TAMPEREEN TEKNILLINEN YLIOPISTO
TAMPERE UNIVERSITY OF TECHNOLOGY

SUVI HARJU

LIQUID PENETRATION IN FOOD SERVICE BOARDS

Master of Science Thesis

Examiner: Professor Jurkka Kuusipalo and D.Sc. Johanna Lahti
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ABSTRACT

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Edge wicking, or raw edge penetration, is a phenomenon which interests the manufacturer of the food service boards. Raw edge penetration occurs for example in the side seams inside the paperboard cups. The inside of the cup is coated with the barrier coating, but the raw edge of the side seam is in touch with liquid. Liquid is able to penetrate into the structure of paperboard through the raw edge. The objective of this thesis is to research the edge wicking phenomenon during the food service board making process. The other aim is to find proper test methods to measure edge wicking.

This master's thesis consists of literature review and experimental part. The literature part focus on the phenomenon and the factors which effect on edge wicking. In the experimental part the structure of the paperboard, the edge wicking and the sizing state were studied. The samples used in this work were commercial Metsä Board paperboard grades. Sampling was executed in five different stages of paperboard manufacturing and converting process in order to study the development of paperboard through the process. The same measurements were also made five weeks after manufacturing to see the effect of time. The liquid penetration is researched a lot, but not the edge wicking. The raw edge penetration was measured with coffee and water, which are the most common liquids served from paperboard cups.

According to the literature review the fibers used in paperboard manufacturing, sizing agents and the liquids served in ready-made paperboard cups have an effect on raw edge penetration. Based on the results of the experimental part, changes in the measured paperboard properties were observed from the samples of different process stages. However, the biggest difference was detected between the first stage, board machine, and the last stage, time tracking samples.

Developing of internal sizing was discovered to have an effect on Cobb and cold edge wicking test results. The results improved as the process steps progressed, and the best results were achieved after five weeks. The results of edge wicking tests can be determined both by visually measuring the penetration length or by calculating the changes in masses. The calculation is more useful, because it is objective method and the standard deviations of the results were smaller. Hot edge wicking was measured with two different tests, whose biggest difference was the cutting direction of the samples. As an outcome of the study and as a proposal for the future, the samples should be cut so that the longer edge of the sample is in the same direction than the raw edge of paperboard cup.

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Reunaimeytyminen, tai tarkemmin raakareunaimeytyminen, on ilmiö joka kiinnostaa ruokatarjoilukartonkien valmistajia. Raakareunaimeytymistä tapahtuu esimerkiksi kartonkikuppien sisäsauman reunassa. Kupin sisäpuoli on suojattu barrier-kerroksella, mutta sauman raakareuna altistuu nesteelle, joka tunkeutuu kartonkiin. Tämän työn tarkoitus on selvittää mitä reunaimeytymisen tuloksille tapahtuu ruokakartonkien valmistus- ja jalostusprosessien aikana. Toinen tarkoitus on löytää sopivat mittaustavat reunaimeytymisen mittaamiseen.

Tähän diplomityöhön kuuluu sekä kirjallinen että kokeellinen osuus. Kirjallinen osuus selvittää ilmiötä ja siihen mahdollisesti vaikuttavia tekijöitä. Kokeellisessa osuudessa testattiin kartongin perusominaisuuksien lisäksi reunaimeytymistä ja liiman kypsymistä. Näytteet ovat kaupallisessa tuotannossa olevia Metsä Boardin kartonkilaatuja, joista otettiin näytteitä koko jalostusprosessin ajalta, viidestä mittapisteestä. Lisäksi samat mittaukset suoritettiin noin viisi viikkoa kartonginvalmistuksen jälkeen. Nesteen imeytymistä kartonkiin on tutkittu paljon, mutta raakareunaimeytyminen on vähemmän tutkittu asia. Reunaimeytymistä tutkittiin sekä vedellä että kahvilla, jotka ovat yleisimmät kartonkikupeista tarjoillut nesteet.

Kirjallisuudesta löytyneiden lähteiden mukaan raakareunaimeytymiseen vaikuttavat muun muassa kartongin valmistukseen käytetyt kuidut ja massaliimat sekä valmiista kupeista tarjottavat nesteet. Kokeellisen osuuden tulosten perusteella kartongin ominaisuudet muuttuvat jonkin verran prosessin aikana, mutta suurin ero oli ensimmäisten, kartonkikoneelta otettujen ja viisi viikkoa kartonginvalmistuksen jälkeen mitattujen näytteiden välillä.

Kokeiden perusteella massaliiman kypsymisellä on vaikutusta Cobb-testin tuloksiin ja kylmän nesteen reunaimeytymiseen. Tulokset paranevat prosessin myötä, ja parhaimmillaan ne olivat viiden viikon kuluttua mitatuissa näytteissä. Reunaimeytymiskokeista saadaan tuloksia sekä visuaalisesti mittaamalla imeytymän pituutta että laskennallisesti massanmuutoksen avulla. Laskennallinen menetelmä on luotettavampi, sillä se on objektiivinen ja tulosten keskihajonta on pienempi. Kuuman nesteen reunaimeytymistä mitattiin kahdella eri testillä, joiden suurin ero oli näytteen leikkaussuunta. Näytteet tulisi leikata siten, että pidempi tutkittava sivu on leikattu samaan suuntaan kuin kartonkikuppien raakareuna.

PREFACE

This thesis was written between July and November 2018 mainly in Tampere and Äänekoski. First I want to thank my bachelor's thesis supervisor, because of her I was interested in forest industry. I want to thank Metsä Board for the interesting subject which of I didn't know much before starting, but I learned a lot during the process. Thank you Terhi for being so flexible with the place to sit during the writing process. I also want to thank my colleagues Martina and Tomas, and all the production workers who were very helpful with the sample picking process. Karoliina, my supervisor gave good advice during the whole process and helped me with everything I needed.

My days in Äänekoski were nice because of the atmosphere in TC, but I liked even more to be at home in Tampere with my dear Henri, who supported me during the whole process even with the stupid little things. Thank you my family, because of you I even considered going to TUT, and thank you all the people I met during my four years in TUT, because of you I stayed.

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Suvi Harju

CONTENTS

ABBREVIATIONS AND NOTATION

AKD	alkyl ketene dimer
ASA	alkenyl succinic anhydride
BCTMP	bleached chemi-thermomechanical pulp
CTMP	chemi-thermomechanical pulp
EWI	edge wicking index
EWT	edge wicking test
LDPE	low density polyethylene
PE	polyethylene
REP	raw edge penetration
RH	relative humidity

1. INTRODUCTION

A food service board is paperboard product, which is used mainly to serve food or liquid. Food service boards are used as food-on-the-go boards and they are usually somehow coated to protect the paperboard from wetting. Liquid penetration is a general term for absorption of the liquid by the paperboard in all its dimensions. The edge wicking is the phenomenon, where only paperboard's raw, unprotected edge is exposed to wetting. The paperboard plate has no raw edges in contact with liquids, but the cup has a side seam, which is vulnerable to wetting despite the coating. (Kastinen 2010; Kline 1982, pp. 196-211; Seppälä 2000, pp. 22-26)

The principal objective of this thesis is to research what happens to the edge wicking phenomenon during the food service board making process. Also the aim is to find proper test methods and tolerances for edge wicking. There are many tests for liquid penetration in literature, but none of them are standard. Edge wicking tests (EWT) interest both food packaging and serving industries, although they do have different requirements for their products. Many properties of the paperboard have an influence on edge wicking. The structure of the paperboard, which includes density, thickness, porosity and moisture content of the paperboard, as well as sizing variables. Also the type of the liquid served in the paperboard product has an effect, because different liquids react in different ways. (Kastinen 2010; Mark et al. 2012; Myllys 2007; Salminen 1988, pp. 41-58; Tufvesson 2006)

This thesis is divided into two parts. The literature part presents the manufacturing and converting processes of the food service board, the edge wicking theory and how different factors affect the phenomenon as well as different test methods found in literature. The experimental part presents tests used in this thesis and their results.

The aim of the experimental part is to find out how much edge wicking variables are changing in process line of paperboard mill during the whole process from the paperboard machine to pigment coater, winder, extruder and another winder. The second aim of the experimental part is to analyze the current test methods and find new possible ways and tolerances to measure the edge wicking.

2. PAPERBOARD PACKAGING

Paper and paperboard are manufactured worldwide, and they are used in many applications. In addition to virgin paper and paperboard products, there are also a lot of applications using recovered fibers as raw material, because they are easy to recycle. Although, it is not allowed to use recovered fibers in all applications, for example often when the package is in direct contact with food. In 2010 51 % of all paper and paperboard production was used in packaging. (Kirwan 2012, pp. 1-19)

Paperboard packaging manufacturing process begins with pulp making and paperboard manufacturing. The paperboard is then converted to suit the intended use. The most common converting processes in the paperboard mill are pigment coating, winding and sheeting. The form of the package is usually made in other mills. (Smook 1992, pp. 343-354)

2.1 Paperboard manufacturing

Typical paperboard has three layers, where the top and bottom layers are chemical pulp and the middle layer usually light-weight and cheaper mechanical pulp (Häggblom-Ahnger & Komulainen 2001, pp. 55-77). Also two layer paperboard is possible, but usually paperboard has three layers. The layers can be seen in Figure 1.

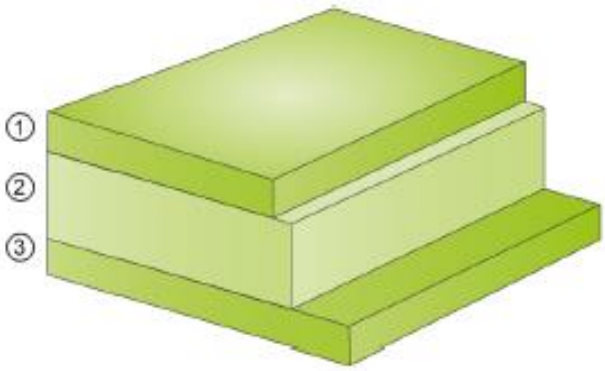

Figure 1. *Structure of typical paperboard (Metsä Group internal, 2018)*

Chemical pulp in the top (1) and bottom (3) layers in Figure 1 makes the surface high quality. Chemi-thermomechanical pulp is a good material for middle layer (2) because it makes paperboard stiff. Also mechanical pulp can be used in the middle layer to add bulk to paperboard. (Häggblom-Ahnger & Komulainen 2001, pp. 31-36; Nemez 2013)

The paperboard manufacturing process begins with pulp making. The main pulp types are chemical, mechanical and chemi-thermomechanical pulp (CTMP). All of them can be bleached or unbleached. The manufacturing process of chemical and mechanical pulp is

different. In the both methods lignin is softened but in chemical pulp it is made with chemicals and in mechanical pulp by mechanical energy. (Smook 1992, pp. 36-44)

Chemical pulp making is based on chemical cooking, made from kraft or sulfite. The method is based on chemical defibering, where the lignin is dissolved with the help of chemicals and heat. The lignin bonds the fibers together and it is removed together with hemicellulose and other components. Bleaching removes the lignin which cooking could not remove from the pulp. The chemical pulp contains only 2 – 5 % lignin (Hintz & Lawal 2018). The chemical pulp is clean, but yield percent is smaller than with mechanical pulp, approximately 40 – 55 %. (Häggblom-Ahnger & Komulainen 2001, pp. 31-35; Niskanen 2008, pp. 55-87; Smook 1992, pp. 65-83)

The manufacturing methods of mechanical pulp are grinding and refining. The lignin is softened with the help of water, heat and mechanical stress. The mechanical method does not dissolve anything and it contains different sizes of fibers. The yield percentage of mechanical pulp is 96 – 98 %. The hemicellulose content of mechanical pulp is high, and the pulp also contains 30 % lignin. (Häggblom-Ahnger & Komulainen 2001, pp. 31-35, KnowPap 2005)

CTMP is made by using thermal mechanical pulping and light chemical treatment. Fibers of CTMP are stiff and bulky with some fine particles. The CTMP made from softwood has yield percentage of 94 – 95 %. The yield percentage of hardwood CTMP is 88 – 92 %. Both CTMP and mechanical pulp have excellent mechanical properties and opacity, as well as high bulk. (Häggblom-Ahnger & Komulainen 2001, pp. 31-35; KnowPap 2005; Metsä Group internal, 2018)

The pulp arrives to the paperboard mill as dry pulp (bales) or as wet pulp straight from the pulp mill. The process in the paperboard mill begins from the pulp making, which includes mixing the various papermaking chemicals together. Paperboard is sized by means of hydrogen bonds between the fibers. Hydrogen bonds form during the process when moisture content is approximately 20 – 30 %. In addition to fibers, pulp also contains non-fibrous additives like pigments, fillers, moisture and sizing agents. They are added to improve either the appearance or performance of the product, or the productivity of the process. Internal sizing additives, mineral pigments, fillers and strength additives are examples of additives added to pulp. (Kirwan 2012, pp. 1-19; Libby 1962, pp. 1-39)

The first section, web forming, consists of headboxes and manifold which spread the pulp suspension evenly in the transverse direction of the paperboard machine and produce an appropriate level of turbulence in a pulp suspension to haul fiber flock. Finally it produces a pulp suspension shower with a desired consistency as well as speed and direction for the wire section. (BeMiller & Whistler 2009, pp. 660-662; Häggblom-Ahnger & Komulainen 2001, p. 131-146) In additon to headboxes, the wet end consists of wire

section and press section. Every layer has its own headbox which can be seen in Figure 2.

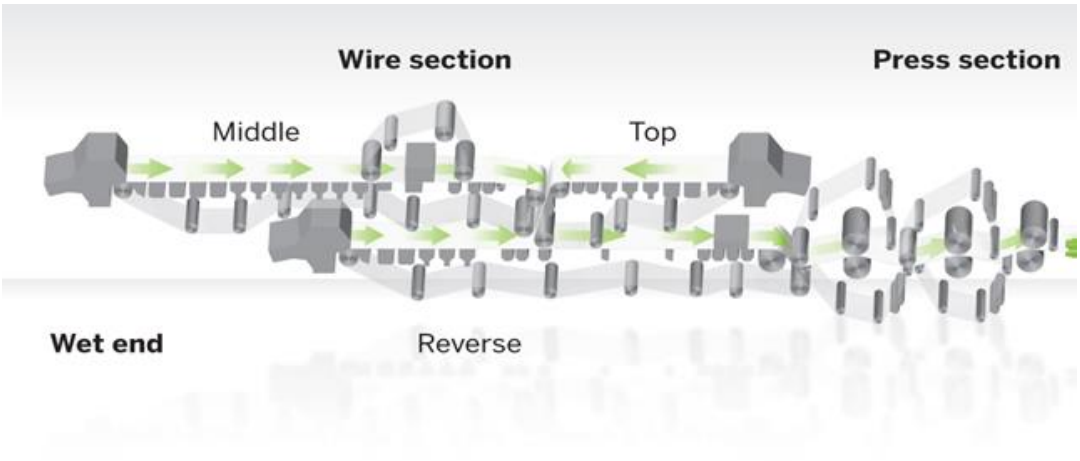

Figure 2. *Wet end (Metsä Group internal, 2018)*

The next section of the wet end (Figure 2) after the headboxes is the wire section, which removes water from the pulp suspension with the help of wires. It also balances the percentages of fibers, fillers and moisture. After wire section the pulp suspension contains dry matter that much (15 – 20 %), that it is easy to move to the next part of the paperboard machine. (Häggblom-Ahnger & Komulainen 2001, pp. 155-162; Kline 1982, pp. 92-135) The wire section consists of as many wires as there are headboxes and layers in the paperboard.

The press section removes water from the web and compresses it. The aim of the press section is to remove as much water as possible and achieve sufficiently high wet strength. The second aim of the press section is to bond the layers together. The layers are pressed between two felts and when the consistencies of the layers are about 10 – 12 %, they will bond together. (Kline 1982, pp. 92-135) Wet compression is made with the help of machine felt, nips and smooth rolls. The compression is made carefully and in stages. The press section contains several nips and the last nip has the most compression. Papers are compressed even more than paperboards, because paperboards need more porous, bulky middle layers. Too much compression makes the paperboard too thin. The press section makes the surface smooth and the paperboard porous and bulky. (Häggblom-Ahnger & Komulainen 2001, pp. 155-162) After wet end there is the so-called dry end which can be seen in Figure 3.

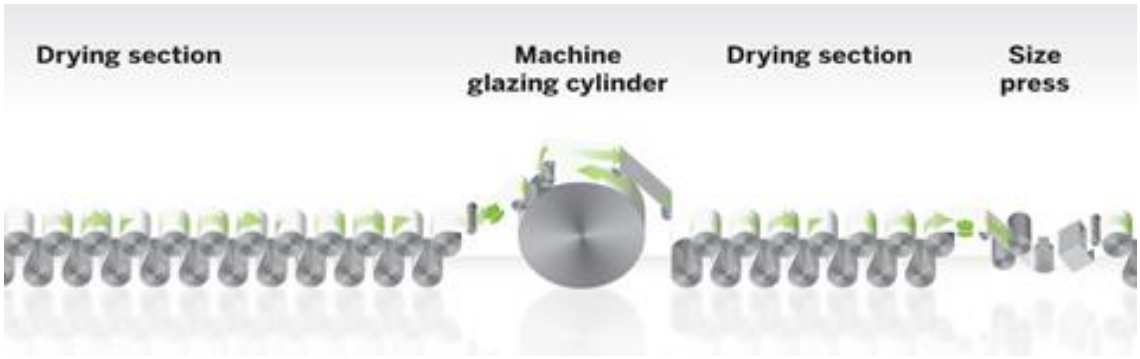

Figure 3. *Dry end (Metsä Group internal, 2018)*

The last part of actual paperboard manufacturing is drying. If the paperboard is pigment coated, the paperboard is dried also after that. More about pigment coating is told in chapter 2.2.1. In the dry end (Figure 3) the water is removed by evaporation. The steam pressure is controlled in the dry end, and it depends on the use of the final product. If the paperboard is coated, it has a different moisture profile than the uncoated product. The methods for drying are conduction, radiation and convection. Mainly conduction is used, but also convection and radiation may be involved. All the methods evaporate the water and air removes water vapor. (Häggblom-Ahnger & Komulainen 2001, pp. 163-172; Kline 1982, pp. 124-130)

2.2 Converting processes

All the paperboards are converted somehow before using. Pigment coating, winding, extrusion coating and sheeting are usual converting processes performed in paperboard mills. (Smook 1992, pp. 343-354)

The purpose of the package is to carry and protect the product. Paperboard itself is very useful package material, but it needs some converting before being used as package. When the paperboard is transported to the converter, it may be printed, cut, folded and glued into its final form in the case of paperboard packaging. (Kline 1982, pp. 196-211; Seppälä 2000, pp. 22-26)

2.2.1 Pigment coating

Pigment coating is made for paperboards to improve printing properties. Paper has a smoother surface, but paperboard needs some converting so that the ink can be applied to the surface of the porous paperboard. The particles of pigment coating are smaller than fibers, thus the coating process makes the surface smoother than the uncoated surface. Coating makes the surface smooth and easy to print, but also bright and white. Coating affects printability by improving smoothness, gloss, surface strength, absorption and retention. (Kline 1982, pp. 141-160)

Paperboard can be coated with pigment coating either one or two sided. The pigment coating can be made several times one after the other, but after every time the coating must be dried. If the paperboard is coated twice from the same side, it is called 2-layer coated. Pigment coating machine can be either online or offline coating machine, which of first means that the machine is at the same line with paperboard machine, directly after that. When the coater is online machine like in Figure 4, it is part of the dry end. The advantage of online coating machine is that the process is continuous and compared to the offline coating machine, storage and one reeling process can be omitted. The disadvantage is that if some difficulties arise in the coating machine, the whole process must be shut down. Offline coating machine is a separate machine and there can be some time between paperboard manufacturing and pigment coating. The advantage of offline coating machine is that the machine is designed precisely for coating purpose, and speed and efficiency are better. The disadvantage is the necessity of extra reeling. (Häggblom-Ahnger & Komulainen 2001, pp. 184-203; Kline 1982, pp. 141-160; Know-Pap 2017; Libby 1962, pp. 273-319; PrintWiki 2015; Smook 1992, pp. 283-296)

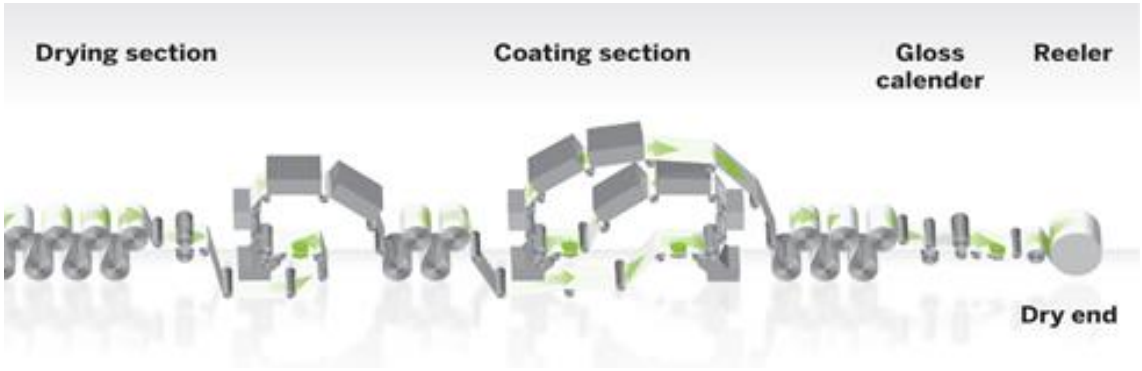

Figure 4. *Online coating machine (Metsä Group internal, 2018)*

The online coating machine follows the drying section which can be seen in Figure 4. In the pigment coating process the applicator cylinder moves lots of color from the color pan to the web and there, depending on the method, some application, for example knife or rod, takes the excess color away. This application makes the pigment surface even and smooth. After coater the web is calendered and reeled to the roll. (Kline 1982, pp. 141-160; Kocurek & Kouris 1990, pp. 76-108; Seppälä 2000, pp. 29-54)

The coating color consists of pigments, binders and additives in water solution. The dry matter content is 75 – 95 % and usually kaolin, talc and calcium carbonate are used as pigments. Binders binds pigment particles to each other and to the web. Starch and proteins are natural binders and artificial ones are latexes and CMC. Additives are 1 % of the coating color and they are used to control pH and viscosity, dyeing, prevent foaming, preserving, and as lubricants. (Häggblom-Ahnger & Komulainen 2001, pp. 184-203)

2.2.2 Winding

Winding is done after pigment coating and its purpose is to produce suitable sized rolls for converting or for customers. Winding is actually cutting process where the full width web is cut into usable widths, but it also includes unwinding and rewinding of the roll. (Holik 2006, pp. 383-400; Kocurek & Kouris 1990, pp. 137-195) The paperboard machine can be up to 9 meters wide, and converting machines cannot take rolls that big (BeMiller & Whistler 2009, pp. 660-662). The size of the roll made by winding is dependent on the following converting machines, like extruder (Smook 1992, pp. 264-282).

The winder consists of a rack, a big number of cylinders, and an air blowing opening. The cylinders are for paper conveying, front supporting and rear supporting. Usually the winding is done with sharp knife, but also a laser and a high pressure water jet may be used. The winder cuts the rolls into a suitable size and also trims off 2-20 centimeters from the both edges of the roll. Those edges are conveyed back to the dry-end pulper. Usually the edges of paperboard are slightly ragged, thinner and dryer than the middle part of the web and for that reason they are cut out. (Jindong & Yuanguang 2017; Metlas 1981, pp. 5-34; Smook 1992, pp. 264-282)

The tension of the roll causes the tension of the surface layer. Winding also affects the density, because when the roll is pressed, the air is removed. The inner layers are pressed more than the outer layers, and the compression stress is bigger inside the roll. At the same time the tensile strength decreases. The aim is not to compress the paperboard web, but in practice it happens when web goes through winder. (Häggblom-Ahnger & Komulainen 2001, pp. 230-239; Kocurek & Kouris 1990, pp. 137-195; Metlas 1981, pp. 5-34)

Some parameters have an effect on the compression. Core diameter, inserts, material and wall weight and narrower roll have especially an influence on core pressure. The knife can cause problems both in process and in final product, if it is improper for its use. The dust which can be in printed webs is the biggest cause of the problems. (Kocurek & Kouris 1990, pp. 137-195) There are guidelines for web tension of winding device. The higher the web tension is, the tighter the roll will be, but if it is too tight, the web may yield or break. If the web tension is too low, the web may loose traction on rollers or flop around. The most common web tension in the machines is 10 – 25 % of the breaking strength of the web material. (Roisum 1994, pp. 1-16)

The rewinding has an influence on the web properties. It improves the smoothness of rough paperboards and has the opposite influence on smooth paperboards. The thickness decreases and density increases. (Metlas 1981) Winding has same effects on paperboard as calendering, because in both methods the web goes through calenders. Calendering can decrease the surface roughness, the pore radius and total pore volume of the paperboard. (Salminen 1988, pp. 59-82)

2.2.3 Extrusion coating

Extrusion coating is made by extruding a polymer film onto a quickly moving paperboard web. It is possible to make single- or multilayer coatings by extrusion. (Kuusipalo 2008, pp. 108-113) Extrusion coating is made to improve the paperboard's barrier properties for example with liquids, because the plain paperboard cannot withstand water as good as extrusion coated paperboard. The low density polyethylene (LDPE) coating makes also fairly good water vapor barrier and it can be used to heat-seal the layers together when forming the end product, for example paperboard cup. (Alsdorf 2009; Kirwan 2012, pp. 353-382) The seal initiation temperature of the LDPE is low and heat sealing range wide (Lahti & Tuominen 2007). Barrier coating can be achieved also with dispersion coating (Kimpimäki & Savolainen 1997, pp. 208-228).

The extrusion coating process requires pretreatment so that the polymer film adheres to paperboard. The most common pretreatments are corona and flame. Both treatments evaporate harmful impurities and water from the surface, burn dust and bristly fibers and increase surface energy as well as adhesion. The pretreatment also improves the printability properties. (Lahti & Tuominen 2007)

The flame treatment burns the paperboard web so gently, that the web does not light up. To provide the effective flame treatment, the gas flame must be sufficiently oxidized, because it oxidizes a thin layer of substrate surface. In the process different thermally activated atoms are formed. These react with the surface substrate and compose adhesion by creating functional groups. (Lahti & Tuominen 2007)

The corona treatment is made by applying the high voltage across an electrode and grounded metal roll to induce ionization of air. The surface energy increases when the polar groups are introduced on the surface. In addition to adhesion properties, the corona treatment also improves wetting properties of the surface. The corona decreases the seal initiation temperature of LDPE. (Lahti & Tuominen 2007)

Extruder consists of hopper, screw, adapter section and die. The machine also has a motor, transmission, cooling zone, pressure control valve, breaker plate and un- and rewinders. Hopper is feeding system, where plastic is dosed in the pellet form. Hopper in single screw extruder is important and too much or too little pellets can cause problems to the whole process. Hopper can be filled both manually and automatically. Manual loading can cause troubles, and automatic filling is more reliable solution. (Kuusipalo 2008, pp. 108-113)

The screw is rotating with the help of motor and the screw has typically three sections. Those sections are feed, compression and metering zones. The main purpose of the screw is to melt the polymer pellets into a homogenous melt. The water cools the screw in the

feed zone to avoid bridging due to premature melting of pellets. (Lohijoki & Järvelä 2000, pp. 3-6)

The melting happens mainly by friction in screw and with the help of heaters. The melt shears against the cylinder and thin melt film is created. When the mass goes to the next section, metering zone, it should be completely melt. The metering zone makes the polymer melt to its final form so that it is suitable to die, by both composition and rheology. The melt which goes to die has to be uniform by temperature profile and structure so that the polymer film is defect free and works as it should. Internal temperature of the extruder is 250 – 300 °C during the PE-coating process. (Kuusipalo 1997, pp. 4-5; Lohijoki & Järvelä 2000, pp. 3-6) When the polymer melt is drawn onto the paperboard web by the laminator nip, the web is dried and rolled back to the roll. The adhesion and cooling takes place in the laminator nip. Those can be seen in Figure 5. (Kuusipalo 1997, pp. 4-5; Kuusipalo 2008, pp. 108-113)

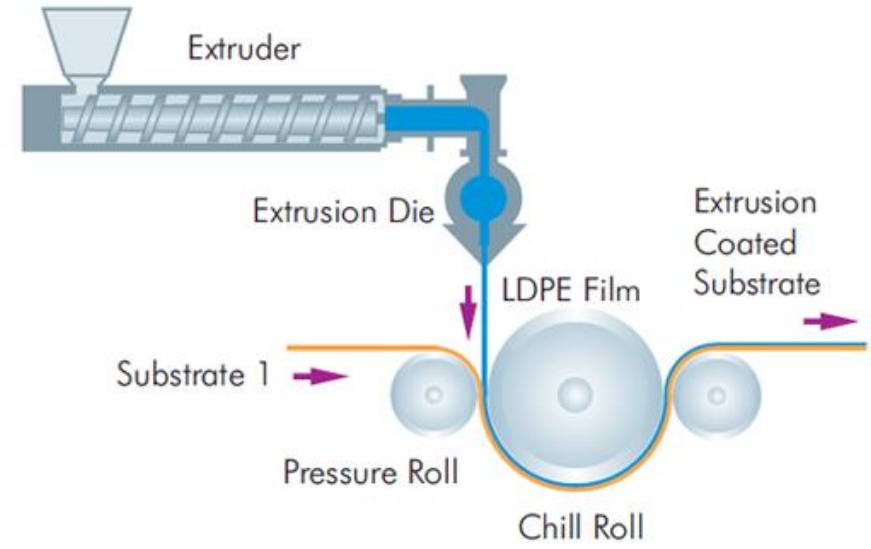

Figure 5. *Extrusion coating line (Safepack Solutions 2018)*

The extrusion die has the form which is also desired from polymer, in this case film. The melt is forced through the die (Figure 5) which makes the desired thickness and width of the film and maintains suitable melt temperature. The die is usually either T-die or coat hanger die, so that the melt comes from the middle of the film and spreads over the entire width. (Kuusipalo 1997, pp. 4-5; Kuusipalo 2008, pp. 108-113)

The paperboard cups for hot drinks are usually coated only inside, and cups for cold drinks on both sides. The cold drink “sweats” from the outside and the paperboard needs protection against the condensed water which moisten the cup, and for that reason it is usually two-side PE-coated. (Rhim & Kim 2009; Seppälä 2000, pp. 27-78) Also varnish can be used to protect the outer layer from wetting (Chen et al. 2013). In addition to

protecting the fibrous structure of paperboard from liquids, extrusion coating causes compression to the paperboard web. The compression increases paperboard density and decreases paperboard thickness, but the total thickness increases, when the polymer layer is added. (Häggblom-Ahnger & Komulainen 2001, pp. 230-239)

2.2.4 Printing, die-cutting and cup making

One example of the final product is paperboard cup, and it is usually made in the separate factory. When making printed cups, paperboard sheets or rolls are printed before die cutting and cup making. Also non-printed cups can be made. (Seppälä 2000, pp. 150-208)

There are different types of printing machines. The main processes used today are offset, flexography and gravure. Also letterpress, silk screen and digital printing are sometimes used. (Kirwan 2012, pp. 265-312) Digital printing is increasing technique in the package printing. The digital printing methods are so called non-impact methods, and the main ones are electrophotography, ink-jet and thermal printing. (Kuusipalo 2008, pp. 243-283) In all kind of printing machines the paperboard sheets run through cylinders, which press paperboard together. The paperboard has to be smooth so it can be printed. Because no paperboard is completely smooth, there has to be compression in the printing process. Very smooth uncalendered paperboard will print better than heavily supercalendered sheet, which means that calendering removes some of the compressibility properties of the paperboard. (Libby 1962, pp. 320-351)

Electrophotography is suitable method for especially extrusion coated paperboards. The paperboard cups which are PE-coated on both sides have the printed image on the top of PE-layer. The method is non-impact method, which means that the image information of this multi-step process is in the form of electronic signals. According to Lahti 2005, the signals are then converted to optical signals, transformed to a two-dimensional latent electrostatic field pattern stored on a photoconductive dielectric material. The charged toner particles are deposited on the field pattern, which makes the latent image visible. It is then made permanent by fixing. The ink can be either liquid solution or dry, made from powder-like materials. In the case of folding box board, the electrical conductivity should be as low as possible to achieve good print quality and runnability. (Lahti 2005)

Migration of printing inks is also a topic which have to be taken into account, because the ink is in the outer layer of paperboard cup, and it is in touch with lips when drinking from the cup. Migration can be measured and there are limits which it cannot cross. The limits may be country specific. All materials in paperboard cups and other food service boards have to be safe and suitable for food contact. (*Kontaktimateriaaleja koskeva lainsäädäntö* 2018; Myllys 2007)

The paperboard web strength is important parameter when printing. It causes runnability problems if web strength is not at the required level. The process also requires tension

control from unwind stand and between printing couples. To keep the printing in control, the longer press leads require higher web tension. (Kocurek & Kouris 1990, pp. 126-136; Smook 1992, pp. 343-354)

The surface tension of ink should be lower than the surface energy of the paperboard to achieve the adhesion. On the other hand, the nip pressure causes the penetration of ink into the porous structure of paperboard, which is not desirable. (Oittinen & Saarelma 2009, pp. 88-146) The surface tension of inks can be controlled by additives either in ink or in paperboard (Kuusipalo 2008, pp. 243-283). Internal sizing is used to prevent penetration and blocking. More about sizing is told later in chapter 4.2.

Water-based inks contain water, pigments, binders, solvents and additives. The additives can be for example waxes or surfactants. In addition to surfactants, also alcohol can be added to decrease the surface tension of ink. When printing PE-coated paperboard, the surface modifications such as corona, flame, plasma and priming are used to improve adhesion properties. Because of smoothness of the PE-surface, adhesion requires other forces than mechanical. Corona treatment improves the surface energy, but it diminishes within time, so the printing should be done immediately after the treatment. (Kuusipalo 2008, pp. 243-283)

Die cutting is the cutting method which is used to cut the paperboard to wanted shape. The blade is sharp and shaped knife-edge, which is pressed through one or several layers of sheets. (Gooch 2007, pp. 283) With the sharp-edged blade, the edges of paperboard stay unsqueezed and the edges are clean and free from the fibrous debris. Those can be loose fibers, fragments of fibers, clumps of fibers or thin slivers of paperboard. There are two common cutting methods, rotary and flatbed. The rotary is usually in-line process right after printing and flatbed can be either sheet-fed or in-line after printing. (Kirwan 2012, pp. 265-312) In Figure 6 are presented the phases of the cup making process.

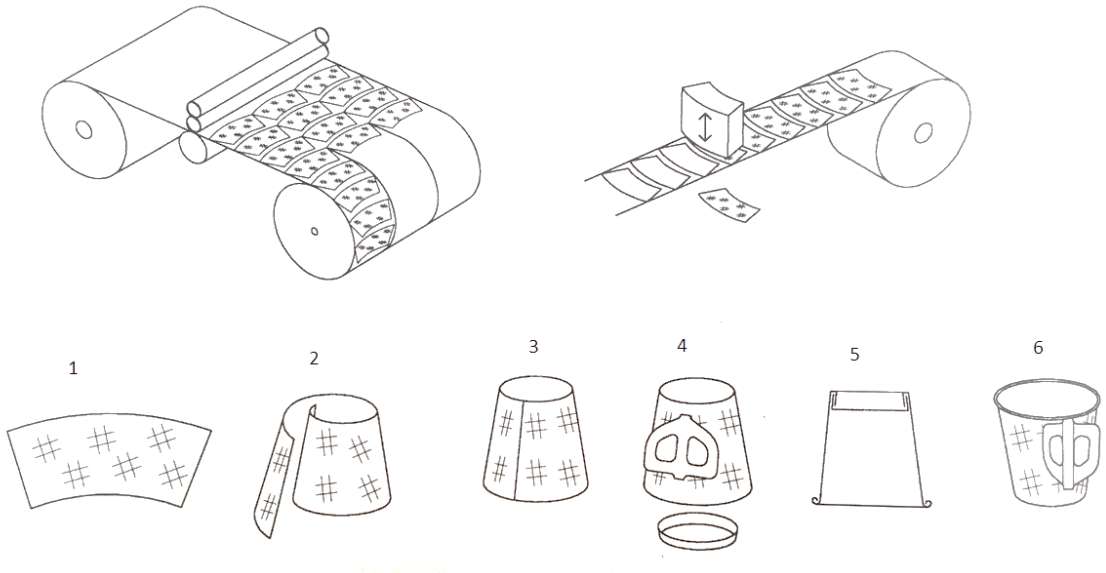

Figure 6. *Paperboard cup making. Original figure from Seppälä 2000, pp. 178*

From the preform of a paperboard cup (1) in Figure 6, the cup is formed with heat sealing (2-3). The finseal to the side seam is made with heat sealing and PE-layer allows it, but the edge of paperboard in the side seam stays unprotected and it touches the liquid. The seams with walls and bottom are pressed together in a way that the raw edges are inside the seams (5). The seams are heat sealed. The upper edges are turned outside of the cup and inside of themselves (5). (Seppälä 2000, pp. 150-208)

3. EDGE WICKING IN PAPERBOARD

Edge wicking, raw edge penetration, in-plane wetting and liquid penetration have almost the same meaning. There are four mechanisms for water transport in paper; diffusion of vapor in the pores, capillary transport of liquid in the pores, surface diffusion in the pores and water transport through the fibers. The mechanisms happen often together with one of them dominating. Temperature, vapor pressure and chemical composition of the liquid are affecting the magnitude of each transport mechanism. (Kastinen 2010; Kissa 1996; Mark et al. 2012; Nemez 2013; Salminen 1988, pp. 17-22) The resistivity of edge wicking is one of the most important properties for liquid service boards (Mark et al. 2012).

The wicking phenomenon covers topics from the contact angle and line, liquid/solid adhesion to the wetting transition and spreading dynamics (Cotorobai et al. 2016). Edge wicking specially means the liquid penetration in paperboard edges i.e. in the area of thickness. Those so called raw edges are for example inside of the paperboard cup next to side seam. Paperboard cups are extrusion coated but the cutting edge is uncoated raw edge. (Kastinen 2010) The raw edge is in contact with the liquid inside the cup, which can be seen in Figure 7.

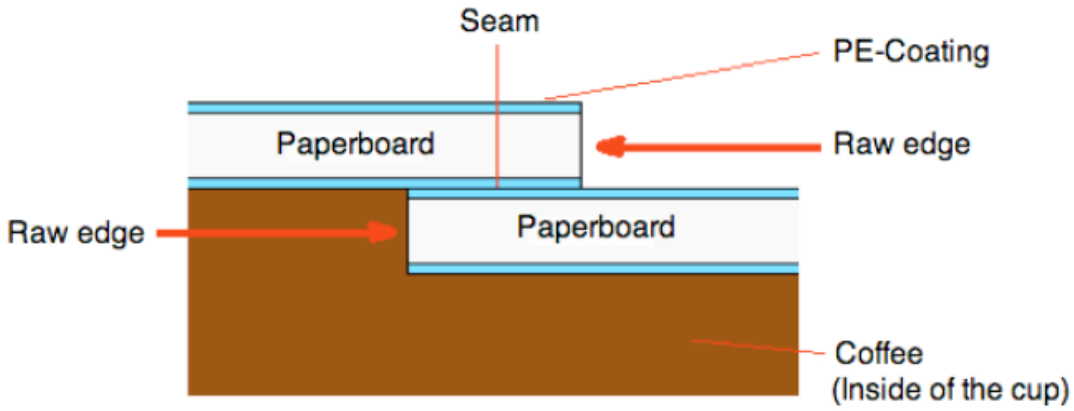

Figure 7. *The unprotected raw edges of a coffee cup (Kastinen 2010)*

As it can be seen in Figure 7, the raw edge is unprotected from liquid and it is vulnerable to edge wicking. PE-coating covers only the wide flat surfaces and raw edge is not large surface, but important factor during the time of usage of the paperboard cup.

The paperboard usually consists of three layers, and the liquid may penetrate into the layer which has the lowest penetration resistivity. Through this layer liquid can enter the other layers. Vapor diffusion and moisture sorption can start such liquid penetration when the pressure of liquid is greater than the flow resistivity of the paperboard. (Myllys 2007) Liquid penetration is described as a linear function of the contact time between paper and water, because the rate determining step includes the molecular processes ahead of the

liquid front. This has been verified with fluorescence size test. (Salminen 1988, pp. 17-22)

One of the mechanisms for water transport in paperboard is diffusion of vapor into the pores. It happens when the transport with capillary pressure is not possible, like in the case of hydrophobic papers. Sizing makes the paperboard more hydrophobic, but it does not protect the paperboard from vapor penetration (Myllys 2007). The movement of water vapor through paperboard is mainly controlled by the adsorption of water vapor on cellulose. This mechanism is not well understood, like the effect of osmotic pressure. The amount of osmotic pressure is depended on the total ion concentration between the exterior and interior of the fibers. It is studied that the osmotic pressure differential between the external medium and the fiber wall can be overcome only by adding water into the cell wall. Diffusion is a complicated phenomenon, because in the paperboard it can happen in the pores, on the fiber surfaces and within the fibers. (Salminen 1988, pp. 23-33)

3.1 Surface properties and contact angle

Surface does not only mean top and bottom sides of the paperboard, but also the surface of the raw edge. The surface tension and the contact angle have close relation to each other, because the contact angle depends on the surface energy of liquid. The surface tension is the force per unit length which can be calculated with formula

$$\gamma = \frac{F}{\delta x}, \tag{1}$$

where F is the force acting tangentially to the surface and at right angles to an element δx, which is the length of edge of the liquid surface in meters. There is an imaginary line drawn in the surface, where the tension is calculated with force F and element δx. The force F tends to reduce the liquid surface. (Barnes & Gentle 2011, pp. 10-40; Kastinen 2010)

The contact angle tells about the relationship between liquid drop and solid surface. The magnitude of the angle determines behavior of liquid, if there is wetting or not. The contact angle θ can be calculated with Young's equation

$$\cos\theta = \frac{\gamma_{SG} - \gamma_{SL}}{\gamma_{LG}}, \tag{2}$$

where γ_{SG} is the surface energy between solid and vapor phases, γ_{SL} is the surface energy between solid and liquid and γ_{LG} the surface energy between liquid and vapor phases. (Barnes & Gentle 2011, pp. 10-40) The contact angle can also be measured by observing it with a digital camera (Tavisto et al. 2003). In the Figure 8 the different contact angles can be seen.

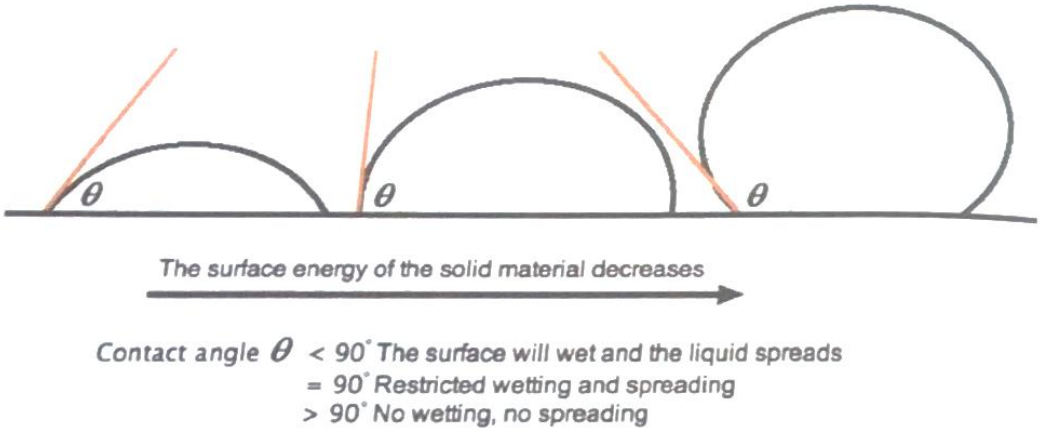

Figure 8. *Different water contact angles (Kastinen 2010)*

When the contact angle is < 90° like in the first picture in Figure 8, the liquid wets the solid and the surface is considered hydrophilic. When the angle is > 90°, wetting does not occur and the surface is considered hydrophobic. The angle cannot be greater than 180°, and perfect wetting happens when the angle is 0. (Barnes & Gentle 2011, pp.10-40; Kastinen 2010)

When the top and bottom surfaces of the paperboard are protected with plastic (extrusion coating, dispersion coating, tape or lamination) against liquids, the raw edge is the only surface which can have the contact angle smaller than 90°. When the paperboard is completely immersed in liquid, the surface energies of plastic covered, highly hydrophobic, top and bottom do not have an effect on the surface energy of the raw edge. (Chattoraj & Birdi 1984)

3.2 Porosity of paperboard

Paperboard has porous structure because of fibers. The microscopic pores are located between the fibers, so they form long, narrow capillary tubes and are connected to each other. Liquid can penetrate into paperboard either via the pores or by via fibers. The larger the radii of pores are, the greater is the probability that the penetration happens via pores than the fibers (Roberts 1991, pp. 97-112). The porous structure allows the liquids and gases to absorb into the paperboard, which effects on many of the paperboard's desired but also non-desired properties. The porosity consists of the pore radius and the total pore volume, and they have an influence on water transport into the paperboard. The pores with different radii affect also the contact angle, and some calculations include the radius of pore. The porosity can be calculated with the help of thickness and density. (Bristow & Kolseth 1986, pp. 183-201; Kastinen 2010; Nemez 2013; Salminen 1988, pp. 59-82)

The porous structure is taken into account in the formula of Darcy's law, which models the flow through porous medium (Pucci et al. 2015). It is usually used to describe slow, linear and steady state flow rate.

$$q = -K\frac{\Delta P}{L_0} = -\frac{k}{\eta}\frac{\Delta P}{L_0}, \quad (3)$$

where q is flow rate, K flow conductivity, k is permeability of the medium, η the fluid viscosity, ΔP net pressure head and L_0 the length of the sample. Porosity, tortuosity and specific surface area are included in the permeability of this equation. (Kastinen 2010)

Kozeny-Carman equation is used to calculate permeability k when the pores are non-circular

$$k = \frac{\phi^3}{k' S_0^2 (1-\phi)^2} \propto \frac{\phi^3}{(1-\phi)^2}, \quad (4)$$

where k' is Kozeny constant, ϕ porosity and S_0 surface area of the channels per unit volume of the solid material. Porosity ϕ is calculated by dividing the pore volume by the total sheet volume. Kozeny constant k' includes tortuosity and shape factors. (Kastinen 2010)

Porosity can be calculated also visually from a cross-section of the paperboard. The ratio of air and fibers is defined from the microscope picture. The effect of the pore size for the edge wicking is processed more precisely in chapter 4.4. (Widiatmoko et al. 2010)

Hard sizing makes the paperboard more hydrophobic, as it is told later in chapter 4.2, but if the paperboard has an open structure, the contact angle will be high. The porosity affects even when internal sizing protects the fibers. (Roberts 1991, pp. 97-112)

3.3 Wetting and swelling

The term wetting means displacement of a fiber-air interface with a fiber-liquid interface in the paperboard. When water and paperboard are in touch, water can penetrate into the porous structure or into fibers, which causes wetting. Wetting causes capillary forces, which control the spontaneous flow of liquid in the pores. That makes wetting as a result of spontaneous edge wicking in a capillary system. The wettability is therefore a perquisite for the occurrence of edge wicking in fibrous materials. (Bristow & Kolseth 1986, pp. 183-201; Kissa 1996)

The wetting happens only if the surface tension of the liquid is lower than the surface energy of the paperboard. When the top and bottom surfaces of the paperboard are protected against liquids, the raw edge is the only surface which can have higher surface energy than the surface tension of liquid. (Chattoraj & Birdi 1984) This applies also the

wetting of the capillary walls. Sizing changes the surface energy of paperboard, which protects the paperboard from wetting. The penetration ability depends on penetrating capacity of liquid and difference of the surface tension of the liquid and the surface energy of the paperboard. (Clark 1978, pp. 467-474; Holik 2006, pp. 87-105)

When wetting occurs in fibers, it causes swelling and weakening of the strength of the fibers (Rhim & Kim 2009). The phenomenon called hygroexpansion covers both swelling and shrinking and is a result of the hygroscopic nature of its constituent wood polymers. The swelling increases the pore size and decreases the contact angle. Swelling of the fibers also destroys the structure of paperboard and is influenced by the chemical and physical properties of the liquid and fiber network. The network structure is opened up and fibers relaxed because of the broken hydrogen bonds. The changes to the structure and properties of the paperboard may be caused by the imbibition of liquid into the interior of the fibers. (Cotorobai et al. 2016 ; Nemez 2013 ; Kastinen 2010)

3.4 Capillary effect

Capillary effect is the tendency of liquids to move along capillary tubes which are in the structure of fibrous materials. Those capillaries are narrow tubes which are located between the fibers in the structure of paperboard. The tendency is a result of the surface-tension force which arises at the tube wall-liquid interface. The capillary effect, when the liquid wets the surface, can be calculated with the increase of the liquid volume with the formula

$$\frac{2\sigma \cos\theta}{\rho g R}, \tag{5}$$

where σ is the liquid-air surface tension, θ is the contact angle, ρ is the liquid density, g and R the radius of tube. (Atkins & Escudier 2013)

The general absorptiveness of paperboard or capillary rise can be calculated with Lucas-Washburn formula

$$\frac{dl}{dt} = \frac{r \times v \times \cos\theta}{4\eta l}, \tag{6}$$

where l is the penetration depth in time t, r is the radius of pore, v is surface tension of liquid, θ the contact angle between liquid and surface and η is the viscosity of liquid. This means that the penetration speed increases when the pore size grows, surface tension increases, the contact angle is small and viscosity decreases. (Häggblom-Ahnger & Komulainen 2001, pp. 78-111) The Lucas-Washburn model assumes that liquid penetrates in an open pore with a constant radius. It also assumes that the pressure drop due to inertial effects and air transport is minor. The driving potential is the sum of the external and

capillary pressure. (Salminen 1988, pp. 17-22) The integrated form of Lucas-Washburn equation is

$$h^2 = \frac{r\gamma \cos\theta t}{2\eta}, \quad (7)$$

where h is depth of the liquid penetration in centimeters, γ surface tension of the liquid in dynes per centimeter and t is time of penetration in seconds. This means that capillary model is valid, when sorption into a pore system is proportional to the square root of the sorption time. (Bristow & Kolseth 1986, pp. 183-201; Libby 1962, pp. 40-59)

The capillary pressure can be expressed by the Young-Laplace equation

$$p_C = \frac{2\gamma \cos\theta}{r}, \quad (8)$$

where γ is surface tension of the liquid, θ contact angle between liquid and board and r constant pore radius of cylindrical capillary. This means, that the capillary pressure is linearly dependent on the cosine of the contact angle and the surface tension of the liquid, and the decrease of surface tension increases the water transport rate and the capillary pressure. (Salminen 1988, pp. 17-22, 41-58)

The capillary pressures are opposed by the pressure drop due to liquid flow which can be calculated with equation

$$p_F = \frac{8\eta l(\frac{dl}{dt})}{r^2}, \quad (9)$$

where η is the viscosity of the liquid, l penetration distance and dl/dt is penetration velocity. The pressure equation at the liquid front is expressed with formula

$$p_E + \frac{2\gamma \cos\theta}{r} = \frac{8\eta l(\frac{dl}{dt})}{r^2}, \quad (10)$$

where p_E is the external pressure and the penetration distance l is proportional to the square root of the penetration time

$$l = \sqrt{\frac{r\gamma \cos\theta + p_E r^2}{2\eta}}\ \sqrt{t}, \quad (11)$$

The classical capillary transport model can be calculated with this formula where the external pressure is taken to be zero. When the external pressure increases, it can cause negative capillary pressure of the paperboard structure. When the pressure is greater than the flow resistivity, the liquid starts to penetrate. (Myllys 2007; Salminen 1988, pp. 17-22)

Because of swelling of the fibers, the calculation of the total water penetration depth requires a correction factor:

$$l = \sqrt{\frac{r\gamma \cos\theta (t - t_k)}{2\eta}} - k\Delta Z, \tag{12}$$

where k is constant and ΔZ is the increase in the thickness at time t. This formula supposes that the mechanism for water transport in the paperboard is diffusion and does not take into account other methods. (Salminen 1988, pp. 17-22)

Capillarity theory has a lot of analytical models but also concrete tests to measure it (Mark et al. 2012a). Klemm test is an old capillarity test, which is performed for absorptive or "closed" products (top and bottom of paperboard protected with plastic) so that the wicking can occur only through raw edges. The lower end of a vertical sample paperboard is dipped in distilled water and after 10 minutes the height of the water rise is measured. The result is given in millimeters and it indicates the lateral porosity of the paperboard to water. The results made in the same conditions can be compared and the test procedure is described more precisely in the ISO Standard 8787. The side effects caused by differences in the wetting of the different pulp fibers may be eliminated by adding the detergent in the distilled water. It also may eliminate the differences in the surface tension caused by impurities dissolved in the water. (Clark 1978, pp. 380-401; Myllys 2007)

4. FACTORS AFFECTING EDGE WICKING

There are several factors which have an effect on the edge wicking of paperboard. Raw materials, manufacturing and converting processes as well as testing conditions affect the edge wicking. The liquids and other food that is kept in the paperboard products cause the edge wicking. Grease, temperature and pH of products may have different effects on the phenomenon. (Salminen 1988, pp. 41-82)

The pore volume and fiber swelling are important factors of the edge wicking. The structure of the paperboard as well as fibers used in pulp have an influence on edge wicking, and to understand the phenomenon it is important to research them. (Bristow & Kolseth 1986, pp. 183-201)

4.1 Fibers

Different fiber types have different effects on the edge wicking. The food packaging and serving field requires high hygiene, and the outer layer of paperboard packages is usually made from bleached chemical pulp. Food service boards do not usually contain recycled pulp, because the food contact materials are not allowed to include any contaminants. (Kirwan 2012, pp. 353-383; Kostamo 2014)

Chemical pulp is made by long, strong, flexible and collapsed fibers. It has low porosity and high pulp strength. The chemical pulp is used for purposes where strength is important. The strength is even better when the pulp is unbleached. The paperboard made from chemical pulp does have better resistivity to edge wicking than for example mechanical pulp, because its density is greater and fibers are longer. (Ek et al. 2009; Häggblom-Ahnger & Komulainen 2001, pp. 18-36; Nemez 2013)

Mechanical pulp is the opposite; it is made from short, weak and uncollapsed fibers and it has high porosity and low pulp strength. The mechanical pulp is used for its good printability. High porosity means more pores between fibers, which means that liquid can penetrate more easily in to the structure of paperboard. Mechanical pulp has higher porosity than chemical pulp so it has also greater edge wicking. Also the higher content of hemicellulose makes the mechanical pulp to swell more, because hemicelluloses work as swelling agents in the wood fibers because they are less restricted from their physical state. (Ek et al. 2009; Henriksson & Gatenhalm 2002, pp. 55-64; Nemez 2013) The mechanical pulp is made from fibers which are more inclined to swelling than whole fibers in the chemical pulp.

The middle layer of the paperboard is bulkier, and it can be for example CTMP. According to Kline 1982, CTMP is produced from chips pretreated with chemicals prior to

steaming. The pulp is then refined in the pressurized refiner with or without subsequent pressurized or atmospheric refining stages. Therefore, the properties of the chemi-thermomechanical pulp are in the middle of the chemical (sulfate) and mechanical pulp (Ek et al. 2009; Nemez 2013). The CTMP has more swell ability than the chemical pulp because of hemicellulose, so the liquid will penetrate more easily to it. This means that because of the wetting of CTMP, the pulp content expands and creates up to three times more pore volume than dry CTMP. The bulky middle layer has lower resistivity to liquid penetration than stiffer outer layers, and when the liquid penetrates into the structure, it can cause delamination. (Henriksson & Gatenhalm 2002, pp. 55-64; Kastinen 2010) The hardwood fibers have more hemicellulose than the softwood fibers, which means that hardwood swells more easily (Moring 2012).

Unrefined pulp makes the paperboard more exposed to edge wicking, and the paperboard made from unbleached pulp has greater swelling potential. The unrefined pulp has weaker bonding strength which may have an effect on the edge wicking results, as well as the lumens at the unsized edge. (Tufvesson 2006) The softwood (spruce, pine) pulp has a larger average pore size, because the softwood fibers are longer than hardwood fibers. Shorter hardwood fibers also create a more dense network. (Kastinen 2010; Myllys 2007) The length of softwood fibers is over twice as big as the length of hardwood fibers. Softwoods also have less cellulose and hemicellulose, but more lignin than hardwoods. (Smook 1992, pp. 9-19)

4.2 Sizing

The purpose of sizing is to improve paperboard's hydrophobicity and resistivity to the penetration of water and water solutions (Libby 1962, pp. 40-59; Roberts 1991, pp. 97-112). It also retards the edge wicking of paperboard (Kirwan 2012, pp. 1-50). The other purpose is to allow inks to remain on the surface and not to penetrate into the paperboard (Libby 1962, pp. 41-59). There are two kinds of sizing processes, internal and surface sizing. The internal sizing process is usually made in wet end, and it is in majority of sizes. The surface sizing process is made in dry end. (Halpern 1975, pp. 126-185; Smook 1992, pp. 283-296) Internal sizing protects the paperboard both from the edge wicking and the liquid which comes from possible pinhole defects of the PE-layer (Myllys 2007).

Hard sizing can retard the edge wicking of paperboard (Kirwan 2012, pp. 1-50). The edge wicking protection effect of sizing comes from the hydrophobicity of the sizing agent. It minimizes the capillary pressure, providing the capillary barrier. The barrier reduces the liquid penetration through the capillary system which means that the capillary transport under no external pressure is prevented. The sizing does not, however, affect the water vapor penetration. Adding the hydrophobic sizing agent on the surface of the fibers and fillers, will control the liquid penetration into the paperboard. The decreased surface energy can cause reduced adhesion between the PE-coating and paperboard and reduced

paper-paper friction values. (Kastinen 2010; Myllys 2007; Nemez 2013; Salminen 1988, pp. 59-82)

4.2.1 Sizing agent

There are three traditional sizing agents for paperboard; rosin, alkyl ketene dimer (AKD) and alkenyl succinic anhydride (ASA). These are internal sizes which are made by wet end fiber modifications (Samyn 2013). The choice of the sizing agent depends on the grades of the paperboard being made and on local water conditions as well as the conditions of the paperboard machine (Libby 1962, pp. 40-59). Different liquids also work better with different sizing agents (Kastinen 2010; Myllys 2007).

Rosin is used as a sizing agent in the combination with the alum (aluminium sulphate) in neutral or acidic conditions and it is the most common sizing system (Myllys 2007). The neutral rosin size (sodium rosinate) is used in soft water conditions, but in hard water conditions the acid size (rosin acid) is easier to use because of its bigger calcium oxide content. When the calcium content is greater than 50 ppm, the sizing is not efficient because the calcium ions and neutral size react and form calcium rosinate which has tendency to precipitate in the lines. (Roberts 1991, pp. 97-112)

Rosin is a natural sizing agent that is obtained from southern pine. The chemical content of rosin is abietic acid and about 15 % rosin acids. It is used as a sizing agent because it is insoluble in water. (Larsson 2008; Salminen 1988, pp. 41-58) Rosin in the paper and paperboard can be measured according to standard procedure of Technical Association of the Pulp and Paper Industry (TAPPI) T-408. The liquid penetration resistivity effect of rosin can be seen immediately, because the anchoring to the cellulosic surface is achieved by electrostatic interactions between the size and adsorbed alum (Larsson 2008). The hydrophobicity of rosin is lowest of hydrophobic sizing agents (KnowPap 2005). Rosin is the best choice for H_2O_2 and coffee, and it has poor penetration resistivity for water and lactic acid. Table 1 summarizes the penetration resistivity of sizing agents for various liquids.

Table 1. *Penetration resistivity of sizing agents for various liquids (Kastinen 2010; Myllys 2007)*

	Water	Hydrogen peroxide	Lactic acid	Coffee
Excellent	AKD	Rosin	AKD	Rosin
Good	ASA	ASA		
Average			ASA	AKD
Poor	Rosin	AKD	Rosin	ASA

Water and coffee are (Table 1) often served from paperboard cups. Milk and juice contain lactic acid, which is the reason why that is interesting to research. Hydrogen peroxide (H_2O_2) belongs to the process of liquid packing; the package is disinfected with H_2O_2.

Alkyl ketene dimer (AKD) is a synthetic, reactive sizing agent. The parent molecule of AKD, diketene, is able to derivate hydroxyl groups of cellulose. AKD is used under neutral or alkaline conditions and it consists of wax particles. Those particles are manufactured from the acid chlorides and they are in the water solution. The cationic particles used as stabilizers prevent colliding and sticking together. Because AKD needs alkaline conditions, the paperboards sized with AKD are suitable for milk and juice, which contain lactic acid (Larsson 2008). The sizing process of AKD begins with the retention on the pulp fibers and continues with spreading AKD over the fiber surface when the web is heated. AKD has a good hydrolytic stability, which can last for several months. The sizing state of AKD can last for several days, and it is difficult to measure for sure. AKD can be used in combination with rosin and it is called dual sizing. (Alén 2007, pp. 122-162; Larsson 2008; Roberts 1991, pp. 114-131) AKD has an excellent penetration resistivity against water, but poor against H_2O_2. This can be seen more precisely in Table 1.

The third common used sizing agent is alkenyl succinic anhydride (ASA). It is used in the alkaline or neutral conditions. ASA reacts easily, much more easily than AKD, and they can be used in sizing without heat treatment. ASA covers better the surface through formation of hydrolyzed products that can have higher contact angles, but it has lower retention than AKD. ASA's sizing state is as short as rosins, except when sizing the mechanical pulp (KnowPap 2005). ASA is prepared from the 1-alkenes by cationic isomerization and after that there is an addition reaction with maleic anhydride. ASA is not stable towards hydrolysis, and they have to be prepared right before the use. The sizing of ASA is ready right after the dryer section. ASA combined with styrene-based sizing can have maximum contact angle up to 100°, while the pure ASA has only 90°. The sizing efficiency can be added also by adjusting the amount of dissolved calcium and bicarbonate

ions, temperature and pH. (Nemez 2013; Roberts 1991, pp. 114-131; Samyn 2013) ASA has good penetration resistivity against water and H_2O_2 but poor against coffee. This can be seen in Table 1. Both synthetic sizes, AKD and ASA, have better sizing efficiency and they can achieve higher absolute sizing levels than rosin sizes (Myllys 2007).

In addition to traditional sizing agents, there are also newer innovations. Examples of those are quaternary ammonium salts, amines, fatty acids and their anhydrides, ethylene copolymers, fluorinated polyurethanes and surface-modified amphiphilic talc. The use of fluorochemicals though has to be reduced because of the environmental issues. Synthetic copolymers can replace the rosin, because there is no need for adding alum as fixation agent. Those can reach the contact angles around 105° and the pH range they can work on is wide. (Samyn 2013)

4.2.2 Amount of sizing

Over 70 % of all paper and paperboard products are sized. Some are lightly and some hard-sized, and all food service boards are internally sized. The paperboard cups for hot liquids require a special surface sizing which prevents the penetration of water above the boiling point. (Libby 1962, pp. 40-59) The internal sizing is called hard sizing when there is large amount of the sizing agent. When there is only a little internal sizing agent, the paper is called slack-sized. These papers are for example newsprints. (PrintWiki 2015)

The food service boards should be protective against the edge wicking, and for that reason they are hard sized. The sizing state can be measured by Cobb test, which tells about the hydrophobicity of paperboard, and about the sizing state. The Hercules size test measures the penetration of liquid through the paperboard in the Z-direction (perpendicular to the plane of paperboard). (Alén 2007, pp. 122-162)

When the portion of the sizing agent is same, ASA and AKD give better hydrophobicity than rosin, which can be seen in Figure 9. When ASA is dosed too much, the sizing may become worse. (KnowPap 2005)

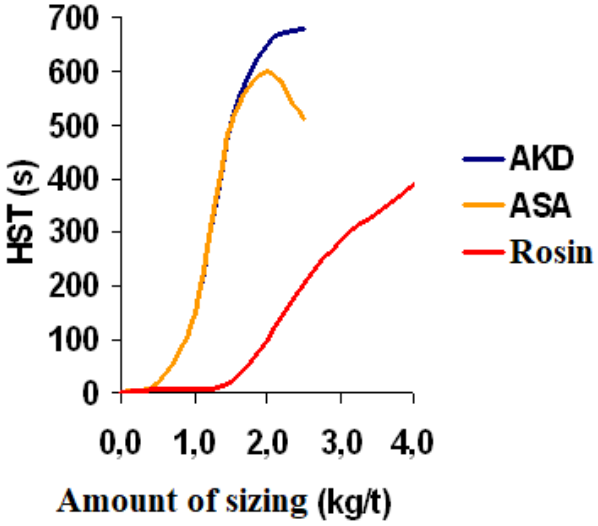

Figure 9. *Comparison of the amount of sizing. Original figure in KnowPap 2005*

In Figure 9 HST in the y-axis means the Hercules size test, which measures time when the test liquid penetrates through the paperboard.

The type of pulp has an influence on the sizing, because bleached pulps are more difficult to size than unbleached pulps. Kraft pulps only need one tenth of the size amount of α-cellulose pulp, which is very difficult to size. When there are lot of fillers in the pulp, also the amount of sizing is required to be bigger. (Libby 1962, pp. 40-59; Roberts 1991, pp. 114-131) Mechanical pulp is more hydrophobic than the chemical pulp, because it contains more lignin which is naturally hydrophobic (Häggblom-Ahnger & Komulainen 2001). The amount of ASA is bigger with the mechanical pulp than with the chemical pulp, because the retention of ASA is better with the chemical mass (Oila 2006).

Every small particle has its own contact angle with the liquid, which means that the amount of particles in the sizing agent is more important than the size of them. On the other hand, when the particles are small, there can be greater amount of them in the same amount of sizing and the effect of sizing agent is better. (Libby 1962, pp. 40-59)

The structure of the network has only minor changes due to the addition of size. On Parker Print Surf porosimetry study it is proofed that the sizing effect is achieved in rosin addition of 0,7 % and adding more rosin there is no effect on the capillary transport rate. (Salminen 1988, pp. 59-82)

4.2.3 Sizing state

Sizing state means the drying time of sizing, and with shorter sizing time the edge wicking results are worse than when the sizing has completely dried, especially in the hot water

EWTs (Tufvesson 2006). The sizing state can be tested by the water resistivity measurements, because the objective of sizing is to give hydrophobicity to the paperboard (Salminen 1988, pp. 59-82). Water resistivity can be tested in many ways, but only some of them are suitable for the EWTs. All sizing tests still tell something about the water resistivity and here are few of them listed: dry indicator, Valley tester, Currier tester, water absorption, Penescope, capillary climb, swelling test, angle of contact, Cobb and cup test. (Libby 1962, pp. 40-59)

There is standard to the Cobb water absorption test, and for that reason it is widely used. The test is performed according to standard ISO 535:2014. The sizing state changes over time and by Cobb test that can be followed. The sizing state can be determined by performing water absorption tests at regular intervals. Cobb water absorption test reveals the amount of water which absorbs to the paperboard in certain time, which indicates the sizing state of the paperboard.

Dry indicator measures the time of color change when paperboard is floated on the water and the mixture of dye and sugar is sprinkled on the upper surface. Valley and Currier testers measure the water penetration by conductivity. In Valley tester there is measured time for electrolyte to penetrate to the sample. In the Currier tester, electrolytes in the paperboard carry the current. Water absorption measures the amount of water absorbed in total immersion within the given time. In the Penescope test the sample forms side of a cell filled with lactic acid or water, where lactic acid penetration is shown by the indicator. The capillary climb test is, as the name says, based on the liquid climbing. The sample is put vertically in the water and it is measured how high the water rises in the sample within the given time. Swelling test measures the increase of thickness, and the angle of contact tells the magnitude of contact angle between the paperboard and the drop of the liquid. In the cup test the sample is formed as a cup and the water, which may be hot, is poured into it. (Libby 1962, pp. 40-59)

Hercules size test (TAPPI T-530) is used to measure the sizing state by the rate of penetration. It measures the resistivity of paperboard to permeation of the liquid and tells also the degree of sizing. The sufficient amount of the internal sizing can be determined with Hercules size test. There is a special device for Hercules size test. The sample is put in the holder so that that the tested side is up. Then the testing ink is poured onto the sample and the device is switched on. When the test is ready, the device will inform that and the time of test is recorded. When using the Hercules size test for mill control sizing tests, there are usually set reflectance endpoints in the range of 50 to 80 %. The test sample should be without any barrier coating so that the test works. In addition to water-based liquid, Hercules size test can be also used to measure the oil resistivity of the paperboard. The test is made the same way as the ink penetration test. (*Hercules sizing tester instruction manual* 2015)

4.3 Structure of paperboard

Porosity of paperboard has a great effect on the edge wicking, because the radii of pores indicate the penetration rate (Roberts 1991, pp. 97-112). Thickness, density, bulk and porosity of the paperboard characterize its physical appearance. The z-directional structure is described by the distribution of mass, size and fillers. (Niskanen 1998, pp. 89-115) The structure of paperboard can be evaluated with some basic tests. Thickness, porosity, bulk and density indicate the structure of the paperboard and that is the reason why they are measured. Also humidity has an important effect on the structure of paperboard. Thickness, bulk and density are measured according to standard ISO 12625-3:2014. (Kastinen 2010; Mark et al. 2012; Tufvesson 2006)

The value of thickness is used in measurements of the edge wicking. Thickness of paperboard should be as even as possible, and the transverse thickness profile affects most the fluency of process (Häggblom-Ahnger & Komulainen 2001, pp. 78-111). Grammage, in other words weight per unit area, is used in both density and bulk calculations. Grammage can be calculated according to standard procedure TAPPI T-410. Bulk is the reversal of density. Big bulk causes stiffness, compressibility and tear resistivity, but when the smoothness of the surface and tensile strength are desirable properties, bulk is usually decreasing. (Häggblom-Ahnger & Komulainen 2001, pp. 78-111) The weaker bonding strength and lumens at the unsized edge may have an influence on the edge wicking results of the low density paperboards. In general the low density paperboards have bigger edge wicking than the high density paperboards. (Tufvesson 2006)

If the relative humidity is too high, the water vapor starts to condense as a liquid in the capillaries and the paperboard loses strength and the dimensions can change. (Clark 1978, pp. 95-96; Kline 1982, pp. 19-28) The barrier coating prevents the paperboard from the moisture increase, but the raw edge is unprotected (Alsdorf 2009). The humidity of the paperboard can be measured according to standard ISO 287:2017(en).

The mechanical pressure occurs in the manufacturing processes like winding, printing and cup making. The mechanical pressure has an effect on the pore structure of paperboard. When the mechanical pressure increases, the surface roughness decreases and the liquid uptake is lowered. The mechanical pressure also diminishes the apparent pore radius, increases the pressure drop and decreases the liquid penetration. Due to the compression, the total pore volume of paperboard is decreased and the liquid penetration becomes more difficult. (Salminen 1988, pp. 59-82)

4.4 Liquids

The water absorptiveness is one of the most important properties of the paperboard cups, because liquids are the main uses of cups (Rhim & Kim 2009). The paperboard cups are tested both with liquids served in them and hydrogen peroxide. That is used to sterilize

liquid packages before filling, but the paperboard cups are not necessary to test with hydrogen peroxide (Larsson 2008).

Different liquids have different effects on the paperboard cups and edge wicking, because for example temperature, grease and pH react different ways. (Salminen 1988, pp. 41-58) The effects of surface tension, pore radius and viscosity are researched in the following chapters.

4.4.1 Food materials

The paperboard cups are often used to serve hot coffee, water, soda or ice cream. Different liquids have different effects on paperboard, because for example the different levels of the surface tension. Those differences are even more visible after few minutes, which can be seen in Figure 10. The diagram is based on the calculations of Laplace equation, which takes into account the surface tension and the contact angle. Those parameters are measured for this diagram. (Kastinen 2010)

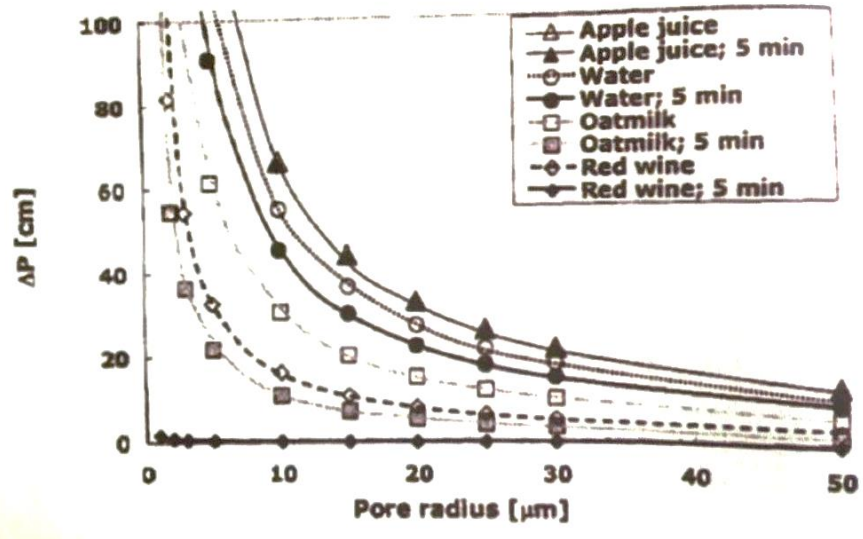

Figure 10. *Relationship between penetration resistivity for different pore radii (Kastinen 2010)*

In the diagram of Figure 10 ΔP in the y-axis is the penetration resistivity. The penetration of every liquid is measured both immediately and after 5 minutes. The oatmilk and red wine have the biggest differences between the penetration in the two measuring points. Red wine has almost not at all resistivity to the penetration after 5 minutes, and that is the reason why alcohol is usually not served in the paperboard cups. (Kastinen 2010)

The temperature of coffee or tea, the most common hot beverages served from the paperboard cups, can be nearly 100 °C. In Figure 11 the diagrams only show the temperature

until 80 °C, but especially the surface tension keeps decreasing when the temperature rises, as it can be seen in the diagram. The viscosity does not change much anymore in the higher range of the water temperatures. (Salminen 1988, pp. 44)

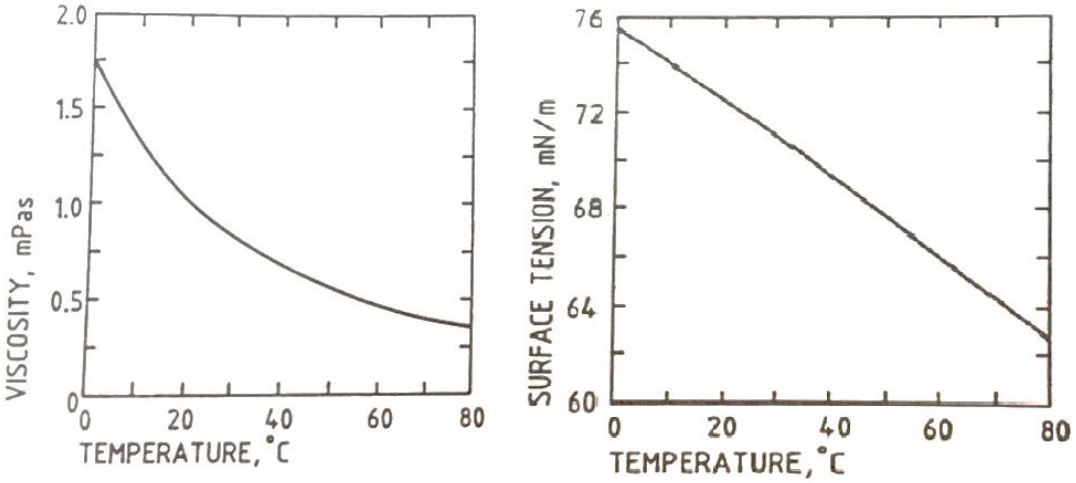

Figure 11. *Effect of temperature on viscosity and surface tension. Original figures from (Salminen 1988, pp. 44)*

The temperature rise of liquids influences on viscosity, surface tension and contact angle. The viscosity decreases when the temperature rises, and the liquid becomes more fluid. It is although researched that viscosity modifications do not have a strong influence on the liquid penetration. Also the surface tension decreases when the temperature rises, and when it happens, the contact angle decreases too and wetting happens more easily. (Salminen 1988, pp. 17-58) Those effects can be seen in Figure 11. The age of liquid may have an effect on the edge wicking. The biggest influence is with coffee, because fresh coffee does not penetrate as much as the 4 hours old coffee (Yinusa 2011).

Water and coffee, because of the grease content, act differently because of the polarity difference. Water and coffee have opposite results when choosing the best sizing agent. The fat molecules are nonpolar and water molecules polar, so they do not interact effectively with each other. In addition to liquids, the paperboard cups can also be used to serve food, which usually contains grease. (Zumdahl & DeCoste 2012, pp. 90-130) Even more grease comes, when milk or cream is added to coffee. The fats coffee contains are called lipids, cofestol and kahweol. The amount of lipids in the finished coffee depends on the manufacturing method. Espressos and coffees boiled without filtering have 40–110 mg lipids per 100 ml. The coffee brew filtered through a metal screener only has 32 mg lipids per 100 ml. Caffeine content has no influence on the amount of lipids, because both regular and decaffeinated instant coffees have approximately 1,2–4,4 mg lipids per 100 ml. (Ratnayake et al. 1993)

The paperboard consists of pores and fibers. Grease is absorbed only into the pores, but water is absorbed both into the pores and fibers, where swelling happens. Mineral oil is

non-swelling liquid, and it only fills the pores but does not go inside the fibers. The sizing effects on the paperboard so that the water does not enter into pores, but the fibers still absorb liquid and swell. (Bristow & Kolseth 1986, pp. 183-201)

The influence of pH is discussed in the literature. In the pH range from 1 to 13 the surface tension of water is almost independent of pH (Beattie et al. 2014, pp. 54-57). On the other hand, acidic liquid penetrates faster than pure water into rosin-sized paperboard. The influence of pH on the swelling of individual fibers may also have an effect on liquid penetration rate, but the influence of ionic strength could be misunderstood as the influence of pH. Milk and juice are end-products which contain lactic acid, which means that their pH is under 7, and they are resistant against alkalis. Many soft drinks contain artificial sweeteners like aspartame, which is manufactured from two amino acids. (Alsdorf 2009; Ashurst & Hargitt 2010, Larsson 2008; Salminen 1988, pp. 41-58; pp. 20-59; Samyn 2013; Zumdahl & DeCoste 2012, pp. 94-136)

Mineral water includes salts. The salt concentration has an effect on the liquid characteristics affecting the transport rate. The salt concentration does only have slightly influence on the viscosity and the surface tension of the liquid. Not even relationship between the salt concentration and the vapor pressure gives explanation the differences in the liquid transport rate. The decrease of the osmotic pressure differential between interior and exterior of the fibers is the most important factor of the salt addition. (Salminen 1988, pp. 41-58)

4.4.2 Test liquids

Surfactants have a low surface tension, which makes them able to penetrate easier into paperboard. Surfactants have a small influence on viscosity, but strong effect on the equilibrium surface tension of aqueous solutions. This is the reason why surfactants are used in the testing of edge wicking. (Salminen 1988, pp. 41-58) Even though the paperboard cup is used for water, the testing with pure water can be difficult, especially when using the optical testing methods. For that reason dyes are used in the measurements. It must be taken into account, that the dye addition modifies the transport velocity of water and changes the surface tension of it (Cotorobai et al. 2016).

Hydrogen peroxide (H_2O_2) is used to sterilize liquid packages before filling. The edge wicking is tested with the hydrogen peroxide solutions for that reason, but the food service boards will not be in contact with the hydrogen peroxide in their normal use. (Larsson 2008)

4.5 Testing variables

There are a lot of variables in the testing of edge wicking, and when the test procedures are performed badly, it may cause changes in the test results. It is important that all the

parallel tests are made the same way. The conditions and testing time should be standardized.

The basic measurements are made in the laboratory conditions where temperature is 23 ± 2 °C and relative humidity (RH) is 50 ± 2 %. The samples are usually held in these conditions for 24 hours before the test. In some occasions, if the humidity in the specific conditions is important to know, the sample conditioning is not used. (Libby 1962, pp. 373-398)

Cutting of the samples is very important, since that is the phase when the tested surface is made. A very sharp cutter or guillotine squeezes the edges of paperboard less and the edge wicking is more reliable than the edges cut with the blunt cutter. The raw edges also have to be clean and free from fibrous debris. (Kirwan 2012, pp. 265-312; Yinusa 2011)

The other part of the sample making is protecting the surface of paperboard. The edge wicking phenomenon only includes the absorption of the edge and does not want to explore the absorption of the top and bottom of paperboard. Some paperboards are extrusion coated, and those surfaces are not needed to protect with any lamination. The hot or cold lamination prevents the surface from the liquid, when only the cutting edge is wanted to be examined. Both of them protect the paperboard and are made before cutting of the samples and it is important that the test is made every time the same way. Also the age of the lamination film, the pressure and temperature of the lamination may effect on the results and they need to be standardized. (Larsson 2008; Yinusa 2011)

Whether the sample is extrusion coated, or hot or cold laminated, the adhesion of the barrier material has an influence on the edge wicking of paperboard. The adhesion of the lamination can be tested by hand peeling tests. It can be seen while measuring that when the lamination strength is good, also the resistivity against the edge wicking is good.

5. EDGE WICKING TESTS

There are many different edge wicking tests (EWT), but no standard. The units in the results of different tests are different and it is not easy to compare the results. Both hot and cold EWTs are made because liquids in different temperatures are often served from the paperboard cups.

The wicking phenomenon occurs in fibrous materials, not only in paper and paperboard. Most of the tests can be divided into two groups; gravimetric and optical methods. The gravimetric methods compare the weight of sample after the contact of test liquid. The optical methods are based on pictures and videos taken from the samples. Those two types of methods have also been used together sometimes. (Cotorobai et al. 2016)

Both sides PE-coated paperboard can be used directly in EWTs. If the paperboard is PE-coated only on one side or without any PE-coating, the sample must be coated on un-coated surfaces with hot or cold lamination. Cold lamination is made with tape and hot lamination with plastic film (Nemez 2013). Plastic coating prevents the liquid to penetrate from the top and bottom of paperboard, when the edge wicking is tested. (Kastinen 2010)

Edge wick with water according to Nemez (2013)

The test sheets are conditioned in 50 % relative humidity for 24 hours before the test and then the thickness is measured. The sheets are taped on both sides and cut into the sample pieces of 2,5 x 7,5 cm (1 hour) or 50 x 150 mm (24 hour). When using the smaller samples, the amount of test pieces is five and with bigger sample only one is needed. The sample pieces were weighed before the test. The test liquid bath is made with dyes, amaranth and allura red AC to see the differences between solutions. The samples are put into the bath completely under the surface with the help of weight. (Nemez 2013)

The samples are taken out from the bath and rolled over with the cob roller, between the blotter papers, for 1-2 seconds. The excessive test liquid was taken away with this procedure, and after that the samples are weighed again. The edge wick index according to Nemez is calculated with formula

$$EWI = \frac{m_2 - m_1}{t \times l}, \tag{13}$$

where m_1 is initial and m_2 final mass of sample in milligrams, t is thickness in millimeters and l is the total open edge length in meters of the sample. (Nemez 2013)

Coffee test

The samples are stored in the temperature of 23 °C and in the 50 % RH over the night. Thickness is measured and samples are coated with Perma Cell 910 -tape. The samples are cut in the pieces of 25 ± 0,5 x 75 ± 0,5 mm so that the shorter edge is in machine direction. The pieces are weighed and put into 80 ± 2 °C coffee for 10 minutes. After that they are slightly dried between blotting papers and weighed again. Raw edge penetration (*REP*) is calculated with the formula

$$REP\ (\frac{kg}{m^2}) = \frac{m_2 - m_1}{p \times t}, \quad (14)$$

where m_1 and m_2 are masses in grams before and after the test, perimeter p is the length of raw edges in meters and t is the thickness in millimeters. The coffee test can also be made by using coffee with cream. (Myllys 2007)

Hydrogen peroxide edge wicking

The samples are laminated with tape and cut into five pieces of 75 x 25 mm. They are weighed together and put into hydrogen peroxide (H_2O_2) bath so that they are completely under the liquid. The testing time is 10 minutes, temperature 70 °C and pH of the liquid is 1,5. The bath is 30 % H_2O_2 and a dye, either allura red AC or amaranth (Nemez 2013). After 10 minutes the samples are put between two blotting papers and rolled back and forth twice with brass roller. The samples are weighed together again and edge wicking index is calculated with formula of EWI (in the chapter 5). (Larsson 2008; Nemez 2013)

Lactic Acid edge wicking according to Larsson (2008)

The lactic acid edge wicking is measured because some end-products like milk and juice contain lactic acid, and paperboard cups have to be resistant to them. The method is similar to hydrogen peroxide edge wicking (0), but the test liquid is 1 % lactic acid solution with pH 2,4. In this method testing time is 24 hours, temperature 23 °C and size of the samples 50 x 150 mm. The test is only carried out for the sheets pressed at 400 kPa and it is made once. After measurements the EWI is calculated. (Larsson 2008)

Edge wick with lactic acid according to Nemez (2013)

The test is made at the same way with the test Edge wick with water (5), but the test liquid bath is 1 % lactic acid (pH 2,4) and dye mixed together. The edge wicking is calculated with the formula of EWI. (Nemez 2013)

5.1 Pressurized edge wicking tests

Liquid packages are often tested with pressurized EWTs and there are also some devices for that. For food service boards the pressurized tests are not that important, because the boards will not be under high pressures, like liquid packages during filling.

Larsson (2008) and Nemez (2013) have been tested pressurized edge wicking in a following way. Samples are laminated and cut into four pieces of 62 x 50 mm. A hole with a diameter of 12 mm was punched in the center and the pieces are weighed together. The test liquid is distilled water in pressure of 0,15 bars and the samples are put there for 20 seconds. After that the samples are put between two blotting papers and rolled back and forth twice with the brass roller. The samples are weighed together again and edge wicking is calculated with formula

$$EW = \frac{(m_2 - m_1)}{((\pi \times d)(n \times t)) \times 10^6}, \quad (15)$$

where m_1 is initial and m_2 final weight of the sample in milligrams, d is diameter of the hole in millimeters, n is the number of sample pieces (4) and t is the STFI-thickness in micrometers. The result is also analyzed visually. (Larsson 2008, Nemez 2013)

Mark et al. (2012a) do have a device to measure the pressurized edge wick and the method imitates the filling machine of liquid packages. The pressure-vessel is 250 mm by diameter and 190 mm by height which is (a) in Figure 12. The vessel is filled with water to a height of 90 mm and with temperature of predefined level.

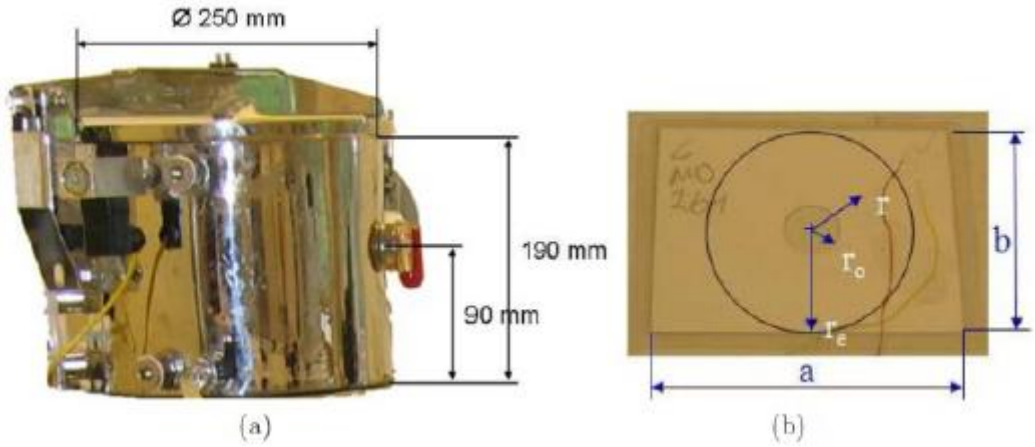

Figure 12. *The pressurized EWT device (Mark et al. 2012a)*

The sample piece (b) in Figure 12 is rectangular, PE-laminated on both sides and there is a punched hole with the radius of 6 millimeters. The sample piece is first weighed and then put to a holder in the water and the lid is closed. The pressure in vessel is 15 kPa and the test time 20 seconds. After that the sample is weighed again and the edge wick index E is calculated with formula

$$E = \frac{\Delta m}{2\pi r_0 h} \left[\frac{kg}{m^2}\right], \quad (16)$$

where Δm is the mass difference, r_0 radius of the hole and h thickness of the sample. (Mark et al. 2012a; Mark et al. 2012b)

Kastinen (2010) has studied the measurement device for vacuum EWT in his thesis. One measurement device records the kinetics of penetrative liquid into the paperboard through the raw edge. It is done visually by using digital camera, because the sample becomes more transparent when it is wetted. Vacuum pressure intensifies the penetration. The parts of this device can be seen in Figure 13.

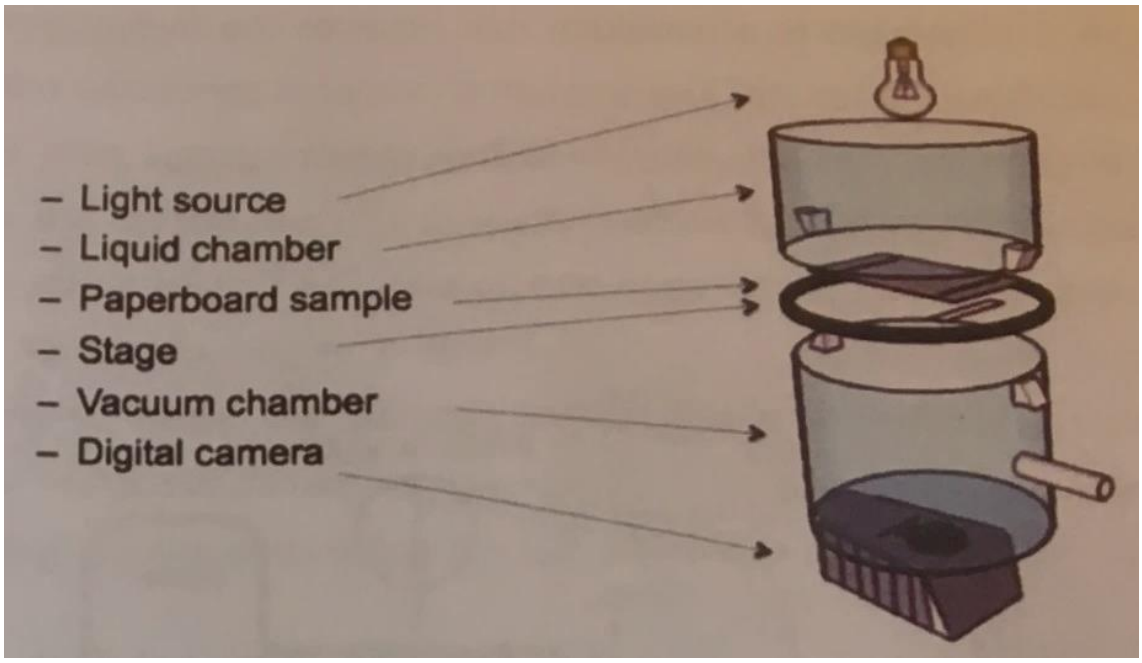

Figure 13. *Measurement device (Kastinen 2010)*

The sample is put on the stage which can be seen in Figure 13 and digital camera is connected to a computer, which calculates the results. (Kastinen 2010)

6. EXPERIMENTAL PART: LABORATORY TESTS

The aim of the experimental part of this thesis was to research how the results of EWTs change during the paperboard making and converting processes, which consists of board machine, pigment coater, winder 1, extruder and winder 2. Also the effect of time was studied by measuring the samples from the second winder after 5 weeks from the board machine.

The experimental part consists of different laboratory measurements, where the edge wicking as well as sizing state are experimented. The edge wicking was measured with different liquids, which model liquids that are in contact with the final products. The sizing state changes by time and it may have some effects on the edge wicking.

6.1 Background and objective of research

The knowledge of the edge wicking is important when making for example paperboard cups, because the liquid is able to penetrate into the paperboard from the raw edge of the product. The cup making industry is interested in the factors that affect the phenomenon and which can improve the resistivity of the product against it.

It has been noticed that the results of the edge wicking are changing during the paperboard manufacturing and converting process and therefore the phenomenon is wanted to study throughout the process. The second aim was to compare the measuring methods with each other and see the differences between methods, because there is no standard for EWT.

6.2 Materials and methods

The paperboards and liquids used in this research were simulating the materials used in the liquid serving. The sample paperboards were collected from Metsä Board's paperboard mill. The liquids used in the measurements, water and coffee, are often served from the paperboard cups.

Also grease could have been studied, if the end-products were used in serving greasy food. Coffee, one of the most served liquids from the paperboard cups, has also other properties which can have an effect on edge wicking e.g. temperature. For that reason the measurements were made with coffee and not only with hot water.

6.2.1 Materials

There were four tested paperboards which were named with numbers 1-4 in Table 2. All of them were Metsä Board's own products which can be used in food service application. The information about the paperboard grades can be seen in Table 2.

Table 2. *Sample grades*

	Bulk	Pigment coating	Grammage (g/m^2)	PE
Sample 1	High	No	225	Yes, back
Sample 2	High	Yes, top	200	Yes, back
Sample 3	Low	No	235	Yes, back
Sample 4	Low	Yes, top	245	Yes, top

The high bulk boards, which were named in Table 2, were sized less than the low bulk grades. All samples had chemical pulp in their top and bottom layers and bleached chemi-thermomechanical pulp (BCTMP) in their middle layer. Two of the samples were pigment coated on the top side. All grades were one side PE coated either on the back or top side. All samples were one side extrusion coated.

In the hot EWT the test liquid was coffee. In the cold EWT 1 there were two different water solutions, test liquids 1 and 2. Test liquid 1 had higher surface energy than test liquid 2. Test liquid 1 was used also in the cold EWT 2.

Sampling was made after each manufacturing and converting machine during the process. The sampling points can be seen in Table 3.

Table 3. *Sampling*

	Board machine	Pigment coater	Winder 1	Extruder	Winder 2	After 5 weeks
Sample 1	x	-	x	x	x	x
Sample 2	x	x	x	x	x	x
Sample 3	x	-	x	x	x	x
Sample 4	x	x	x	x	x	x

The measurements were made after every step which can be seen in Table 3. The last sampling point, the time tracking sample, was measured after 5 weeks from paperboard manufacturing. The samples were taken from the second winder and they waited for measuring until 5 weeks had passed from the day they were finished on the board machine.

The sample 4 was extruded 5 weeks after finished on the board machine. For that reason the samples from the second winder were measured after 5 weeks, as well as the time tracking samples. The separate time tracking measurements were not performed of the sample 4.

6.2.2 Methods

Moisture

The moisture content was measured immediately after each process step. The sample was weighed and put into the oven of 105 °C for 1 hour. After that the sample was measured again and the moisture content was calculated with the formula

$$Moisture\ content\ \% = \frac{m_1 - m_2}{m_1} \times 100\ \%, \tag{17}$$

where m_1 and m_2 are initial and final masses. The samples were not stored in the standard conditions between the measurements after second winder and time tracking measurements.

Thickness

Thickness was measured in two different ways. The first way was to measure the total thickness with the AB Lorentzen & Wettre Micrometer (type 221) for paper thickness testing. The test was performed in accordance with the standard ISO 534:2011(en). The theoretical thickness of PE-layer was excluded from the total thickness. The result after excluding the thickness of PE-layer was the thickness of paperboard.

The second way was to measure the thickness of the prepared samples from their cross-section with the help of stereomicroscope. The preparation of samples included laminating and cutting, and this method took into account the effects of them. This method measured the thickness of raw edge. Thickness was measured because the value was needed in the formula of EWI in both cold and hot EWTs.

Cobb test

Cobb test was made as $Cobb_{180}$ according to the standard ISO 535:2014(en). Cobb cannot be measured on the top of pigment coating or extrusion coating. Four parallel measurements were performed from each test.

The experimental way was to test the ready-made paperboard cup's $Cobb_{180}$ values on the area of side seam. The paperboard cup was cut so that Cobb value of the side seam could be measured. Because the paperboard cup was extrusion coated inside of the cup, the liquid was able penetrate into the paperboard only through the raw edge. Four parallel measurements were performed from each test.

Cold EWT 1

The samples were cold laminated with tape on both sides, except the PE-coated samples, which were cold laminated only on the uncoated side. The sample was cut to the certain piece so that the longer edge was cut in machine direction and then weighed. The sample piece was placed in the test liquid. The testing time was 30 minutes and after that the sample was dried and the penetration length was measured visually by using the microscope or loupe. The sample was weighed again and the EWI was calculated with the formula 18 below

$$EWI\ (\frac{kg}{m^2}) = \frac{m_2 - m_1}{t \times l}, \tag{18}$$

where m_1 is initial and m_2 final mass of the sample in grams, t is the thickness in millimeters and l is the total open edge length of the sample in meters. The cold EWT was made with both test liquids. The cold EWT was made both by the cold lamination with tape and hot lamination with film to see the differences of different lamination methods. Three parallel measurements were performed from each test.

Cold EWT 2

The samples were held in the oven of 105 °C for 10 minutes. After that they were conditioned in the room temperature for 10 minutes and hot laminated. The sample pieces were made with the die of 10x10 cm, put into the test liquid and held there for 15 minutes. Then they were dried gently and the penetration length was measured visually by using the microscope or loupe.

The cold EWT 2 was made also without keeping the sample in the oven to compare the results and the effect of aging of the paperboard. The oven accelerated the aging of the paperboard and the developing of the size. Both tests were made with test liquid 1. Three parallel measurements were performed from each test.

Hot EWT 1

The samples were cold laminated with tape on both sides, besides the PE-coated samples, which were cold laminated only on the uncoated side. The coated sample was cut to the certain piece so that the longer edge was cut in machine direction, weighed before the test and then put to a stick so that the certain part of the sample was in coffee. The sample

was held in the coffee for 15 minutes, then dried quickly and weighed again. The result was calculated with formula

$$Hot\ EWI\ (\%) = \frac{m_2 - m_1}{m_1} \times 100\ \%, \quad (19)$$

where m_1 is initial weight and m_2 final weight of the sample in grams. The penetration length was experimented also visually with the help of the microscope or loupe. The hot EWT was made both by the cold lamination with tape and the hot lamination with film to see the differences of different methods. Three parallel measurements were performed from each test.

Hot EWT 2

The hot EWT 2 was made as presented in the Coffee test (Myllys 2007) in chapter 5. Thickness was measured and the samples were cold laminated with tape. The extruded samples were cold laminated only on the uncoated side. The samples were cut in the pieces of 25 x 75 mm. The pieces were weighed and put into 80 °C coffee for 10 minutes. After that they were slightly dried between the blotting papers and weighed again. The edge wicking index was calculated with the formula

$$EWI\ (\frac{kg}{m^2}) = \frac{m_2 - m_1}{t \times l}, \quad (20)$$

where m_1 and m_2 are the initial and final masses in grams, t is the thickness in millimeters and l is the length of raw edges in meters. Three parallel measurements were performed.

6.3 Results and discussion

The measurements were made in two laboratories. The equipment were similar, and the earlier measurements were made in the first laboratory and the time tracking samples in the second laboratory.

There have been made different calculations and the results were given in different units from the same measurements. The results have been compared to each other and discussed. The standard deviations were calculated and they gave explanations to some results. The results have been compared to the theory.

6.3.1 Moisture

The moisture content of the paperboard changed during the process, probably because of the effect of time. The results calculated with formula 17 can be seen in Figure 14.

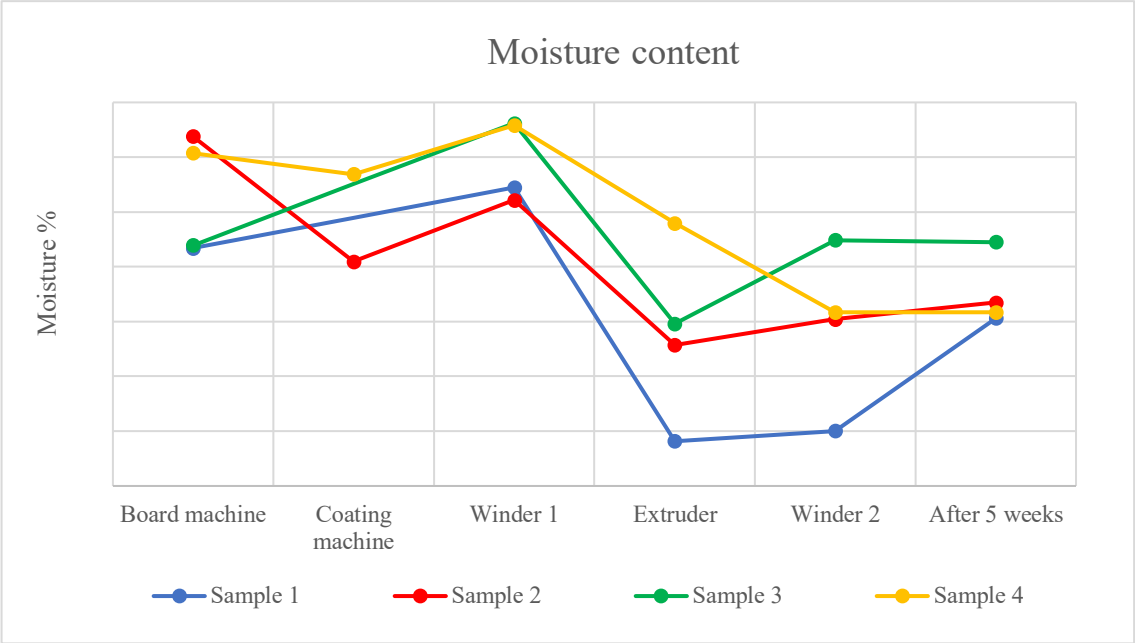

Figure 14. *Moisture content*

The lowest moisture content of all samples (except 4) seemed to be after extruder, which can be seen in Figure 14. The first winder and the extruder were placed in separate manufacturing halls, and it could have taken time the paperboard tambours to go from the first winder to the extruder. This may explain the fact that the moisture content decreased between the first winder and the extruder.

The moisture contents of the samples after 5 weeks were not easy to compare to other measurements, because the samples had not been stored in standard conditions. During the process the paperboard tambours were held in the paperboard mill, where the climate was warmer and more humid than the room temperature and humidity. The differences of the moisture content were not significant. The sample 4 reacted differently, because of the longer time between the first winder and extruder.

6.3.2 Cobb test

The $Cobb_{180}$ test was made on the top side of the uncoated paperboards and on the bottom side of the coated paperboards. The samples 1 and 3 were uncoated, 2 and 4 were coated and the results can be seen in Figure 15.

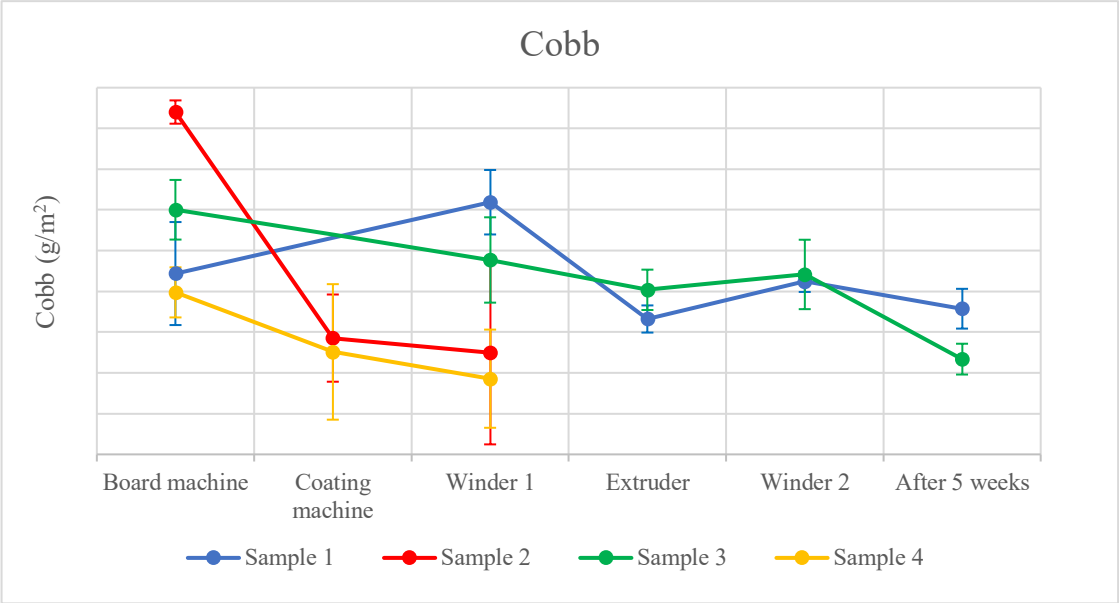

Figure 15. *Cobb results*

The lines in y-axis in Figure 15 are in increments of one unit and the unit is g/m^2. Cobb results of the uncoated samples 1 and 3 were measured after all machines, because Cobb was measured on the top side of the paperboard and there was not any coating. Cobb test results of the coated samples 2 and 4 were measured only after board machine, coating machine and first winder, because Cobb was measured on the bottom side of the paperboard, where the PE-layer was extruded. Cobb could not have been measured on the other side, because the pigment coating was on the top side of the paperboard.

The measurement days of Cobb can be seen in Table 4. The calculation began from the board machine. Day 0 was the day when the paperboard was finished at the board machine.

Table 4. *Measurement days of Cobb*

	Board machine	Coating machine	Winder 1	Extruder	Winder 2	After 5 weeks
Sample 1	1	-	1	4	4	34
Sample 2	0	4	4	-	-	-
Sample 3	1	-	2	2	2	35
Sample 4	0	3	2	-	-	-

All the Cobb tests were made within 4 days and the time tracking samples in 5 weeks which can be seen in Table 4. Behavior of the sample 1 in the Cobb test was slightly

different than the behavior of the other samples, but the measuring points where the curve increased, were at the same days, which could explain that.

Cobb values got smaller during the process, and the big difference between the measurements between the board machine and the coating machine can be explained by the fact that there were few days between them. The values from the coating machine and winder were measured at the same day. The first result was, however, quite big. The standard deviation of the sample 2 was quite big at the coating machine and at the winder. When taking that into account, the decrease between the board machine and the coating machine was not very significant.

The results of the low bulk sample 3 decreased during the process. The last value was smallest as expected, because of the development of sizing. The value after the second winder was bigger than after the extruder, but also the standard deviation was bigger at that point.

The differences between the Cobb values of sample 4 were rather small and they decreased during the process. The measurements were performed close to each other, during 4 days, but still the behavior of sizing could be seen. The effect of time to sizing state caused it that the values are smaller, because of the development of sizing.

The curves of coated samples 2 and 4 decreased during the process, and all the measurements were done within 3 days. The curves of uncoated samples 1 and 3 did also decrease from the board machine to the time tracking sample. Sample 1 had values measured at the same day, and they did increase. This means that the development of sizing happened, but the results were not necessarily seen during one day. Cobb test measures the development of sizing, and the results showed that development occurred during the process.

Experimental Cobb test

Experimental $Cobb_{180}$ 2 was performed so, that the ready-made paperboard cup was cut and Cobb was measured on the area of side seam. This area can be seen in Figure 16. Cobb was measured on the top of PE-layer, so the result was assumed to be very small.

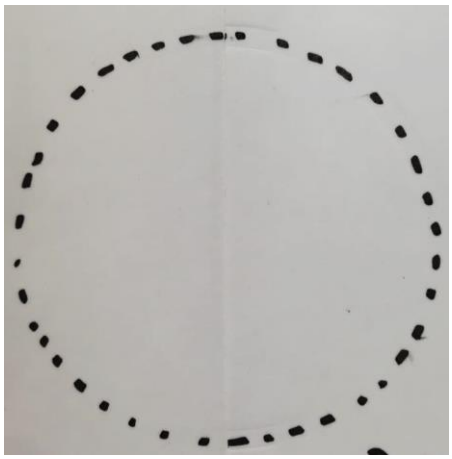

Figure 16. *Cobb test paperboard cup, tested area*

Figure 16 shows the area where Cobb was tested. The Cobb result of the paperboard cup without seam was assumed to be 0, so all the liquid which was penetrated into paperboard, was penetrated through the raw edge of side seam. Cobb results were calculated with the formula

$$A = (m_2 - m_1) \times F \tag{21}$$

where F is 10 000 divided with the tested area. The area of Cobb cylinder in this measurement was 25 cm^2, and the diameter was 5,6 centimeters. The area where the liquid was able to penetrate to the paperboard was very small in this experiment. Thicknesses and areas of the paperboards can be seen in Table 5. Thicknesses were measured from the microscope picture. The amount of water in the Cobb test depends on the area of cylinder. Because the experiment was performed with 25 ml of water, the area in the measurements was 25cm^2 and not the area of raw edge. The areas of raw edge can also be seen in Table 5.

Table 5. *Cobb test comparison*

	Cobb value (g/m^2)	Standard deviation	Thickness of sample (mm)	Area of raw edge (cm^2)
PE-coated cup for hot drinks	6,4	1,095	0,34	0,1904
PE-coated cup for cold drinks	1,6	0,490	0,32	0,1792
Dispersion coated cup for cold drinks	10,1	0,714	0,33	0,1848

The results of Cobb test can be seen in Table 5 and they were rather small. The polymer layer of the dispersion coated cup was thinnest, which could have an effect on the Cobb value. The amounts of sizing were not known, but the biggest effect on the Cobb result is the amount of internal sizing. Also the barrier properties of dispersion coating may not have been as good as the properties of extrusion coating.

The experiment was not easy to perform, because the Cobb equipment had to be very tight so that the liquid would not leak from the side seam. This experiment is reasonable when comparing the cups with each other. This has not been studied, so there are no reference values. All the cups are available to consumers, so their edge wicking is good enough, although the results differ from each other.

6.3.3 Thickness

The total thickness of paperboard was measured in two different ways. The samples measured by the micrometer were conditioned for 24 hours in the standard conditions (23 °C, RH 50 %) and then measured by the micrometer. The thickness of PE-layer was excluded. The thickness of PE-layer was actually not even, but the theoretical thickness was used in these calculations. The microscope thickness samples were measured from the stereomicroscope pictures (Figure 17), which were taken from the prepared samples. Those samples were cold laminated with tape to measure the edge wicking. When measuring from the microscope pictures, the effect of laminating the prepared sample could be excluded. This was made because the tape was believed to have some effect on the thickness of paperboard itself, and only excluding the thickness of tape would not give the exact result.

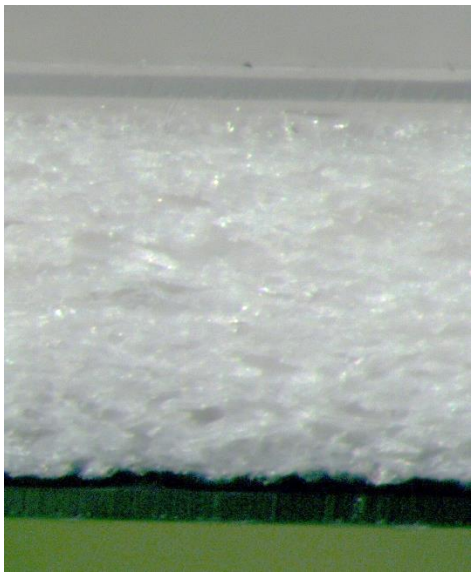

Figure 17. *Stereomicroscope picture of prepared sample*

The sample in Figure 17 was cold laminated with tape on the both sides. The plastic layer (tape) is easily shown in the picture, as well as the paperboard in the middle of the sample. The less clear areas between the paperboard and tape are the glue of tape.

The thickness of paperboard changed during the process. In Figure 18 the changes of thicknesses of the samples 1 (high bulk) and 3 (low bulk) can be seen.

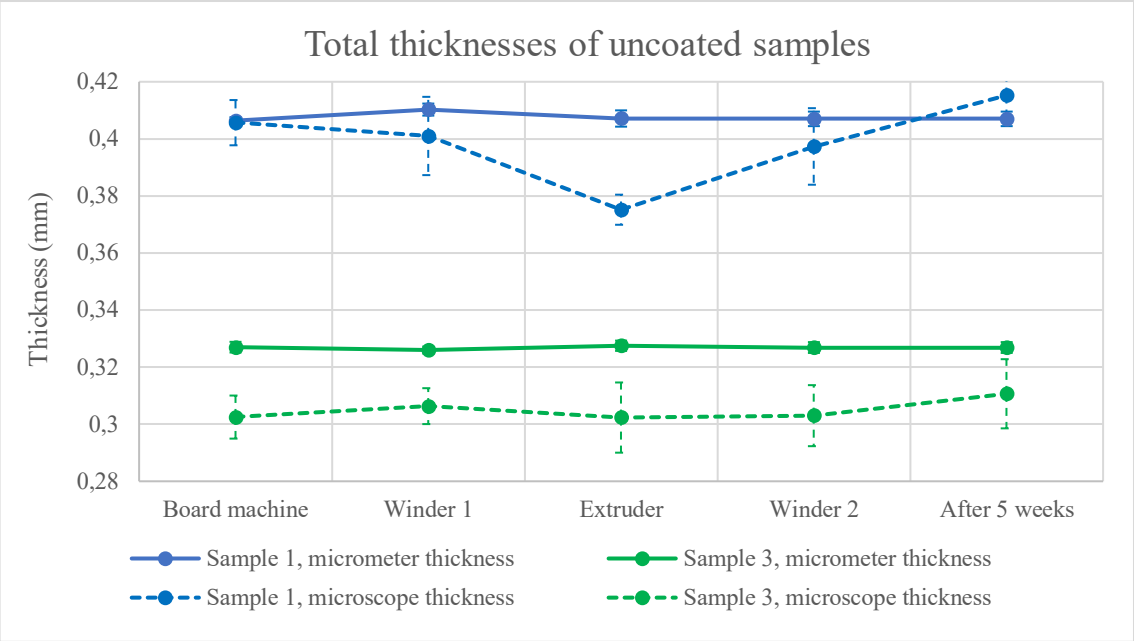

Figure 18. *Thicknesses of samples 1 & 3*

The diagram in Figure 18 shows that the thickness of the paperboard was smaller when measured from the microscope pictures. The difference was rather small especially when looking at the sample 3, approximately 0,025 millimeters in all measuring points. The sample 1 had even smaller difference in all the other measuring points, but after the extruder the difference was 0,032 millimeters although the theoretical thickness of the PE-layer was excluded.

The standard deviations of the thicknesses measured by the micrometer were very small. The biggest standard deviation of the sample 1, measured from the microscope picture, was 0,013 mm which was only 3,42 % of the mean thickness. This was measured after the first winder. The biggest standard deviation of the sample 3, measured from the microscope thickness, was measured after the extruder. The value was 0,012 mm and it was 4,07 % of the mean thickness.

There were two reasons to the difference between the thicknesses measured by the micrometer and from the microscope pictures. One was that cutting of the sample squeezed the edges of paperboard. The other was that the glue of tape spreads over the raw edge because of cutting. The thickness of the laminated paperboard, which could be detected

from the microscope pictures, was smaller than the thickness of the unlaminated paperboard. The spreading of tape's glue is difficult to prevent, because it happens also when cutting as sharp knife as possible.

Thickness should not change over time but a small difference could be seen when measured with the microscope. Micrometer thickness was not measured again. The difference of thicknesses measured with the microscope could be explained partly by laboratory changes. The measurements were not made in standard conditions.

The samples 2 (high bulk) and 4 (low bulk) had a pigment coating on the top of them, and when thickness was measured, thickness of the pigment coating was included in the results. Thicknesses in Figure 19 were measured the same way than thicknesses of the samples 1 and 3.

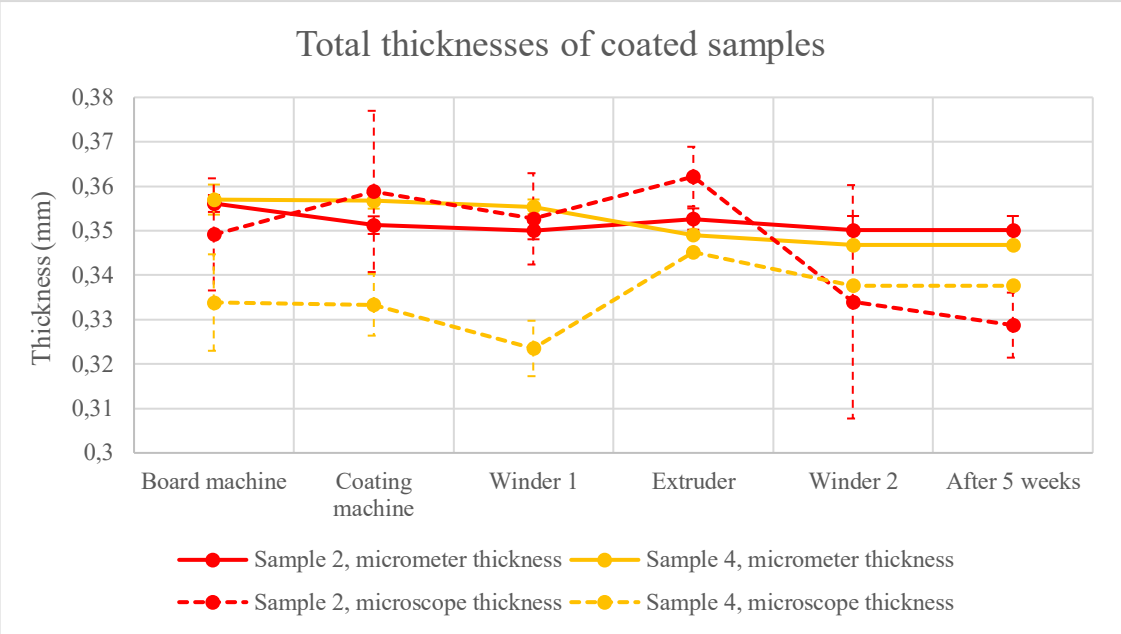

Figure 19. *Thicknesses of samples 2 & 4*

Same reason explained the difference of the thicknesses measured in different ways in Figure 18 and Figure 19. Difference of the thicknesses of the sample 2 was not linear. The thickness measured by the micrometer was almost the same all the time, and the standard deviation was also rather small, only 0,0032 mm. The standard deviation of the thickness measured from the microscope picture was quite big at the coating machine and at the second winder, 0,018 and 0,026 mm.

The difference of the sample 4 was more linear in the beginning. It was significant between the different measuring methods. After the extruder the differences were smaller. The standard deviations were rather small, but the difference between the thicknesses was approximately 0,026 mm from the board machine to the first winder. From the extruder to the second winder the mean difference was 0,014 mm.

The thickness of sample 1 measured from the microscope picture had the biggest deviation at the extruder. The curves were still close to each other. The sample 3 had two similar, linear curves with a constant difference to each other. The thicknesses of samples 2 and 4 measured by the micrometer were at the same level all the time. The thicknesses measured from the microscope pictures varied and the standard deviations did not explain the results. The microscope pictures were not easy to read, because measuring the interfaces of paperboard is subjective. The spread glue did not have clear interface with the paperboard.

6.3.4 Cold EWT 1

The diagram in Figure 20 shows the results of the cold EWT 1, uncoated high bulk sample 1. The results are given both as EWIs and penetration lengths in millimeters. The EWIs were calculated with the formula 18. The thickness measuring method was compared by using both thickness measured by the micrometer and from the microscope pictures. Test liquid 1 had bigger surface tension than test liquid 2.

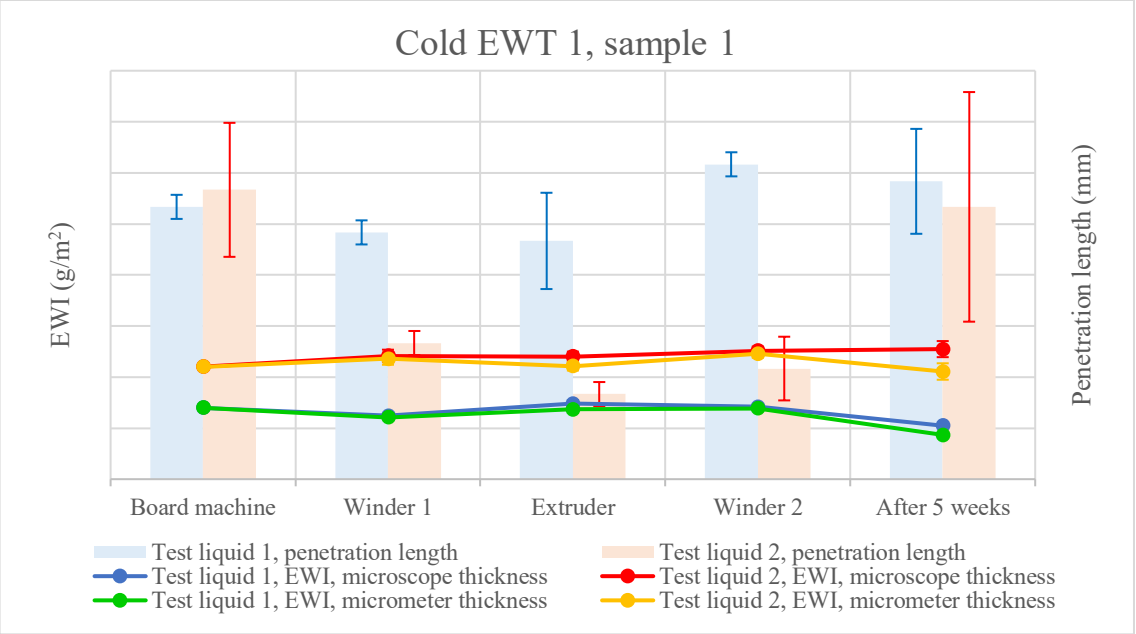

Figure 20. *Results of cold EWT 1, sample 1*

The lines in y-axis in Figure 20 are in increments of 0,2 unit. The results of EWI are curves and the results of penetration lengths are bars. Penetration length of the test liquid 1 was quite linear through the process and the standard deviations were not very big. Test liquid 2 had bigger standard deviations, and the values after the board machine and after 5 weeks were both bigger than the other values and had bigger standard deviations. At the lowest points those values were quite close to the other values.

The correlation between penetration length and EWI was not very clear. The EWI had bigger values with the test liquid 2 and the values of penetration length were bigger with

the test liquid 1. The test liquids reacted different ways, which can be seen in Figure 21 and Figure 22.

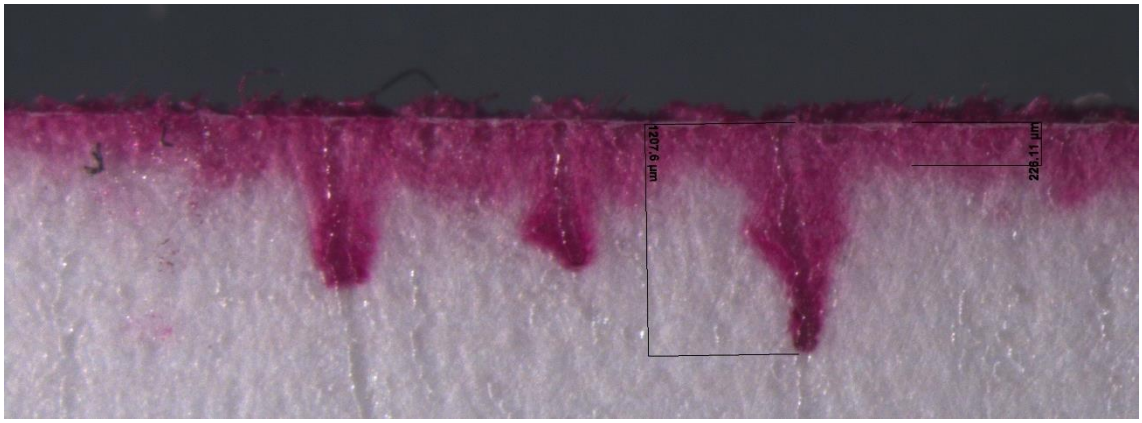

Figure 21. *Penetration of test liquid 1*

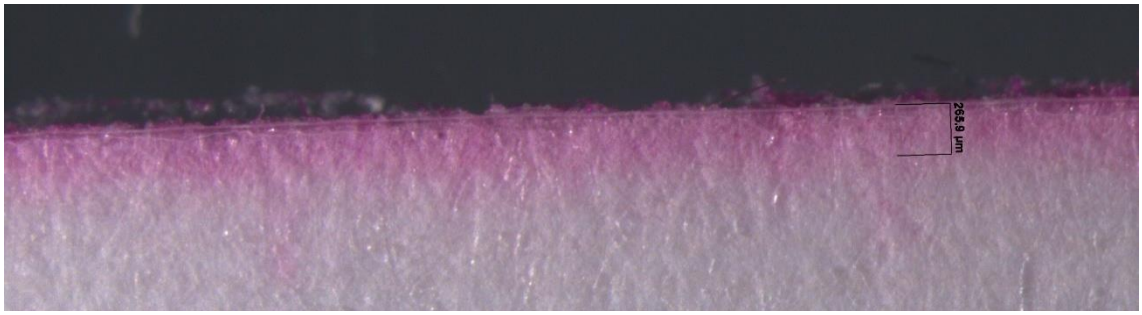

Figure 22. *Penetration of test liquid 2*

Test liquid 1 in Figure 21 penetrated with long, individual lines. The uniform front was much shorter than the individual lines. The penetration length was measured from the longest individual line. The test liquid 2 in Figure 22 did not form clearly visible individual lines and this made the difference of penetration lengths. Because the test liquid 1 had the visible narrow lines which give bigger penetration length results, the masses did not correlate and the EWI values were smaller than with the test liquid 2. The penetration lengths in Figure 21 were 1208 µm and 226 µm, but the length in Figure 22 was 266 µm. When the major penetration length was only 226 micrometers compared to 266 micrometers, the amount of penetrated liquid was smaller. Then also the final mass and the value of EWI were smaller. Test liquid 1 had bigger surface energy than test liquid 2, which meant that the test liquid 2 should have penetrated more to the paperboard. The EWI values did correlate with that, but the measured penetration lengths did not.

The length should have been measured from the uniform front (mean value), but it could be difficult to see how long it was. Detecting the uniform front was demanding and the differences between individuals might have arose. For that reason it would be more reliable to use the EWIs as results of edge wicking. Measuring the length is subjective method, but weighing and calculating the EWI is objective. The liquids seemed to act differentially, but the test liquid 2 had lighter color, which made it more difficult to detect.

This is another reason why the objective method would be better. If the penetration length is not needed to measure, the cold EWT may be performed without any dye. The dye may have an effect on edge wicking, which the liquids served in paperboard cups do not have.

The diagram in Figure 23 shows that the effect of measuring method of the thickness on the results of cold EWI, coated high bulk sample 2, was very small. The results of test liquid 1 were even closer to each other, and the biggest difference between the EWIs calculated with the thickness measured by the micrometer and the thickness measured from the microscope picture was under 3 %. This did not concern the time tracking results.

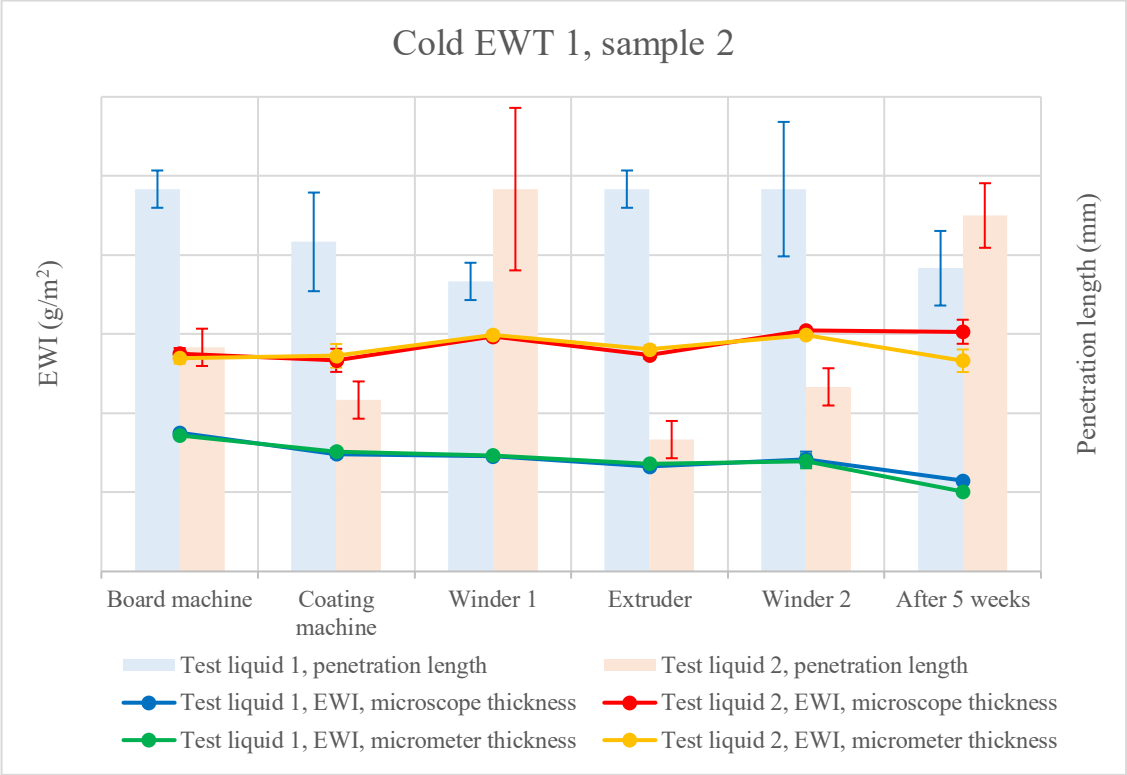

Figure 23. *Results of cold EWT 1, sample 2*

The lines in y-axis in Figure 23 are in increments of 0,2 unit. The results of EWI are curves and the results of penetration lengths are bars. The EWIs were quite close to each other despite the thickness measuring method. Measuring the thickness from the microscope pictures took a lot of time, and the estimate value of EWI which the micrometer gave, was very close to the value where the thickness was measured from the microscope picture. This means that the thicknesses could be measured by the micrometer which makes the measuring easier and there is no need for stereomicroscope.

The biggest difference between the EWIs of sample 2 was measured after 5 weeks. The thicknesses and lengths of the open edges should not change over time, but the microscope thickness slightly changed. Because the thickness was smaller, also the EWI was smaller which means that the sample was more resistant to the edge wicking after 5 weeks.

Otherwise the penetration length of test liquid 2 in millimeters was smaller than the penetration length of test liquid 1, but after the first winder and after 5 weeks it was a lot bigger. The EWI curve did not have very big increase, which indicated that the value of penetration length must have come from the individual line. According to this diagram, the EWI would have been more reliable way to measure edge wicking than the penetration length.

The results of the cold EWT 1, (uncoated, low bulk) sample 3 can be seen in Figure 24. The results are given both as EWIs and penetration lengths in millimeters.

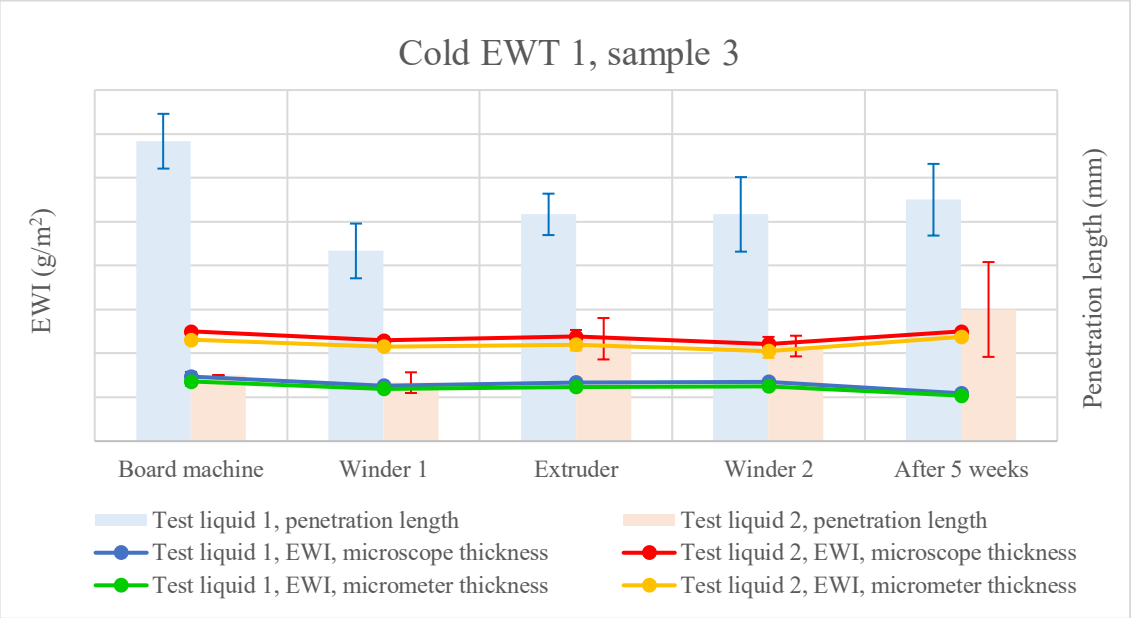

Figure 24. *Results of cold EWT 1, sample 3*

The lines in y-axis in Figure 24 are in increments of 0,2 unit. The results of EWI are curves and the results of penetration lengths are bars. The results of sample 3 had good correlation between the thickness measuring methods. The difference between the thickness measured by the micrometer and the thickness measured from the microscope picture was approximately 7 – 8 %.

The curves and bars had good correlation with each other. When the curves decreased, also the bars decreased. The standard deviations of the EWI curves were rather small. The penetration lengths of test liquid 1 had bigger standard deviations. The thickness curve of sample 3 was linear but there was a difference between the measuring methods. This made also the EWI curve linear. The result after 5 weeks was approximately the same as after the board machine.

The results of the cold EWT 1, sample 4 (low bulk) can be seen in Figure 25. The results are given both as EWIs and penetration lengths in millimeters. The results after the second winder and after 5 weeks are same, because the time between the board machine and the second winder was 5 weeks.

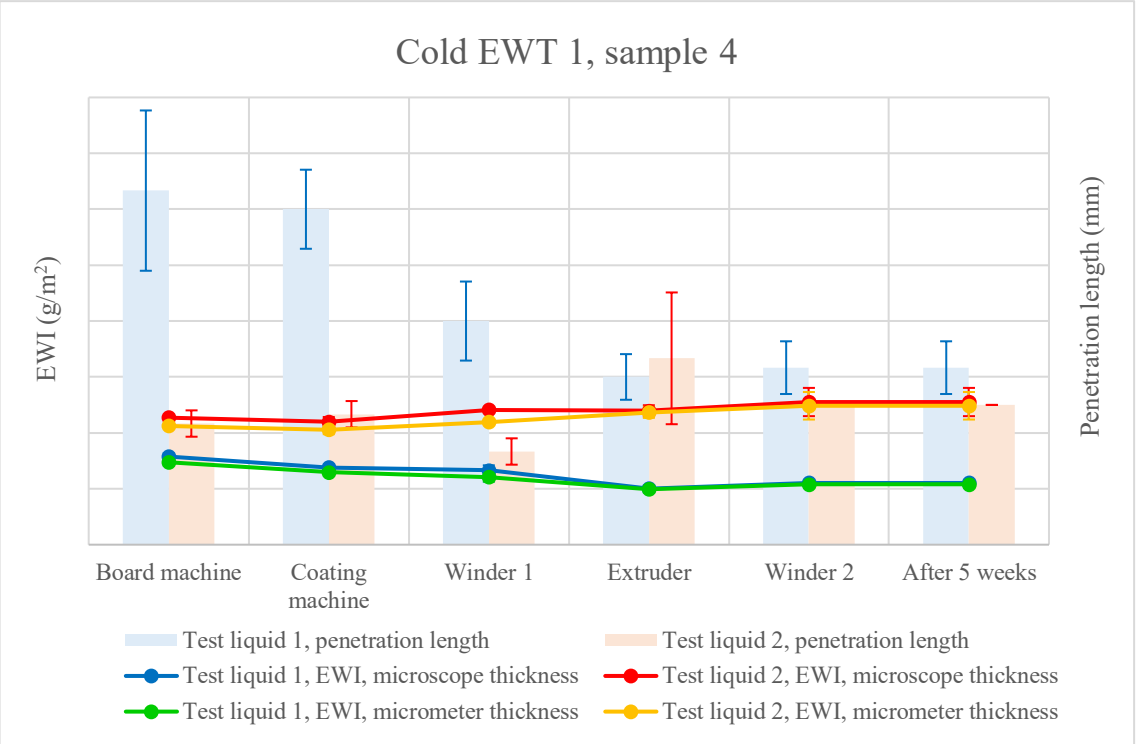

Figure 25. *Results of cold EWT 1, sample 4*

The lines in y-axis in Figure 25 are in increments of 0,2 unit. The results of EWI are curves and the results of penetration lengths are bars. The other three samples had decreasing EWI curves with the test liquid 1 between the second winder and after 5 weeks, but the sample 4 had decreasing EWI curve between the first winder and the extruder. Penetration lengths of test liquid 1 were bigger than penetration lengths of the test liquid 2. Penetration length of test liquid 2 was bigger only at the extruder, but also the standard deviation was quite big. The EWI of the test liquid 2 had linear curve during the process. The standard deviation of the EWI with the test liquid 2 was bigger after the second winder than after other machines.

Comparison of the hot and cold lamination (laminate and tape) in the cold EWT 1 was made with test liquid 2. The cold EWT 1 comparison was made with two samples, 1 and 3. The results of the visual measurements can be seen in Table 6, where the penetration lengths in millimeters are shown. Standard deviations were calculated and marked after the values in Table 6.

Table 6. *Cold EWT 1 comparison*

	Sample 1, tape (mm)	Sample 1, laminate (mm)	Sample 3, tape (mm)	Sample 3, laminate (mm)
Sampling point A	1,133 ± 0,262	3,167 ± 1,087	0,3 ± 0	0,467 ± 0,047
Sampling point B	0,533 ± 0,047	0,833 ± 0,340	0,267 ± 0,047	0,4 ± 0

The penetration values of the cold EWT 1 in millimeters were bigger when the paperboard was hot laminated (laminate) than cold laminated (tape) which can be seen in Table 6. The difference was quite big after the sampling point A, but also significant after the sampling point B. In every case the value was lower with the taped samples. The reason of differences between penetration lengths may be that in hot lamination there was no tape which meant that the glue of tape could not protect the raw edge from edge wicking. This can be seen in the microscope pictures.

The results of the cold EWT 1 can be seen in Figure 26 and Figure 27. In Figure 26 there were the EWIs measured with test liquid 1 and the thicknesses measured from the microscope pictures. The high bulk samples 1 and 2 did not differ from the low bulk samples 3 and 4, what could have happened. Test liquid 1 had bigger surface tension than test liquid 2.

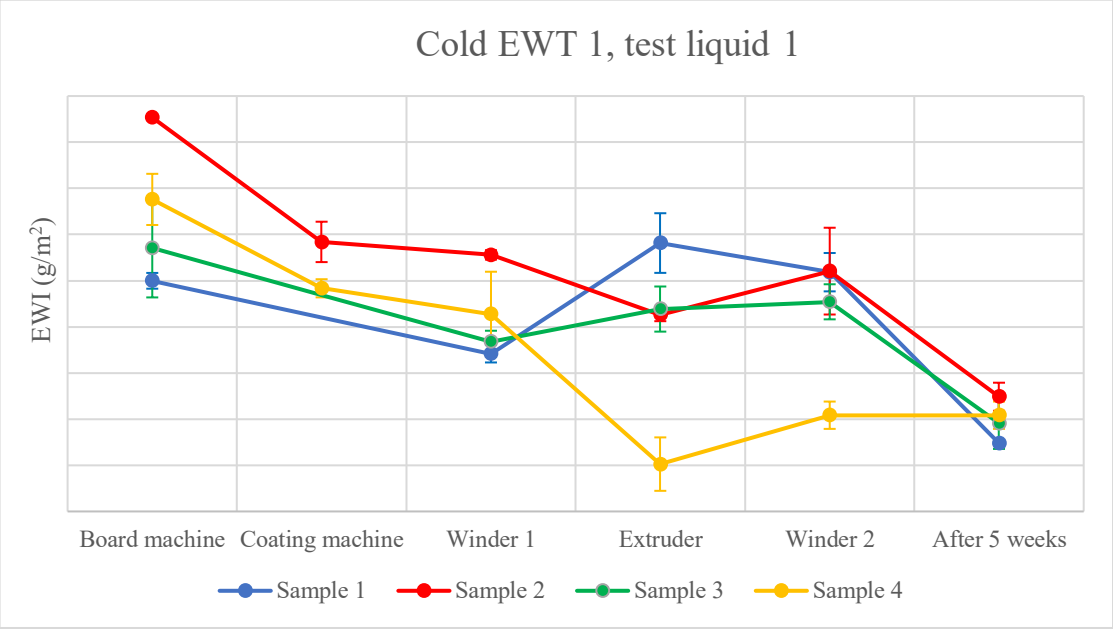

Figure 26. *Results of the cold EWT 1, test liquid 1*

The lines in y-axis in Figure 26 were in increments of 0,02 unit. The differences between samples were quite close to each other and all of them decreased from the board machine to the first winder. The coated samples 2 and 4 had lower values at the extruder than at the first winder. The uncoated samples had higher values at the extruder than at the first winder. The standard deviations at the extruder were big, which may explain the increase of the curves. The results of all the samples were smaller after 5 weeks, which was expected, because of the development of sizing. The big decrease happened to the other samples between the second winder and the time tracking sample, but the sample 4, which was extruded 5 weeks after the board machine, had the decrease already between the first winder and the extruder. This means, that the sizing development had a big effect on the EWI measured with the test liquid 1.

The test liquid 1 had bigger surface tension than the test liquid 2. The cold EWIs measured with the test liquid 2 can be seen in Figure 27.

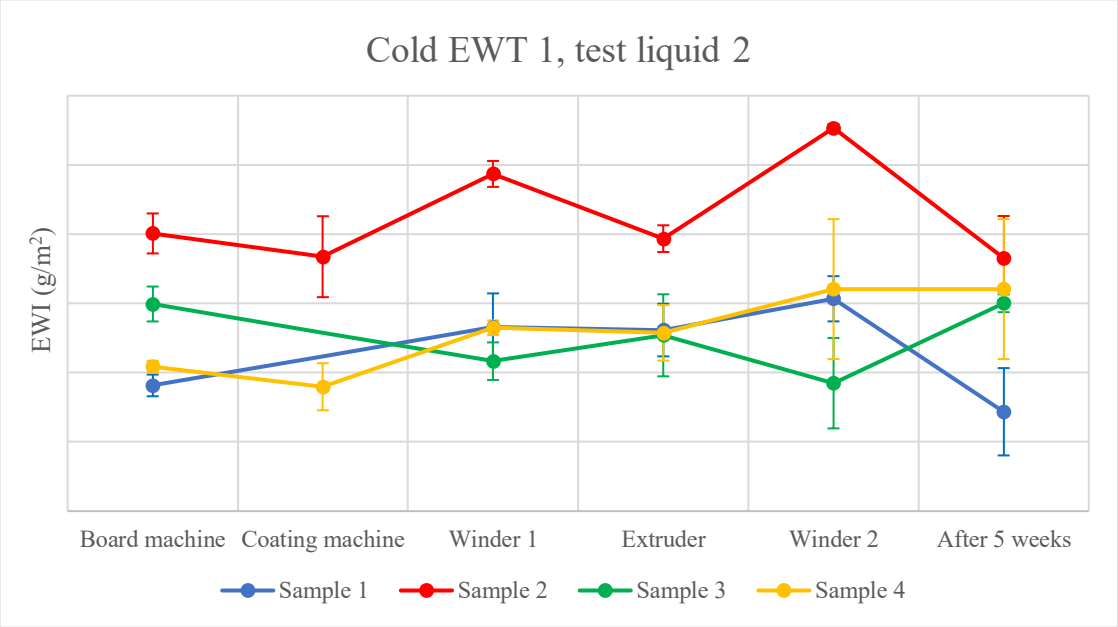

Figure 27. *Results of the cold EWT 1, test liquid 2*

The lines in y-axis in Figure 27 are in increments of 0,05 unit. The EWI values measured with the test liquid 2 were approximately on the same level after the board machine and after 5 weeks. The results varied a lot during the process, but the standard deviations were not very big.

The cutting edges of the samples looked slightly different even when the thickness had same values. The two different laboratories had different cutters. The cutter in the second laboratory cut more beautiful edge than the cutter in the first laboratory. The raw edge cut with the cutter in the first laboratory could have been slightly smaller. It is not sure if the cutter had an effect on the results, because the differences in the thicknesses were very

small. The unit of thickness in calculations was millimeters, and the differences were in the range of one hundredth of a millimeter.

6.3.5 Cold EWT 2

The cold EWT 2 results of the samples 1 and 3 in millimeters can be seen in Table 7. The results are shown as a comparison of the samples which were held in the oven and the samples which were not. Otherwise the experiments were made the same way. The oven simulated the effect of time between the measurements, the oven aged paperboard faster than room temperature. Standard deviations were calculated and marked after the values in Table 7.

Table 7. *Cold EWT 2 results, samples 1 & 3*

	Sample 1, no oven (mm)	Sample 1, oven (mm)	Sample 3, no oven (mm)	Sample 3, oven (mm)
Sampling point A	0,8 ± 0,1	0,55 ± 0,05	1,3 ± 0,4	0,4 ± 0,1
Sampling point B	0,85 ± 0,25	0,4 ± 0	0,7 ± 0	1,0 ± 0,1

The oven accelerated the sizing state, and the oven held samples had smaller values, which means that they had better resistivity against the raw edge penetration. The results of the samples 1 and 3 can be seen in Table 7 and the results of samples 2 and 4 can be seen in Table 8. Standard deviations were calculated and marked after the values in Table 8.

Table 8. *Cold EWT 2 results, samples 2 & 4*

	Sample 2, no oven (mm)	Sample 2, oven (mm)	Sample 4, no oven (mm)	Sample 4, oven (mm)
Sampling point A	0,8 ± 0,2	0,55 ± 0,05	0,4 ± 0	0,4 ± 0,1
Sampling point B	0,65 ± 0,05	0,5 ± 0	1,2 ± 0	0,55 ± 0,05
Sampling point C	0,5 ± 0	0,45 ± 0,05	0,55 ± 0,05	0,4 ± 0

The samples 2 and 4 in Table 8 had same kind of action than the samples 1 and 3 in Table 7. The results were bigger when the samples were not held in the oven. This means that the edge wicking resistivity was better, when there was more time between the paperboard manufacturing and measuring the edge wicking. The result was expected.

6.3.6 Hot EWT 1

The hot EWI %-values in the hot EWT 1 were calculated with the formula 19 and the penetration lengths were measured in millimeters. The hot EWT % values had the same kind of action between the first winder and the extruder. The values were at biggest after 5 weeks, which means that the coffee penetrated most to the paperboard at that point. The results can be seen in Figure 28.

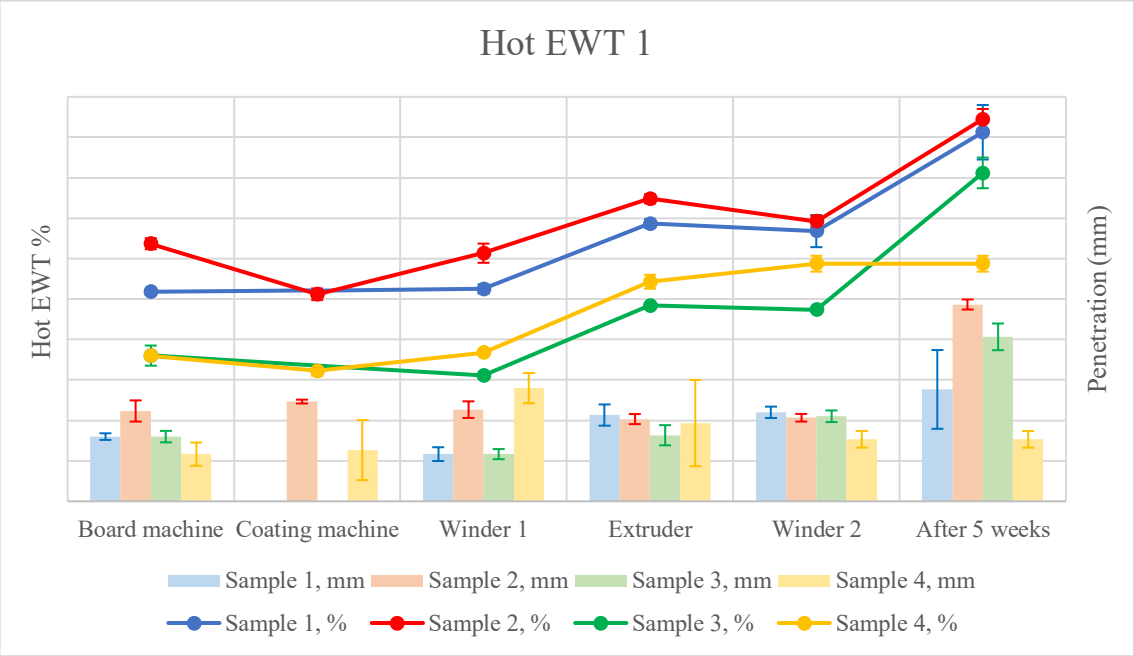

Figure 28. *Results of the hot EWT 1 in percentages and millimeters*

The lines in y-axis in Figure 28 are in increments of one unit. The results in percentages are shown as curves and the results of penetration lengths are bars. The form of the curves was same when comparing the results in different units. When the penetration length increased, also the percent value increased. Exception to this was the sample 2, whose penetration length was quite big after the coating machine. The time tracking samples had the biggest results, which means that the resistivity was worse after 5 weeks.

The sample 4 was extruded 5 weeks after the board machine, and it acted differently compared to the other samples. The time tracking samples were measured in the second laboratory, where the coffee remained hotter longer. The samples from the extruder and the second winder of the sample 4 were measured in the second laboratory and the sample from the second winder was also the time tracking sample due to the time between the board machine and the second winder. The highest value of the sample 4 was also at the last measuring point.

The hot EWT 1 results were calculated also with the formula 18 and they can be seen in Figure 29. The samples were not completely under the liquid, but the open edge length of the sample which was under the liquid was measured.

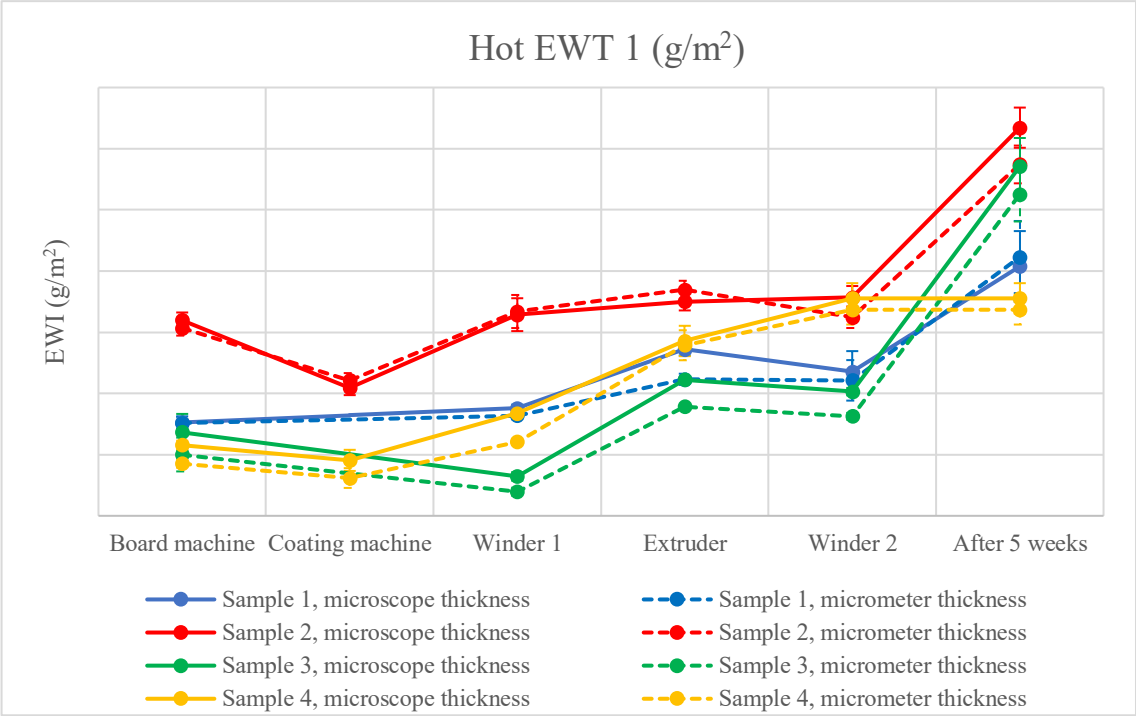

Figure 29. *Results of the hot EWT 1 in g/m²*

The lines in y-axis in Figure 29 are in increments of 0,2 unit. The behavior of all the samples was similar. The values were at biggest after 5 weeks and the correlation between the thickness measuring methods was good. The standard deviations were rather small and the curves look same than the curves in Figure 28.

The results were bigger at the last measuring point, which can be explained at least partly with the laboratory changes. The laboratory was different when measuring the samples after 5 weeks, and also the coffee was from different pouch. The cooking plate was different, and the plate used in the last measurements let the coffee stay hotter longer. The experiment was rougher to the paperboard, which may explain the fact that the resistivity got worse at the last point. The standard deviations were very small at the other points, but after 5 weeks they were significant.

High bulk samples 1 and 2 had slightly smaller values than the low bulk samples 3 and 4. Even bigger difference would have been expected, because the high bulk samples were less sized, more porous and they had more BCTMP in their middle layer. The liquid was able to penetrate better to the porous structure of paperboard. The other samples had a small decrease between the extruder and the second winder. The exception was the sample 4, which was measured at the extruder and at the second winder after 5 weeks from the board machine. It increased slightly. The value of the sample 4 after 5 weeks was smaller than the values of the other samples, but it was also at biggest at that point.

The biggest difference between the hot EWT 1 and 2 was the cutting direction of the sample. The cutting direction of the samples in the hot EWT 1 can be seen in Figure 30, where the arrow shows the machine direction.

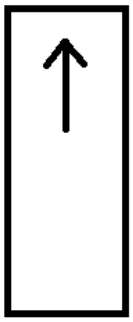

Figure 30. *Cutting direction of the sample in hot EWT 1*

The comparison of the effects of tape and laminate in the hot EWT 1 can be seen in Table 9. This comparison was made to the samples 1 and 3. Standard deviations were calculated and marked after the values in Table 9.

Table 9. *Hot EWT 1 comparison*

	Sample 1, tape (mm)	Sample 1, laminate (mm)	Sample 3, tape (mm)	Sample 3, laminate (mm)
Sampling point A	1,6 ± 0,082	1,6 ± 0,294	1,6 ± 0,141	1,7 ± 0,707
Sampling point B	1,167 ± 0,170	1,167 ± 0,047	1,167 ± 0,125	1,567 ± 0,330

Table 9 compares the penetration lengths of cold and hot lamination. The sample 1 gave exactly the same penetration results with tape and laminate. With the sample 3 the results were slightly different, but the difference was not significant. Also the standard deviations were rather small. This measurement indicates that it does not matter whether tape or laminate is used in the hot EWT 1.

The glue of tape spread over the raw edge and protected that from the liquid. Laminate did not have glue. In the cold EWT results there was a difference between the sample making methods. Taped samples got smaller values. In the hot EWT there was no difference, which may be caused by the hot coffee. The hot coffee may have dissolved the glue of tape so that it did not protect the raw edge.

6.3.7 Hot EWT 2

Hot EWT 2 was performed according to coffee test (Myllys 2007) in the chapter 5. The cutting direction of samples can be seen in Figure 31, where the arrow shows the machine direction.

Figure 31. *Cutting direction of the sample in hot EWT 2*

The cutting direction of paperboard cups is opposite, which made this experiment exceptional. The results of hot EWT 2 can be seen in Figure 32.

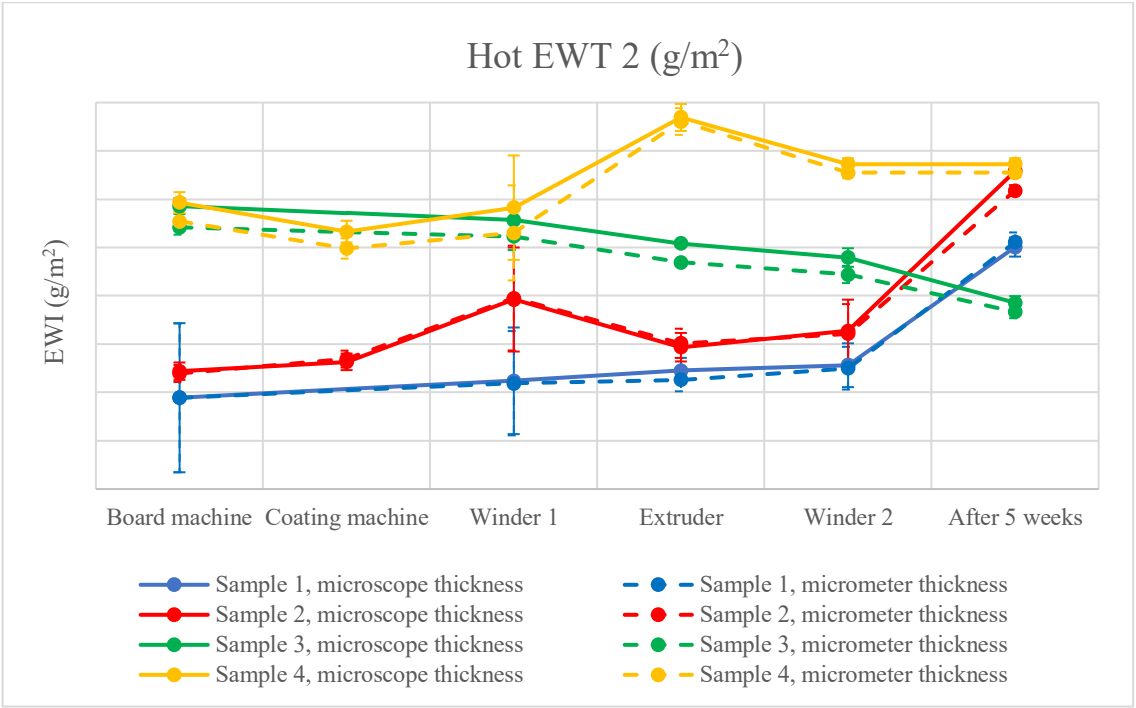

Figure 32. *Results of hot EWT 2*

The lines in y-axis in Figure 32 are in increments of one unit. The results were surprising, because the high bulk samples 1 and 2 had smaller results than the low bulk samples. This was unexcepted, because the high bulk samples were more porous and there was more air between fibers, where the liquid was able to penetrate. For comparison, the hot EWT 1 was performed so that the samples were cut both machine and cross direction to see if the cutting direction explains the behavior of the results. The results can be seen in Table 10. Standard deviations were calculated and marked after the values in Table 10. The MD

sample is cut the same way than in Figure 30 and the CD sample is cut the same way than in Figure 31.

Table 10. *Hot EWT 2 comparison*

	EWI (g/m^2), MD	EWI (g/m^2), CD
Sample A	0,257 ± 0,005	0,295 ± 0,011
Sample B	0,234 ± 0,010	0,250 ± 0,003

The results in Table 10 did not explain the behavior which can be seen in Figure 32. The results of high bulk sample A were bigger than the results of low bulk sample B. The hot EWT 2 was not a realistic test because of the cutting direction of samples. In some measurements tape was even loosen from the paperboard. The standard deviations were very small.

7. CONCLUSIONS

The porosity of paperboard has an effect on edge wicking. The more porous the paperboard is, the easier liquid penetrates into it. Porosity may be studied from the cross-section, where the areas of air and fibers may be calculated. The high bulk samples are more porous, and the liquid is able to penetrate more into high bulk samples than into low bulk samples.

The internal sizing makes the paperboard more hydrophobic. The hard sizing effects on fibers, not on air between the fibers. The sizing states are different for different sizing agents, and all sizing agents are not developed directly after the paperboard machine. Cobb test is used to measure the development of sizing. The results of Cobb test were as expected, the values decreased during the process, while the sizing develops. The effect of oven was compared using the cold EWT 2. Aging the sample in the oven gave lower results, which means that the development of sizing improved the resistivity against raw edge penetration. The experimental Cobb test is useful only if the results are compared to other similar experiments, but otherwise there is not much use for that test. The experiment is difficult to perform and there are no reference values for the paperboard cups.

Thickness can be measured in many ways. There was not a big difference between thickness measured from the microscope picture and thickness measured by the micrometer. The microscope thickness was measured visually which makes it subjective measuring method, and the method was also slower and more difficult than measuring by micrometer. What happened in the sample making was, that the glue of tape spread on the raw edge. The thickness of paperboard was difficult to determine from the microscope pictures because of the spread glue of tape. The PE-layer in the ready-made cups do not perform like that. Thickness should be measured by micrometer, which is faster and objective method. It is also accurate enough compared to thickness measured from the microscope pictures.

The cold EWT 1 had bigger values with test liquid 2, which was expected because the surface tension of test liquid 2 was smaller than the surface tension of test liquid 1. The penetration lengths were difficult to compare and measuring them was subjective. Also the standard deviations were sometimes very big, which makes the measuring method less reliable. The liquid penetration resistivity of all the sample paperboards when measured with the test liquid 1 improved during the process so, that usually the highest values were measured after board machine and the lowest values after 5 weeks. The range was much bigger with the test liquid 2, but the values measured after board machine and after 5 weeks were very close to each other.

When comparing the sample preparation methods in cold EWT 1 it could be seen, that the penetration length was bigger when the sample was hot laminated with film. The reason for that may be, that there was no glue in laminate like in tape, which is able to spread on the raw edge and protect it from liquid penetration.

The percentage values of hot EWT 1 became worse during the process and the penetration lengths increased. The coffee remained hotter longer in the second laboratory, where the time tracking samples were measured. The penetration lengths varied and especially when the coffee had dried, they were very difficult to detect and measure, so it is irrelevant to measure the penetration length. All the samples were close to each other, but the high bulk samples 1 and 2 had little worse values. This was expected, because they had more air and capillaries in their structure, where liquid was able to penetrate. The low bulk samples had more sizing, which increases the hydrophobicity of paperboard.

When comparing the sample preparation methods in hot EWT 1 it could be seen, that the results were almost the same with both hot laminating with film and cold laminating with tape. The coffee may have dissolved the glue of tape so that it did not protect the paperboard from liquid.

The hot EWT 2 did not act at all like expected, because it gave better results for the high bulk samples. Also the results crossed each other on the last point, which was strange. The high bulk samples got worse during the time and the low bulk samples got better. The comparison of sample making direction did not give the answer why the high bulk samples were better than the low bulk samples.

The behavior of paperboard in EWTs during the process was studied. The calculation of EWI was detected to be more reliable method to measure edge wicking than visual inspection of penetration length. It may be interesting to consider whether it is necessary to use two different liquids in cold EWTs. If the penetration lengths would not be measured anymore, the dye could be omitted from the test liquids. Coffee was more aggressive to the paperboard than the cold liquids. Both temperature and grease may have effect on that.

REFERENCES

Alén, R. (2007). Papermaking science and technology: Book 4, Papermaking chemistry, 2nd, totally updat version. ed, Finnish Paper Engineers' Association, Helsinki.

Ashurst, P.R. & Hargitt, R. (2010). Ingredients in soft drinks and fruit juices, Woodhead Publishing, pp. 20-59.

Atkins, T. & Escudier, M. (2013). Dictionary of Mechanical Engineering, Oxford University Press, Oxford.

Barnes, G. & Gentle, I. (2011). Interfacial science: an introduction, 2nd ed., Oxford University Press, New York, pp. 10-40.

Beattie, J.K., Djerdjev, A.M., Gray-Weale, A., Kallay, N., Lützenkirchen, J., Preočanin, T. & Selmani, A. (2014). pH and the surface tension of water, pp. 54-57.

BeMiller, J. & Whistler, R. (2009). Starch: Chemistry and Technology, 3rd ed., Academic Press, US, pp. 660-662.

Bristow, J.A. & Kolseth, P. (1986). Paper: structure and properties, Marcel Dekker, pp. 183-201.

Chattoraj, D.K. & Birdi K.S. (1984) Adsorption and the Gibbs surface excess, Plenum Pr, New York.

Chen, W., Wang, X., Wang, X., Wang, J., Tao, Q. & Zheng, Z. (2013). Lotus-like paper/paperboard packaging prepared with nano-modified overprint varnish, Applied Surface Science, Vol. 266, pp. 319-325.

Clark, J.D. (1978). Pulp technology and treatment for paper, Freeman, San Francisco, pp. 95-474.

Cotorobai, V.F., Zgura, I., Birzu, M., Frunza, S. & Frunza, L. (2016). Wicking behavior of fabrics described by simultaneous acquiring the images of the wet region and monitoring the liquid weight, Colloids and Surfaces A: Physicochemical and Engineering Aspects, Vol. 497, pp. 146-153.

Ek, M., Gellerstedt, G. & Henriksson, G. (2009). Pulp and Paper Chemistry and Technology. Vol. 2: Pulping Chemistry and Technology, 1st ed., Walter de Gruyter GmbH Co.KG.

Gooch, J.W. (2007). Encyclopedic Dictionary of Polymers, Springer, New York, pp. 283.

Halpern, M.G. (1975). Paper manufacture, Noyes, Park Ridge, NJ, pp. 126-185.

Henriksson, Å. & Gatenhalm, P. (2002). Surface properties of CTMP fibers modified with xylans, Cellulose, Vol. 9(1), pp. 55-64.

Hercules sizing tester instruction manual (2015). The Aderhold Firm.

Hintz, H. L. & Lawal, S. A. (2018). Paper: Pulping and Bleaching in: Reference Module in Materials Science and Materials Engineering.

Holik, H. (2006). Handbook of paper and board, 1st ed., Wiley-VCH, Weinheim, pp. 87-400.

Häggblom-Ahnger, U. & Komulainen, P. (2001). Paperin ja kartongin valmistus, 2nd ed., Opetushallitus, Helsinki, pp. 18-239.

Jindong, Y. & Yuanguang, H., Winding device for slitting type roll paper and winding method.

Kastinen, H. (2010). Development of a measuring device for paperboard raw edge penetration in vacuum pressure, Aalto University.

Kimpimäki, T. & Savolainen, A. (1997). Barrier dispersion coating of paper and board, pp. 208-228.

Kirwan, M.J. (2012). Handbook of paper and paperboard packaging technology, 2nd ed., Wiley-Blackwell, Iowa, West Sussex, pp. 1-383.

Kissa, E. (1996). Wetting and Wicking, Textile Research Journal, Vol. 66(10), pp. 660-668.

Kline, J.E. (1982). Paper and paperboard: manufacturing and converting fundamentals, 2nd ed., Miller Freeman, San Francisco, CA, pp. 19-211.

KnowPap KnowPap -oppimisympäristö, Prowledge Oy, web page. Available (accessed 2018, 07/25): http://www.knowpap.com/suomi/index.htm.

Kocurek, M.J. & Kouris, M. (1990). Pulp and paper manufacture: Vol. 8, Coating, converting, and specialty processes, 3rd ed., TAPPI, Atlanta (GA), pp. 76-195.

Kontaktimateriaaleja koskeva lainsäädäntö, Evira, web page. Available (accessed 2018, 07/19): https://www.evira.fi/elintarvikkeet/valmistus-ja-myynti/kontaktimateriaalit/lainsaadanto/.

Kostamo, P. (2014). Elintarvikekontaktimateriaalien turvallisuus -materiaalien valmistus Ajankohtaista elintarvikevalvonnasta ISAVI 25.3.2014, Evira.

Kuusipalo, J. (1997). PHB/V in Extrusion Coating of Paper and Paperboard, Tampere University of Technology.

Kuusipalo, J. (2008). Papermaking science and technology: Book 12, Paper and paperboard converting, 2nd, totally updated version. ed., Finnish Paper Engineers' Association, Helsinki.

Lahti, J. (2005). Dry toner-based electrophotographic printing on extrusion coated paperboard, Tampere University of Technology.

Lahti, J. & Tuominen, M. (2007) The Effects of Corona and Flame Treatment: Part. PE-LD Coated Packaging Board, Tampere University of Technology.

Larsson, C. (2008). Sizing of different pulp qualities, Karlstads Universitet.

Libby, C.E. (1962). Pulp and paper science and technology: 2, Paper, McGraw-Hill, New York, pp. 1-398.

Lohijoki, A. & Järvelä, P. (2000). Ekstruusiopäällystys ja -laminointi: Extrusion coating and laminating, Tampereen teknillinen korkeakoulu, Tampere, pp. 3-6.

Mark, A., Tryding, J., Amini, J., Edelvik, F., Fredlund, M., Glatt, E., Lai, R., Martinsson, L., Nyman, U., Rentzhog, M., Rief, S. & Wiegmann, A. (2012)b. Modeling and simulation of paperboard edge wicking Vol. 27 (Nordic Pulp and Paper Research Journal).

Mark, A., Berce, A., Sandborg, R., Edelvik, F., Glatt, E., Rief, S., Wiegmann, A., Fredlund, M., Amini, J., Rentzhog, M., Lai, R., Martinsson, L., Nyman, U. & Tryding, J. (2012)a. Multi-scale simulation of paperboard edge wicking using a fiber-resolving virtual paper model, Tappi Journal, Vol. 11(6), pp. 9.

Metlas (1981). Paperin ja kartongin käsittely, 1st ed., Metlas, Lappeenranta, pp. 5-34.

Metsä Group internal, (2018).

Moring, H. (2012). Puolikemiallisen sellu- ja kartonkitehtaan lietteen hyötykäyttö, Aalto University.

Myllys, T. (2007). Raw edge protection of packages, Tampere University of Technology.

Nemez, E. (2013). Substitution of amaranth as dye in edge wicking test, Linköping University.

Niskanen, K. (1998). Papermaking science and technology: Book 16, Paper physics, Fapet, Helsinki, pp. 89-115.

Oila, H. (2006). ASA Sizing of Mechanical Pulp, Aalto University.

Oittinen, P. & Saarelma, H. (2009). Papermaking science and technology: Book 13, Print media : principles, processes and quality, 2nd, totally updat version. ed., Paper Engineers' Association : Paperi ja puu, Helsinki, pp. 88-146.

PrintWiki On-Machine Coating, PrintWiki – the Free Encyclopedia of Print, web page. Available (accessed 2018, 07/25): http://printwiki.org/On-Machine_Coating.

Pucci, M.F., Liotier, P. & Drapier, S. (2015). Capillary wicking in a fibrous reinforcement - Orthotropic issues to determine the capillary pressure components. Vol. 77, pp. 133-141.

Ratnayake, W.M.N., Hollywood, R., O'Grady, E. & Stavric, B. (1993). Lipid content and composition of coffee brews prepared by different methods. pp. 263-269.

Rhim, J. & Kim, J. (2009). Properties of poly(lactide)-coated paperboard for the use of 1-way paper cup, Journal of Food Science, Vol. 74(2), pp. E105-E111.

Roberts, J.C. (1991). Paper chemistry, Blackie, Glasgow; New York, NY, pp. 97-131.

Roisum, D.R. (1994). The mechanics of winding, Tappi, Atlanta (GA), pp. 1-16.

Safepack Solutions Multilayer Flexible Packaging Solutions - Extrusion Coating & Lamination, Safepack Solutions, web page. Available (accessed 2018, 08/27): http://www.safepack.com/productfirst/laminates.

Salminen, P. (1988). Studies of water transport in paper during short contact times, Åbo Akademi pp. 15-82.

Samyn, P. (2013). Wetting and hydrophobic modification of cellulose surfaces for paper applications, Journal of Materials Science, Vol. 48(19), pp. 6455-6498.

Seppälä, M.J. (2000). Paperin ja kartongin jalostus, Opetushallitus, Helsinki, pp. 22-208.

Smook, G.A. (1992). Handbook for pulp & paper technologists, 2nd ed., Angus Wilde Publications, Vancouver, pp. 36-354.

Tavisto, M., Kuisma, R., Pasila, A. & Hautala, M. (2003). Wetting and wicking of fibre plant straw fractions, Industrial Crops & Products, Vol. 18(1), pp. 25-35.

Tufvesson, H. (2006). The mechanisms of edge wicking in retortable paperboard, Stockholm, Division of Fibre Technology, Department of Fibre and Polymer Technology, Royal Institute of Technology, Fiber- och polymerteknologi, KTH, Skolan för kemivetenskap (CHE).

Widiatmoko, E., Abdullah, M., Khairurrijal (2010). A Method to Measure Pore Size Distribution of Porous Materials Using Scanning Electron Microscopy Images (The Third Nanoscience and Nanotechnology Symposium 2010), Faculty of Mathematics and Natural Sciences, Institut Teknologi Bandung.

Yinusa, A.M. (2011). Factors affecting measurement of edge penetration/edge-wicking tendency.

Zumdahl, S.S. & DeCoste, D.J. (2012). Chemical principles, 7th ed., CENGAGE Learning Custom Publishing, Belmont, CA, pp. 90-136.