and OGR, while the Ref 1 & Ref 2 no significant difference was observed.	Mixed effect on barrier properties depending on material properties, commercial reference material possesses good OGR compared to experimental samples

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LIST OF APPENDICES

Appendix I

Grammage determined for the samples

Sample No.	RER1	REF2	REF3	1	2	3	4	9	10	11	12
Total mass of paper (g)	36.0756	35.7237	34.1526	36.2102	36.007	36.2428	30.041	36.8414	36.8215	36.5601	36.8258
No.of sheet	6	6	6	6	6	6	6	6	6	6	6
Area of trimmed sheet in(m^2)	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02
Grammage g/m^2	301	298	285	302	300	302	250	307	307	305	307

Appendix II

Samples thickness data as obtained, average and STD.VE calculation

Sample no	REF1	REF2	REF3	1	2	3	4	9	10	11	12
1	375	424	322	387	378	380	387	383	373	383	377
2	379	425	329	372	379	377	387	379	368	381	380
3	377	429	330	382	385	377	378	379	371	388	379
4	381	423	319	388	382	374	388	386	367	387	381
5	380	430	335	393	387	375	384	385	365	387	382
6	375	424	322	379	391	377	381	387	369	383	374
Average thickness(μm)	378	426	326	384	384	377	384	383	369	385	379
STD.VE (μm)	2.56	2.93	6.11	7.45	4.97	2.07	3.97	3.49	2.86	2.86	2.93

Appendix III

Basic densities, bulk, densities and bulk data before and after calendering process.

Sampling	Basic Bulk(cm^3/g)	Density (kg/m^3	Density (kg/m^3)	Bulk(cm^3/g)	75 KN/m Density (kg/m^3)	Bulk(cm^3/g)	99 kN/m Density (kg/m^3)	Bulk(cm^3/g)	123 kN/m Density (kg/m^3)	Bulk(cm^3/g)
REF 1	1.256	796	869	1.151	901	1.110	933	1.072	950	1.053
REF 2	1.429	700	739	1.353	770	1.299	780	1.282	791	1.264
REF 3	1.144	874	916	1.092	958	1.044	972	1.029	991	1.009
1	1.276	784	844	1.185	863	1.159	917	1.091	913	1.095
2	1.28	781	833	1.200	852	1.174	867	1.153	914	1.094
3	1.248	801	838	1.193	882	1.134	909	1.100	926	1.080
4	1.536	651	696	1.437	721	1.387	743	1.346	762	1.312
9	1.247	802	862	1.160	883	1.133	921	1.086	940	1.064
10	1.202	832	861	1.161	883	1.133	918	1.089	925	1.081
11	1.072	933	846	1.182	870	1.149	906	1.104	912	1.096
12	1.235	810	860	1.163	882	1.134	927	1.079	937	1.067

Appendix IV

Roughness values as obtained from test.

Sample no	Roughness Basic Mean value in (ml/min)	Mean value in (ml/min) 27 kN/m	75 kN/m	99 kN/m	123 kN/m
REF 1	226.2	102.9	111.3	94.7	91.5
REF 2	152.5	139.2	109.2	104.9	127.6
REF 3	462	291	274.5	296	263.7
1	297.5	213.7	193.1	157.8	187.6
2	401	310	177.7	242.3	159.2
3	268.9	270	191.6	192.1	215.1
4	374	219	207.9	193.4	205.6
9	228.1	170.3	130.6	146.2	164.3
10	223.4	158.3	121.2	99.8	139.7
11	294	198.1	146.4	153.2	133.9
12	268.9	188.8	143	126.3	143.9

Appendix V

Air permeability values for Uncalendered and different calendered point.

Air permeability mean value (µm/Pa s)					
Sample No.	Basic sample (Uncalendered)	Calendered 27 kN/m	Calendered 75 kN/m	Calendered 99 kN/m	Calendered 123 kN/m
REF1	5.79	0.27	0.26	2.34	2.05
REF2	3.43	0.24	0.10	0.46	0.14
REF3	1.55	0.34	0.31	0.38	0.24
1	0.69	0.29	0.17	0.41	0.18
2	1.92	0.30	0.43	0.63	0.35
3	0.76	0.85	0.54	0.70	0.56
4	3.88	0.82	0.71	0.79	0.63
9	1.80	0.50	0.31	0.41	0.34
10	3.89	0.34	0.33	0.60	0.30
11	2.33	0.65	0.35	0.91	0.30
12	1.93	0.41	0.25	0.89	0.33

Appendix VI

Calendered Cobb 60 test value

Sampling	Final weight (g)	Initial weight (g)	Absorbency value	Average Absorbency value
REF1	6.291	5.986	30.5	
	6.25	5.971	27.9	29.2
REF2	5.216	5.208	0.8	
	5.163	5.154	0.9	0.85
REF3	5.712	5.679	3.3	
	5.703	5.679	2.4	2.85
1	5.657	5.326	33.1	
	5.633	5.342	29.1	31.1
2	5.667	5.279	38.8	
	5.765	5.268	49.7	44.25
3	5.585	5.206	37.9	
	5.575	5.227	34.8	36.35
4	5.621	5.237	38.4	
	5.588	5.239	34.9	36.65
9	5.65	5.358	29.2	
	5.705	5.381	32.4	30.8
10	5.667	5.346	32.1	
	5.644	5.335	30.9	31.5
11	5.606	5.286	32	
	5.597	5.284	31.3	31.65
12	5.663	5.338	32.5	
	5.695	5.337	35.8	34.15

Appendix VII

Uncalendered Cobb 60 values

Sampling	Final weight (g)	Initial weight (g)	Absorbency value	Average Absorbency value
REF 1	5.423	5.121	30.2	
	5.495	5.123	37.2	33.7
REF 2	5.223	5.214	0.9	
	5.188	5.178	1	0.95
REF 3	4.938	4.932	0.6	
	5.009	4.952	5.7	3.15
1	6.492	6.155	33.7	
	6.456	6.146	31	32.35
2	7.056	6.607	44.9	
	7.034	6.581	45.3	45.1
3	6.437	6.028	40.9	
	6.445	6.026	41.9	41.4
4	6.427	5.989	43.8	
	6.436	6.009	42.7	43.25
9	7.062	6.7	36.2	
	7.108	6.728	38	37.1
10	6.991	6.711	28	
	7.08	6.706	37.4	32.7
11	7.03	6.664	36.6	
	7.034	6.688	34.6	35.6
12	6.442	6.011	43.1	
	6.418	6.11	30.8	36.95

Appendix VIII

Line load, Nip load and paper width

DT Laboratory Calender | Line Load | DT Paper Scier

Line load

Pressure bar	Nip Load kN/m	Paper Width m
10	18,7488	0,3
20	52,2976	0,3
25	69,072	0,3
30	85,8464	0,3
35	102,6208	0,3
40	119,3952	0,3
45	136,1696	0,3
50	152,944	0,3
55	169,7184	0,3
60	186,4928	0,3
65	203,2672	0,3
70	220,0416	0,3
75	236,816	0,3
80	253,5904	0,3
85	270,3648	0,3
90	287,1392	0,3
100	320,688	0,3
120	387,7856	0,3
140	454,8832	0,3
160	521,9808	0,3

A= Pressure Display
B = Nip Load
C = Paper Width 0,3 m