

LAPPEENRANTA UNIVERSITY OF TECHNOLOGY

LUT School of Engineering Science

Chemical Engineering

Chemical Process R&D

*Jenni Latva-Kokko*

**OPTIMIZATION OF A NEW SURFACE SIZING PROCESS IN THE  
PRODUCTION OF FOLDING BOXBOARD**

Examiners: Dr. Sc. (Tech.) Eeva Jernström

Prof. Tuomas Koiranen

Advisor: DI Erja Nevanperä

## **TIIVISTELMÄ**

Lappeenrannan teknillinen yliopisto  
LUT School of Engineering Science  
Kemiantekniikka

Jenni Latva-Kokko

### **Uuden pintaliimausprosessin optimointi taivekartongin valmistuksessa**

Diplomityö

2017

114 sivua, 54 kuvaa, 15 taulukkoa ja 8 liitettä

Tarkastajat: Dr. Sc. (Tech.) Eeva Jernström  
Prof. Tuomas Koiranen

Hakusanat: taivekartonki, lyhytviipymäpäällystin, pintaliimaus  
Keywords: folding boxboard, surface sizing, short dwell coater

Työssä selvitettiin kartonkikoneeseen asennettavan uuden lyhytviipymäpäällystysaseman vaikutusta taivekartongin valmistukseen. Päällystysasemaa käytetään kartongin selkäpuolen pintaliimaukseen tärkkelyksellä. Työn tavoitteena oli löytää pintaliimauksen oikea ajotapa optimoiden kartongin tärkeät ominaisuudet, kuten taivutusjäykkyys ja kartongin selkäkerroksen liitoslujuus runkokerrokseen. Tämän työn kannalta tärkeitä muuttujia ovat pintaliiman ominaisuudet, päällystysaseman sekä kuivainten ajoparametrit ja kartongin käyristyminen.

Kirjallisuusosassa tarkastellaan kartongin monikerrosrakennetta ja tekijöitä, jotka vaikuttavat kartongin laatuominaisuuksiin. Myös lyhytviipymäpäällystimen teknologiaa ja pintaliimatärkkelyksen ominaisuuksia käsitellään. Kokeellisessa osassa optimoidaan entsyymaattisen keittoprosessin ajoparametrit sekä tutkitaan kuinka tärkkelyksen viskositeetti ja kuiva-ainepitoisuus vaikuttavat pintaliimauksella saavutettaviin laatuparametreihin, kuten taivutusjäykkyyden kasvattamiseen. Uuden pintaliimauslaitteiston käyttöönottoa käsitellään lyhyesti.

Koeajojen perusteella pintaliimauksessa käytettävän tärkkelyksen määrää säädetään muuttamalla tärkkelyksen kuiva-ainepitoisuutta. Ajettaessa matalalla tärkkelysmäärällä (0.5-0.9 g/m<sup>2</sup>) tärkkelyksen kuiva-ainepitoisuus on pidettävä välillä 8-12 % ja korkealla tärkkelysmäärällä (1.4-1.7 g/m<sup>2</sup>) välillä 16-20 %. Taivutusjäykkyysindeksi parani matalalla tärkkelysmäärällä vain marginaalisesti (2 %), mutta korkealla tärkkelysmäärällä noin 5 %. Kartongin palstautumislujuus parani molemmilla tärkkelysmäärillä ajettaessa saman verran. Tärkkelysmäärällä ei voitu vaikuttaa käyryyden voimakkuuteen. Kartonki käyristyi tavoitellusti selkäpuolen kostuessa pintaliimauksen seurauksena.

## **ABSTRACT**

Lappeenranta University of Technology  
LUT School of Engineering Science  
LUT Chemical Engineering

Jenni Latva-Kokko

### **Optimization of a new surface sizing process in the production of folding boxboard**

Master's thesis

2017

114 pages, 54 figures, 15 tables, 8 appendices

Examiners: Dr. Sc. (Tech.) Eeva Jernström

Prof. Tuomas Koiranen

Keywords: folding boxboard, surface sizing, short dwell coater

The aim of this thesis was to determine the running parameters of a new surface sizing process in the production of folding boxboard. Surface size is applied on the backside of the board with short dwell coater. Native wheat starch is used as a surface sizing agent and it is degraded enzymatically before applied to the surface of the paperboard. Purpose of this work is to find the suitable process parameters for the coater and to optimize the important properties of the paperboard. Important parameters considering this work are properties of starch, the process parameters of the coater and enzymatic cooking process, and curl of the board.

In the theoretical part, the structure of folding boxboard and parameters that influence on the optimized quality properties are determined. In addition, the technology and parameters of surface sizing and enzymatic cooking process are explained. In the experimental part, the running parameters of the enzymatic cooking process are optimized and the effect of starch properties on the board quality are determined. Additionally, start-up of the surface sizing process is described shortly in the experimental part.

Based on trials, the amount of surface size can be adjusted by changing the solids content of starch. As low amount of starch (0.5-0.9 g/m<sup>2</sup>) is applied to the board solids content is kept between 8% and 12%. With the higher amount of starch (1.4-1.7 g/m<sup>2</sup>) solids content of starch is kept between 16% and 20%. With low amount of starch, bending stiffness index improved only marginally (2%) but with high starch amount the improvement was 5%. The increase in delamination strength was the same with both starch amounts. The changes in starch amount did not influence on board curl. Board curled towards back layer as desired when it was moisturized in the surface sizing process.

## **ACKNOWLEDGEMENTS**

This Master's thesis was carried out at Stora Enso Ingerois mill between December 2016 and May 2017.

First, I would like to thank my advisor Erja Nevanperä and production manager Antti Veitola for patiently instructing me during this challenging and interesting work. I want to thank the staff in the production process and laboratory for co-operation and help during trial runs. The atmosphere at Ingerois mill was welcoming and supportive, thank you all for that.

In addition, I would like to thank my examiners at Lappeenranta University of Technology, Eeva Jernström and Tuomas Koiranen, for examining my thesis.

I want to direct my special thanks to my family and friends for endless support. Especially, I want to thank my parents for always believing in me. Last but not least, I want to thank Ville for cheering me up in both rainy and sunny days.

Jenni Latva-Kokko

Inkeroinen, 19<sup>th</sup> May 2017

## LIST OF ABBREVIATIONS AND SYMBOLS

### ABBREVIATIONS

AKD	alkyl ketene dimer
ASA	alkenyl succinic anhydride
CD	cross direction
CMC	carboxymethyl cellulose
FBB	folding boxboard
IR	infrared
MD	machine direction
PPS	Parker Print Surf
PVA	polyvinyl alcohol
RAP	retrograded amylose particle
RMS	root mean square
TSI	tensile stiffness index

### SYMBOLS

b	basis weight, $\text{g/m}^2$
d	thickness, $\mu\text{m}$
E	elastic modulus, Pa
$E_{us}^w$	grammage indexed tensile stiffness index, $\text{kNm/g}$
F	force, N
G	PPS roughness, $\mu\text{m}$
$h_i$	the distance of mid plane of the layer from middle plane of the whole sheet
K	curl, $1/\text{m}$
k	geometric constant
l	length, m
P&J	Pulsey & Josey hardness
Q	airflow rate, $\text{ml/min}$
$S_b$	bending stiffness, $\text{mNm}$
$S_{b,s}$	bending stiffness index, $\text{Nm}^7/\text{kg}^3$
$S_{DIN}$	bending stiffness, $\text{mNm}$
T	Peeling number/value, $\text{g/cm}$
v	ultrasonic velocity, $\text{km/s}$
w	basis weight, $\text{g/m}^2$
z	middle plane
$z_0$	neutral plane
$\rho$	density, $\text{kg/m}^3$
$\mu$	viscosity, $\text{mPas}$
$\varepsilon$	strain, % of cross sectional length of the sample
$\sigma$	stress, GPa
$\beta$	hygroexpansivity, %

## TABLE OF CONTENTS

1	INTRODUCTION .....	8
1.1	Objectives and scoping of the thesis .....	8
2	MULTILAYER STRUCTURE OF FOLDING BOXBOARD .....	9
3	IMPORTANT PROPERTIES OF PAPERBOARD .....	11
3.1	Basis weight, thickness, density and bulk .....	13
3.1	Elastic modulus .....	13
3.2	Bending stiffness .....	18
3.3	Surface roughness .....	24
3.4	Strength properties .....	25
3.5	Dusting .....	30
3.6	Curl .....	30
4	SURFACE SIZING .....	33
4.1	Pond size press .....	34
4.2	Film size press .....	34
4.3	Application techniques of coating or size .....	37
4.4	Drying .....	40
4.4	The effect of web properties .....	45
4.5	Surface sizing agents .....	46
4.6	Surface size additives .....	51
5	MODIFICATION OF NATIVE STARCH .....	53
5.1	Thermochemical oxidation .....	54
5.2	Enzymatic modification .....	54
	EXPERIMENTAL PART .....	56
6	INGEROIS BOARD MILL .....	57
6.1	Production process from the drying section to the reeling .....	57
6.2	UMV INVO Coater .....	58
6.3	Enzymatic cooking process .....	60
7	START-UP .....	62
8	THE ENZYMATIC COOKING .....	63
8.1	Pre-trials .....	63
8.2	Effect of the enzyme dose and converting time .....	65
8.3	The change of surface size starch .....	71
8.4	Conclusions .....	73
9	EFFECT ON BOARD QUALITY .....	73
9.1	Solids content .....	74
9.2	Viscosity .....	88

9.4	Conclusions.....	93
10	DRYING AND CURL CONTROL .....	97
10.1	Drying requirement of last drying group .....	98
10.2	Curl control with the seventh drying group .....	102
10.3	Moisturizing device .....	103
10.4	Conclusions.....	105
11	FINAL CONCLUSIONS .....	107
	REFERENCES .....	110

## 1 INTRODUCTION

Important development areas in paperboard production are minimizing the basis weight and improving the converting properties. Almost all paperboard properties are influenced when basis weight is altered. Especially important properties are bending stiffness and the strength values that are both decreased when the basis weight is lowered. Paperboard packages do not achieve the wanted rigid and strong structure without proper bending stiffness. Compromises between different properties have to be done to achieve proper values in every important category. (VTT, 2015; Nevalainen, 2012)

### 1.1 Objectives and scoping of the thesis

Good bending stiffness of the board can be achieved by high bulk middle layer and strong top and back layers. The desired structure is described as an I-beam structure. There are different ways to improve the wanted structure such as adding bulkier mass to the middle layer and increase basis weight of the product. Typically, customer prefers lighter product and objective of producer are low raw material costs. For these reasons, increase of basis weight is not the optimal option when bending stiffness has to be increased.

One way to achieve stronger outer layers without significant increase in basis weight is surface sizing. Surface sizing of the board increases the elastic modulus of the outer layers of the board. Surface sizing does not cause loss of bulk in the middle layer and bending stiffness of the board increases. Surface sizing influence also on the curl of paperboard, which can cause problems in finishing and converting processes. Ingerois mill produces folding boxboard that is coated only from one side. Asymmetric coating curl the board more to the coated side, which is not desirable. Curl is controlled with moisturizing unit and with asymmetric drying of top and backside of the web. When uncoated backside of the board is surface sized the moisturizing unit is not used.

Wheat starch that is used as a surface sizing agent is degraded enzymatically. Determination of the running parameters of the enzymatic cooking process was part of the experimental part. The most important process parameters of starch are solids content and viscosity. The aim of the cooking trials was to optimize these

properties to achieve good runnability of surface size at the coating station and control the amount of surface size on the board.

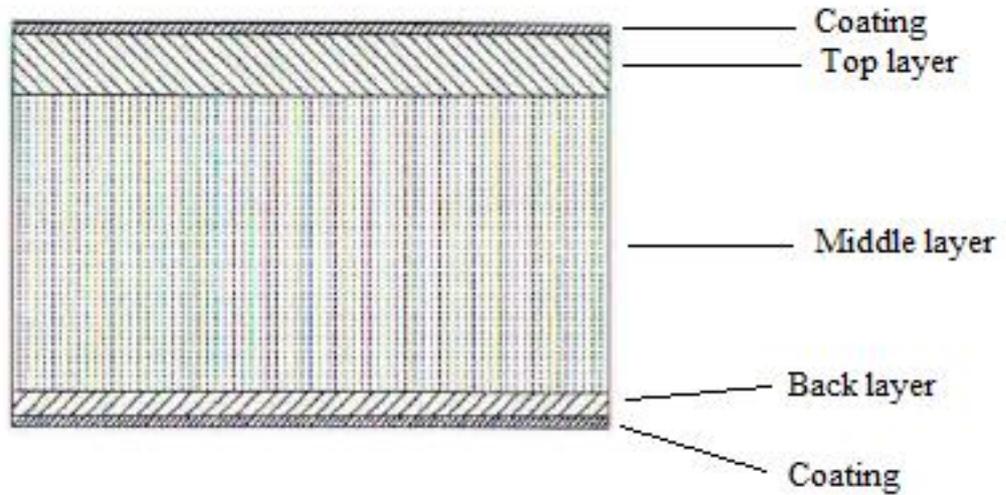
The aim of these trials was to determine the running parameters of a new surface sizing process. The parameters are solids content and viscosity of starch, and enzyme dose and converting time of the enzymatic cooking process. The start-up parameters and plan of the new process are described in the experimental part. Purpose of determining the right running parameters was to achieve higher bending stiffness, surface strength of the backside and delamination strength between middle and back layer of the board. In addition, drying parameters of the surface sized board are determined.

This thesis was limited to consider only the manufacturing process from intermediate calender to the reeling. The wet end parameters such as beating are considered shortly in the literary part of the work.

## **2 MULTILAYER STRUCTURE OF FOLDING BOXBOARD**

Paperboards are typically multilayer structured products (Figure 1) in which every layer has its own function. Paperboard is often used as a packaging material so it has to have good stiffness and strength properties but also good printability properties. Paperboards are divided into three groups based on their end use: cartonboards, containerboards and graphical boards. Folding boxboard is cartonboard that can be used for packaging of food, medicine, cosmetics, alcohol and cigarettes. (Hägglom-Ahnger, Komulainen, 2001; Kiviranta, 2000)

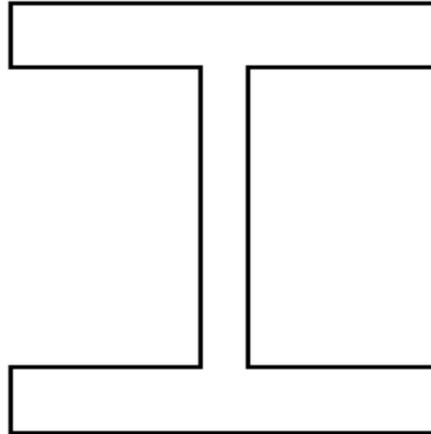
Layers of the paperboard differ from each other due to their different functions. As can be seen from the Figure 1 the thickest layer of the paperboard is its middle layer and it forms 50-80% of the basis weight of the paperboard. High bulk, mechanical pulp is used as a raw material in middle layer to achieve demanded thickness of the paperboard. Mechanical pulp is produced typically in two ways: Grinding and refining. Regardless of the process technology the demands for the mechanical pulp are the same. Desired properties of mechanical pulp are high lignin content, bulk, stiffness, good opacity and smoothness. (Kiviranta, 2000; Tillmann; VTT, 2015; Hägglom-Ahnger et al., 2001)



**Figure 1** Paperboard has multilayer structure and every layer has their own function. (Kiviranta, 2000)

The top and back layers of the board are consisted of chemical pulp because the long fibers ensure good strength properties and even paperboard surface. Unbleached chemical pulp has better strength properties than the bleached one and it is typically used for the manufacture of liner. Bleached chemical pulp is used for the top and back layers of the better quality paperboards. Basis weight of top layer is 40 to 60 g/m<sup>2</sup> and basis weight of back layer is half of that (20-30 g/m<sup>2</sup>). (Kiviranta, 2000; Tillmann; VTT, 2015; Häggblom-Ahnger et al., 2001)

The strongest materials are chosen to the top and back layer to achieve high elastic modulus. It is important to have as strong top and back layers as possible because outer layers of the board bear the biggest part of the stress when it is bent. This can be seen in the Figure 2 where the structure of desired folding boxboard is portrayed as an I-beam structure. The aim is to keep the outer layers far from each other by choosing high bulk pulp to the middle layer. (Häggblom-Ahnger et al., 2001)



**Figure 2** The desired I-beam structure of board includes strong top and back layers and high bulk middle layer. Figure is cross sectional presentation of the desired board structure.

Cartonboards are usually coated to achieve required surface properties. Coating can be applied for one side or both sides of the board. The amount of coating layers varies usually from one to three. There are two types of coating materials for different purposes: barrier- and mineral coating materials. Barrier coating gives paperboard impermeability properties and mineral coating improves the printing surface of paperboard. (Kiviranta, 2000)

### **3 IMPORTANT PROPERTIES OF PAPERBOARD**

The most important properties for folding boxboard are listed in Table I. Also the reason for importance of the property, measuring method used at Ingerois mill and its accuracy are included in to the Table I. Properties are described more detailed in the following sections. (Hägglom-Ahnger et al., 2001)

**Table I** The most important properties of folding boxboard, relevance to the quality, measurement method of property and method accuracy.

Measured quantity	Relevance to the quality	Measurement method	Measurement method accuracy
Elastic modulus	<ul style="list-style-type: none"> <li>• Big impact on bending stiffness of the board.</li> <li>• Strong top and back layer.</li> </ul>	<ul style="list-style-type: none"> <li>• The ultrasonic measurement</li> </ul>	<ul style="list-style-type: none"> <li>• 0,01 MPa</li> </ul>
Bending stiffness	<ul style="list-style-type: none"> <li>• Strength of the package.</li> <li>• Runnability in packaging machine</li> <li>• Folding and stapling in printing presses</li> </ul>	<ul style="list-style-type: none"> <li>• Taber stiffness (ISO 2493)</li> <li>• L&amp;W Bending Tester (DIN 53121)</li> </ul>	<ul style="list-style-type: none"> <li>• 0,1 mNm</li> <li>• 1 mN</li> </ul>
Surface strength	<ul style="list-style-type: none"> <li>• To avoid dusting</li> <li>• To avoid the sticking of the fibers in the offset printing.</li> </ul>	<ul style="list-style-type: none"> <li>• IGT (modified from ISO 3783)</li> </ul>	<ul style="list-style-type: none"> <li>• 0,01 m/s</li> </ul>
Delamination strength	<ul style="list-style-type: none"> <li>• Offset printing</li> <li>• To avoid delamination of plies.</li> </ul>	<ul style="list-style-type: none"> <li>• Scott Bond (TAPPI 569)</li> <li>• L&amp;W ZD Tensile Tester (modified from SCAN-P 80)</li> <li>• Peeling test at Ingerois mill</li> </ul>	<ul style="list-style-type: none"> <li>• 1 J/m<sup>2</sup></li> <li>• 1 kPa</li> <li>• 1 g/cm</li> </ul>
Curl	<ul style="list-style-type: none"> <li>• To avoid problems in finishing processes</li> <li>• Packaging machine runnability</li> </ul>	<ul style="list-style-type: none"> <li>• “Measurement box” at Ingerois mill</li> </ul>	<ul style="list-style-type: none"> <li>• 1 mm.</li> </ul>
Thickness	<ul style="list-style-type: none"> <li>• Important for bulky structure.</li> </ul>	<ul style="list-style-type: none"> <li>• L&amp;W Micrometer (ISO 536)</li> </ul>	<ul style="list-style-type: none"> <li>• 1 μm</li> </ul>
Bulk	<ul style="list-style-type: none"> <li>• Big impact on bending stiffness of the board.</li> <li>• To keep the distance between outer layers of the board.</li> </ul>		
Smoothness/ Roughness	<ul style="list-style-type: none"> <li>• To avoid uneven printing result.</li> </ul>	<ul style="list-style-type: none"> <li>• Bendtsen (modified from ISO 8791-2)</li> <li>• PPS (ISO 8791-4)</li> </ul>	<ul style="list-style-type: none"> <li>• 1 ml/min</li> <li>• 0,01 μm</li> </ul>

### **3.1 Basis weight, thickness, density and bulk**

Basis weight of paperboard is the weight of the paperboard per square meter ( $\text{g/m}^2$ ). Normally, basis weight of the paperboard varies between  $100 \text{ g/m}^2$  and  $600 \text{ g/m}^2$ . Increase of basis weight improves many properties of paperboard but it is not the ideal parameter for the purpose. Both customer and producer typically prefer lighter paperboard. The raw material costs increases as the basis weight increases. Because refining process can be adjusted quite well to match up with the properties of the paperboard, more important than the level of properties is the evenness of them. Especially basis weight that influences many other properties should not have any rapid changes. Variation in basis weight causes problems in machine runnability and control. (Hägglom-Ahnger et al., 2001)

Thickness of paperboard can be defined in two different ways. Sheet thickness is calculated based on the thickness of one paperboard sheet and stack thickness is calculated based on several sheets that are piled in stack. Selling prices of paperboard derives usually from the thickness of the board. This is due to the fact that bending stiffness is dependent on thickness to the power of three. (Hägglom-Ahnger et al., 2001; VTT, 2015)

Bulk ( $\text{cm}^3/\text{g}$ ) and density ( $\text{kg/m}^3$ ) are properties that are depended on each other. Bulk is the inverse value of apparent density which is calculated by dividing basis weight with the stack thickness of the paperboard. Density of paperboard is low because high bulk is desired property of paperboard. Refining of pulp increases density of pulp but decreases the bulk. By increasing the bulk of paperboard many other properties like opacity, stiffness and tear strength improve also. At the same time, smoothness of paperboard surface and tensile strength are decreasing. Calendaring improves smoothness but at the same time bulk decreases. Compromises between properties are unavoidable in paperboard making. Density of paperboard is influenced the most by the density of the raw materials and the porosity of paperboard. For example, density of fillers and coating pigments are about  $2600 \text{ kg/m}^3$  (dry fiber wall  $1500 \text{ kg/m}^3$ ). Increase of coating or fillers increase the density of paperboard. (Hägglom-Ahnger et al., 2001; VTT, 2015)

### **3.1 Elastic modulus**

Elastic modulus is a strength property that can be used as an indicator of other strength properties and dimensional stability. Elastic modulus measures how

much force needs to be applied to the material to produce deformation in it. Elastic modulus is a basic quantity in standard engineering mechanics. Directly measured elastic modulus in a certain strain is determined with following Equation:

$$E = \frac{F}{Wd\epsilon} \quad (1)$$

where E = elastic modulus [GPa], F = force [N], W = width of the sample [m], d = thickness of the board [m] and  $\epsilon$  = strain [% of the sample cross sectional length]. (Niskanen, Kärenlampi, 1998)

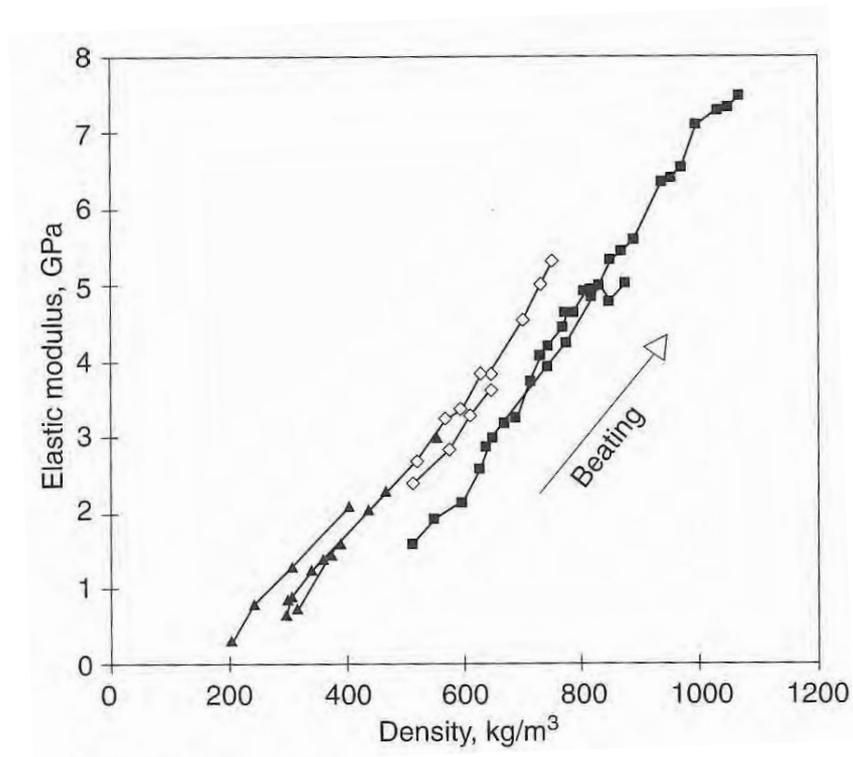
Elastic modulus is used for controlling the web tension and bending stiffness. When elastic modulus increases bending stiffness (described better in next chapter) increases. Elastic modulus is traditionally defined with stress-strain curve or load-elongation curve, and more specifically as the slope of that curve. Elastic modulus is then described as follows:

$$E = \frac{d\sigma}{d\epsilon}, \sigma \rightarrow 0 \quad (2)$$

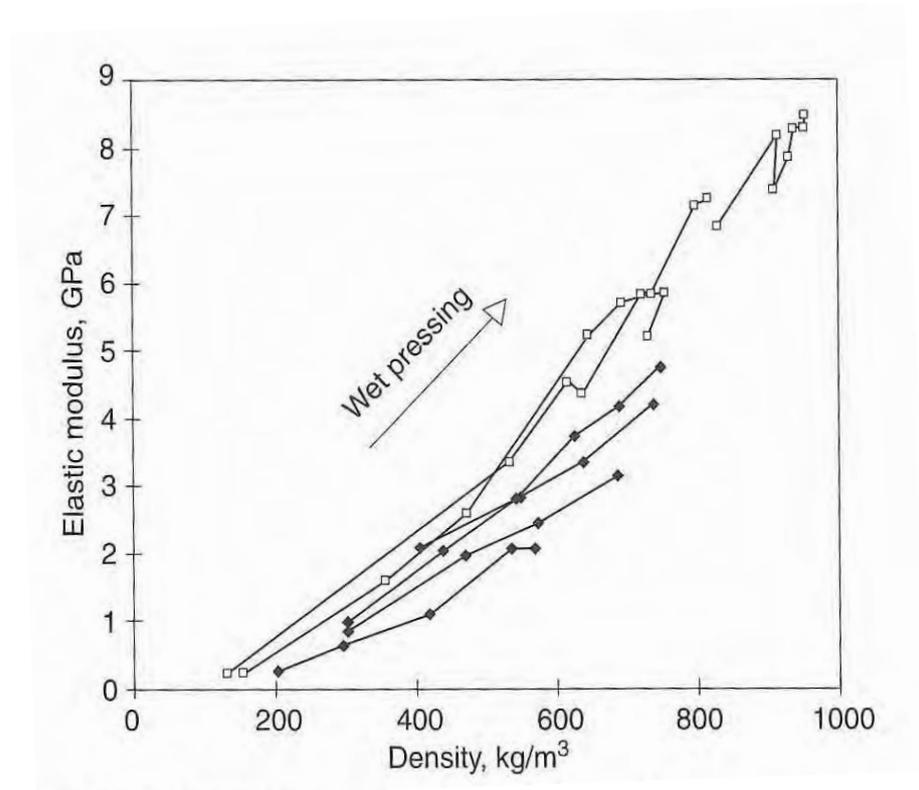
where  $\sigma$  = stress applied over the cross sectional area of the sample [GPa] and  $\epsilon$  = strain caused by stress [% of the sample cross sectional length]. When elongation is large Equation 2 results only the tangent modulus that is smaller than the actual elastic modulus. Elastic modulus is important to define in three different dimensions: Machine direction, cross-machine direction and thickness direction. Elastic modulus is measured by stretching the sample in the tensile mode when in-plane directions are concerned. In z-direction compression mode is more useful because the fiber network acts differently in z-direction than in in-plane directions. (Muhonen, 2013; Niskanen et al., 1998)

When elastic modulus is evaluated based on the load-elongation curve it has to be done carefully. Range of the measurement should not be too long to underestimate the modulus but long enough to achieve good accuracy. Typically, linearity of the curve occurs only in small ranges. Elastic modulus is defined as the maximum slope of the curve if linearity occurs with delay after the start. (Niskanen et al., 1998)

The relation between the density of the measured component and elastic modulus in different wood species and pulp type is described in Figures 3 and 4. Density of the pulp is dependent on beating, wet pressing and pulp mixture. Based on the Figures 3 and 4 can be said that elastic modulus of handsheets increases when beating or wet pressing increases. The differences in wood species do not influence the slope of elastic modulus significantly. The wet pressing level or beating level is fixed for every line. (Niskanen et al., 1998)



**Figure 3** Elastic modulus of paper (handsheets) as a function of density in a varied beating level. Every line in the Figure presents data for one wood specie, pulp type and fixed wet pressing level. (Niskanen et al., 1998)



**Figure 4** Elastic modulus of paper (handsheets) as a function of density in a varied level of wet pressing. Every line in the Figure presents data for one wood specie, pulp type and fixed wet pressing level. (Niskanen et al., 1998)

Drying stress influences the elastic modulus of the paperboard. When shrinkage is limited during drying the elastic modulus is increased. This is based on the behavior of the fibers. Fibers shrink when they are dried, especially in the thickness direction. In the fiber network shrinking of one fiber influences all of the fibers attached to it. Microcompression occurs at the bond and bends free fiber segments. The bending can be avoided by mechanically preventing the sheet shrinkage. Mechanical stretching prevents bonding while the free fiber segments and microcompression are straightened. This theory of fiber shrinkage is Page's shrinkage theory. The elastic modulus is higher in MD due to the orientation of the fibers but also because the shrinkage is prevented in drying section by web tension. (Niskanen et al., 1998; VTT, 2015)

The specific elastic modulus can be measured with ultrasonic measuring technology. The specific elastic modulus is independent on the thickness of the sample because it is divided by the density:

$$\frac{E}{\rho} = \frac{F}{Wb\varepsilon} \quad (3)$$

where  $b$  = grammage [ $\text{g}/\text{m}^2$ ].

The principle of measurement is to place transmitter and receiver on the paperboard 100 mm apart from each other and send an ultrasonic impulse (frequency 60 kHz) along the paperboard. The time that impulse need for propagating to the receiver is detected and mean velocity is calculated. The ultrasonic velocity is related to tensile stiffness index (TSI) as following Equation expresses:

$$E_{us}^w = c \times v^2 \quad (4)$$

where  $E_{us}^w$  = grammage indexed tensile stiffness index [ $\text{kNm}/\text{g}$ ],  $c$  = dimensionless constant close to unity,  $v$  = ultrasonic velocity [ $\text{km}/\text{s}$ ]. Tensile stiffness index is related to elastic modulus as follows:

$$E_{us} = \rho \times E_{us}^w \quad (5)$$

where  $E_{us}$  = modulus of elasticity [ $\text{MPa}$ ],  $\rho$  = density [ $\text{kg}/\text{m}^3$ ]. (Lindblad, Fürst, 2001; Niskanen et al., 1998)

Elastic modulus of the board is increased if the coating layer has higher elastic modulus than the board. In that case, coating can be considered as its own layer and bending stiffness is increased. Elastic modulus of coating is dependent on the shape of the pigment particles. Binders of the coating has significant effect on the elastic modulus. Hard binders such as latexes and starch give the strongest effect due to their high glass transition temperature. Negative effects of binders are poor brightness and print mottle. Too high elastic modulus of coating can cause cracking of the coating layer. (Kajanto, 1998)

Surface sizing increases the elastic modulus of the layer and bending stiffness of the board. Thickness of the board does not increase significantly in surface sizing. The biggest improvement in bending stiffness is achieved when surface size stays on the surface of the board layer. The sizing equipment influence significantly to

the size penetration. For example, with short dwell application technique size stays at the surface of the board and with size press the size penetrates much more into the board structure. (Kajanto, 1998)

### 3.2 Bending stiffness

Bending stiffness is one of the most important properties of paperboard. Without proper bending stiffness runnability of the board machine, printing presses and converting machine suffers. Good bending stiffness is especially required in finishing and converting processes of paperboard. Rigid package cannot be achieved if bending stiffness of the board is low. Generally, stiffness of the paperboard is rarely too high. Basis weight influence significantly on bending stiffness but usually it is not possible to increase basis weight to raise bending stiffness. Instead, basis weight is lowered and bending stiffness is tried to be kept constant. (Kajanto, 1998)

Bending stiffness is dependent on the elastic modulus and the effective thickness of the paperboard as can be seen in Equation 6:

$$S_b = \frac{Ed^3}{12} \quad (6)$$

where  $S_b$  = bending stiffness [Nm],  $E$  = elastic modulus [Pa] and  $d$  = effective thickness [m] that does not include the contribution from surface roughness. Bending stiffness of different paperboard grades can be compared by calculating bending stiffness index ( $S_{b,s}$ ):

$$S_{b,s} = \frac{S_b}{b^3} \quad (7)$$

where  $b$  = grammage [ $g/m^2$ ] and  $S_b$  = bending stiffness [Nm].

Bending stiffness is determined in two dimensions: cross- (CD) and machine direction (MD). Usually CD stiffness causes more problems than 2-4 times higher MD stiffness. This is due to the fiber orientation and the elastic modulus that is influenced by CD drying shrinkage. (Kajanto, 1998; Markström) If the “overall” bending stiffness of the board is determined geometrical bending stiffness is calculated with Equation 8:

$$S_{b,geom.} = \sqrt{S_{b,CD} \times S_{b,MD}} \quad (8)$$

where  $S_{b,geom.}$  = geometrical bending stiffness [Nm],  $S_{b,CD}$  = cross directional bending stiffness [Nm] and  $S_{b,MD}$  = machine directional bending stiffness [Nm].

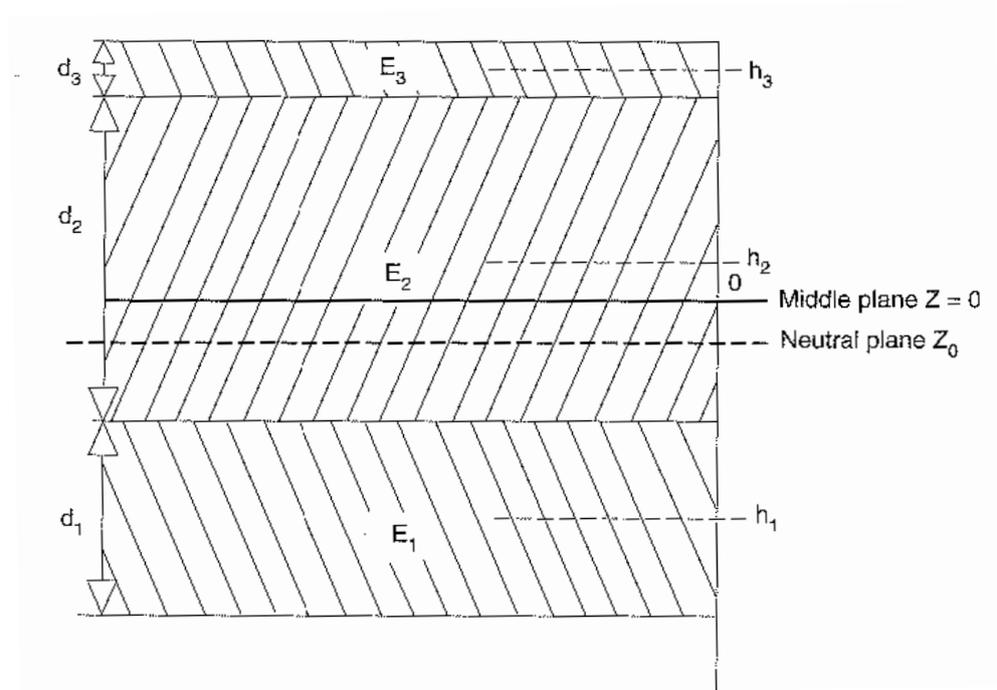
As mentioned earlier in this chapter, basis weight of the board influences on bending stiffness. There are few possibilities to improve bending stiffness without increasing basis weight. First way is to increase the bulk and thickness of the paperboard by adding bulkier pulp, like mechanical pulp or broke, to the middle layer. Other way is to increase elastic modulus of the board surface layers. Multilayer structure of the paperboard makes it easier to improve bending stiffness but still maintain low basis weight. (Kajanto, 1998)

To define the bending stiffness of multilayer sheets by using Equation 9 every layer is assumed to be homogeneous:

$$S_{b,i} = E_i \left[ \frac{d_i^3}{12} + d_i \times (h_i - z_0)^2 \right] \quad (9)$$

where  $S_{b,i}$  = bending stiffness of the layer,  $h_i - z_0$  = the distance between “i”th ( $h_i$ ) layer and the neutral plane ( $z_0$ ) [m],  $E_i$  = elastic modulus of the layer [Pa] and  $d_i$  = thickness of the layer [m]. If the value of the term  $h_i - z_0$  is high, it means that the contribution of that layer is high. In Figure 5 multilayer sheet structure and its parts are presented. By summing the contribution of every layer the bending stiffness of the whole sheet can be calculated with Equation 10:

$$S_b = \sum S_{b,i} \quad (10)$$



**Figure 5** The structure of a multilayer sheet where  $h_i$  is the distance of layer's mid plane from middle plane of the whole sheet,  $d_i$  is the layer thickness,  $z_0$  is the distance between the neutral plane and the middle plane. (Kajanto, 1998)

Coating and surface sizing increase the amount of bonded material on board surface that improves the bending stiffness. Bending stiffness is increased by coating if elastic modulus of coating is higher than elastic modulus of board. Coating layer can be then considered as its own layer. The high thickness of coating layer does not cause any excess benefit on bending stiffness. Because of the high density of coating, uncoated paper grades have usually higher bending stiffness index than coated ones. The effect of pigment to the bending stiffness have been researched in earlier studies. As a result, the coat weight, that was required to achieve the same bending stiffness as the base paper, was at least  $10 \text{ g/m}^2$ . The paper was coated only from one side. Explanation for the low bending stiffness with small coating amounts could be that the water of the coating is influencing the paper structure in a way that the bending stiffness of the paper is lowered. The required coating amount would have been lower if paper had been coated on both sides of the paper. (Kajanto, 1998; Kajanto, 1995)

Bending stiffness of the paperboard product is influenced by pulp properties. Every pulp has its own characteristic bending stiffness that influences on the properties of the final product. Bending stiffness of the final product cannot be

evaluated only based on the potential bending stiffness of the pulps because the conditions of the board machine and converting treatments lower the value. Bulk of the pulp has the biggest influence on bending stiffness. Bulky paperboard and high bending stiffness are achieved with stiff fibers and bonds. By comparing chemical and mechanical pulps with each other chemical pulp has better bonding ability but mechanical pulp has stiffer fibers. Stiff fibers lead to higher thickness of the paperboard. Based on the Equation 6 thickness influences bending stiffness more than elastic modulus that leads to a conclusion that mechanical pulp has a higher potential bending stiffness than chemical pulp. When paperboard is calendared to a constant thickness higher bonding ability and elastic modulus of chemical pulp provide higher bending stiffness than mechanical pulp. (Kajanto, 1998)

Fibers of the pulp can be modified by beating. Beating increases both bonding ability and flexibility of fibers. Elastic modulus of board increases but thickness of the board decreases due to the increase of fiber flexibility. The sum of these effects may cause negative effect on bending stiffness. In addition to bending stiffness, other criteria have to be taken into consideration to achieve the wanted qualities for the paperboard. Due to that, pulp cannot be chosen and modified only based on the bending stiffness. (Kajanto, 1998)

Z-directional variation of the board structure influences on bending stiffness. The forming section of the board machine influences Z-directional variation and especially the fines and filler distribution. Anisotropy or MD/CD ratio of bending stiffness changes when fiber orientation is changed. MD bending stiffness can be increased by increasing the fiber orientation anisotropy. At the same time increased fiber orientation anisotropy and drying shrinkage decrease CD bending stiffness and especially the bending stiffness of the edges of the web. (Kajanto, 1998)

At Ingerois Mill, bending stiffness is measured in two ways: Taber stiffness and by calculating bending stiffness based on the TSI-test results from L&W Autoline measurement unit. Calculation is performed with following Equation:

$$S_{b,MD/CD} = TSI_{MD/CD} \times \frac{d^2}{10^8} \times w \quad (11)$$

where  $S_{b,MD/CD}$  = MD or CD bending stiffness [Nm],  $TSI_{MD/CD}$  = MD or CD TSI-measurement value [Nm/g],  $d$  = thickness [m] and  $w$  = basis weight [ $g/m^2$ ].

Bending stiffness of paperboard can be determined in laboratory with two different principles: beam bending principle and the resonance stiffness principle. Taber stiffness tester (Figure 6) is a common way to test bending stiffness (used at Ingerois mill) and it utilizes the beam bending principle. The paperboard sheet is cut to 3.8 cm wide and 5 cm long sample. The top end of sample is placed into the jaws of clamping block and the lower end is placed between two rollers that are attached to the rotating disc. When sample is in the right place power is applied to lower end of the sample and the force results torque to the sample. Bending stiffness is indicated from the angular deflection in the pendulum caused by the torque. (Testing Machines, Inc.)



**Figure 6** Taber tester measures bending stiffness of paperboard by measuring the force needed for bending the lower end of the specimen. (Testing Machines, Inc.)

The resonance test is based on the assumption that there is a correlation between the resonance length of a vibrating sample and the bending stiffness of that

sample. The sample strip is placed between clamps from one end and the clamp is vibrated at a specific frequency. The resonance length of the strip caused from vibration is measured and the bending stiffness is calculated based on that and the basis weight of the material with the Equation 12:

$$S^b = \frac{4\pi^2}{k^4} l^4 w f^2 \times 10^{-12} \quad (12)$$

where  $S^b$  = bending stiffness [Nm],  $l$  = resonance length of a test strip [m],  $w$  = grammage [ $\text{g}/\text{m}^2$ ],  $f$  = frequency of vibration [Hz],  $k$  = dimensional constant (equal to 1.875 to fundamental frequency of oscillation). (TAPPI, 1996)

Resonance method is not reliable test to use with every paper or board grade. Results of the resonance test are not reliable when the plies of the multilayer board moves separately due to the vibration, paper or board is very curled, or when the basis weight of paper is under  $40 \text{ g}/\text{m}^2$ . For example, resonance method cannot be used for measuring bending stiffness of corrugated board. (TAPPI, 1996)

Lorentzen & Wettre is also producing testing equipment for bending stiffness measurement called L&W Bending Tester (Figure 7). The test sample is placed in a clamp that is closed from the other end of the specimen when the measurement process is started. When the clamp is closed bending of the sample begins. Bending of the free end of the sample is continued to the selected angle (standard is  $5^\circ$ ) and the force needed for bending is measured throughout the whole process. Bending stiffness is then determined based on the following Equation:

$$S_{DIN} = \frac{60 \times F \times l^2}{\pi \times \Phi \times b} \quad (13)$$

where  $S_{DIN}$  = bending stiffness [Nm],  $F$  = force [N],  $l$  = length [m],  $b$  = width [m] and  $\Phi$  = bending angle [°]. (Lorentz & Wettre, 2013)



**Figure 7** L&W Bending tester measures resistance of board to bending. The other end of specimen is placed between clamps when free end is bent. (Lorentz & Wettre, 2013)

### 3.3 Surface roughness

Surface smoothness and roughness are important properties for packaging boards, printing papers and graphical boards. Surface smoothness and roughness are actually describing the same thing, how even the surface of the paper or board is. (Kajanto, Laamanen, Kainulainen, 1998)

The variation of roughness is divided into three categories based on its scale. Macro roughness scale is the biggest (0.1-1 mm) of the categories and it is a result of paperboard formation. The properties of printing and coating are primarily due to the macroscale roughness. The next category is micro roughness (1-100  $\mu\text{m}$ ) which describes the shapes and placing of the fibers and fines in the network. Paperboard gloss and its uniformity are both influenced by macro and micro scale roughness. The smallest scale is the optical roughness (<1  $\mu\text{m}$ ) and it describes the surface properties of single pigment particles and fibers. (Kajanto et al., 1998)

The factors that influence the most on surface roughness are coating and calendaring. The roughness of the paperboard base can be partly covered with an increase of coating. Roughness should not rise so high that it results to uneven printing result but some roughness is required to achieve a good adhesion of the coating layer. Formation, evenness of wire surface and the surface that is

compressed in wet pressing influence the surface roughness of the paperboard. (Kajanto et al., 1998)

Roughness is determined by measuring the deviation of surface height. This is executed by calculating the root mean square (RMS) of the high peaks of surface.  $R_{RMS}$  calculation by line scanning is performed with the Equation:

$$R_{RMS}^2 = \frac{1}{L} \int_0^L (z(x) - z_0)^2 dx \quad (14)$$

where  $L$  = length of the measured line [m],  $z(x)$  = local surface height and  $z_0$  = mean surface height [m]. (Kajanto et al., 1998)

In practice, roughness is usually measured indirectly. The most common indirect measurements are based on the permeability of air between the paperboard and a ring placed on it. Bendtsen and Parker Print Surf tests are the most used indirect roughness measurements. The result of the Bendtsen test is directly the flow rate of air [ml/min] that is forced through the space between the hard ring and the paperboard surface. Bendtsen determines mainly macro roughness because the hard ring does not comply the surface of paperboard.

Parker Print Surf (PPS) measurement is sensitive to micro roughness due to softer measurement head. The right term when PPS test is used is smoothness instead of roughness. PPS method determines the mean separation between the soft ring and a smooth surface by evaluating it based on the resulted flow rate. (Kajanto et al., 1998; Häggblom-Ahnger et al., 2001; VTT, 2015)

### **3.4 Strength properties**

#### **Delamination strength**

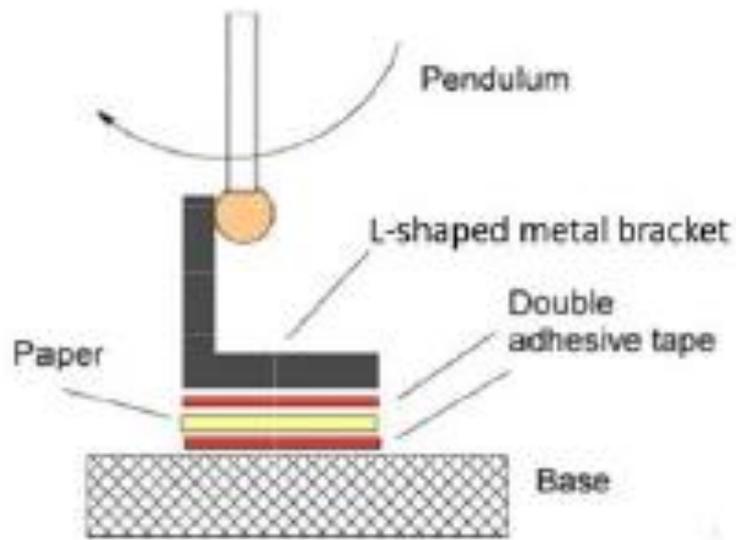
Multilayer structure of paperboard is depended on the bonds between fibers. These bonds are primarily due to hydrogen bonds between cellulose and hemicellulose hydroxyl groups. In addition to hydrogen bonds, van der Waals forces influence on inter-fiber bond strength. The bonding energy of van der Waals forces are smaller than hydrogen bonds but still necessary. Van der Waals forces form the cohesion of the wet web. Delamination occurs if these inter-fiber bonds yield. Delamination strength measures how much tensile stress paper or board endures in the direction of thickness. The cracking happens in the weakest

spot of the paper or board. Delamination strength is described in many different terms such as ply-bond strength, z-strength, Scott bond strength and internal bond strength. (Kajanto, 1998; Pöllänen 2008)

The term ply-bond strength is used when the delamination between different plies are discussed. Cracking of the bond occurs if the weakest internal bond is smaller than the ply bond. The internal delamination occurs usually in the middle layer because it is consisted of mechanical pulp that has a low bonding capacity. Regardless of the pulp type, the weakest spot in the multilayer board is the interface between the layers. The fiber network is not continuous in this interface because layers of board are formed in separate head boxes and couched together not until the end of the former section. Ply-bond strength increases in wet pressing, as the contact area between layers grows, and when the fine content of contacting layers increases. Fourdrinier wire section is acting on this principle as the fines concentrate close to the wire side and the wire sides are pressed against each other. (Kajanto, 1998; Pöllänen, 2008)

Delamination strength grows as the density increases due to wet pressing or beating. Increase in delamination strength is mainly due to the increased bonded area. Especially Condebelt pressing increases the delamination strength. It has been proven to give two to three times better delamination strength values compared to the conventional drying. Sizing improves the delamination strength because the bond strength increases.

There are many ways to measure the delamination strength but in every method tensile force is applied to the surface of the sample. One of the most known tester used for measuring delamination strength is the Scott bond tester (Figure 8). It measures the delamination energy by using pendulum principle. L-shaped metal hanger is attached to the surface of board with double-sided tape. Then pendulum hits the L-shaped bracket that causes delamination in the sample. The needed energy for the delamination of the paperboard is estimated based on the position that pendulum reached after hitting the bracket. The unit of the determined delamination energy is  $J/m^2$ . (Fellers, Östlund, Mäkelä, 2012)



**Figure 8** Principle picture of Scott bond tester. Both sides of the sample are covered with tape and tensile force is applied to the surface in which the bracket is placed. The required delamination energy is determined based on the pendulum movement. (Fellers et al., 2012)

At Ingerois mill delamination strength is measured with two other methods on top of Scott Bond. First is called Peeling that is performed with tensile strength measurement equipment (Figure 9). The 10 cm wide sample is cut in machine direction and measurement is done in two directions: fiber direction and against the fiber direction. The “peeling value“ is measured separately for surface and back layers by separating the layer from the board by hand. The sample is attached to metallic roll with tape and then the separated layer is put between the clamps as can be seen in the Figure 9. The measurement is started and the separated layer is pulled with the speed of 19 cm/min to the direction of clamp. The lower scale is showing the force needed for the detachment of the layer. The measurement is ready when the pointer in the lower scale is stabilized. The resulted value in that point is read from the upper scale and the result of the measurement is calculated with Equation 15:

$$T = \frac{G}{A} \quad (15)$$

where T = peeling value/number [g/cm], G = resulted value from the smaller scale [g], A = wide of the sample [cm].



**Figure 9** Delamination strength is measured at Ingerois mill with a tensile strength measurement equipment.

Delamination strength is also measured with L&W ZD Tensile Tester (Figure 10). First the sample is set to the measurement table and then the start button can be pushed. Automatic measurement process is started with applying of the tape on both sides of the sample. After tape has been set steel clamps are pressed against the taped surfaces with the force of 3000 N. The clamps are then started to pull apart and the force needed for detachment of board surfaces is the result of the test. The sample is then moved forward to continue the measurement process for the next measurement point. The result describes the force needed to split the sample. (Lorentzen &Wettre, 2013)



**Figure 10** Delamination strength is measured with L&W ZD Tensile Tester from every other machine roll. (ABB Inc., 2016)

### **Surface strength**

Surface strength describes an ability of paperboard surface to resist the strain without coating or fibers rising out from the paperboard. Surface strength depends on the internal hydrogen bonds. When printing process is considered, strain is caused by sticky printing material and the demands for the surface strength grows as the printing ink gets stickier. Good surface strength is a demand to achieve good printing result. Poor surface strength causes problems as the material looses from the surface of paperboard and sticks to the surface of rubber roll in the printing press. This prevents a proper transfer of printing ink to the printing sheet. Surface sizing can be used as a way to improve surface strength. Surface size is applied to the surface of paperboard and it forms hydrogen bonds with the fibers. These bonds enhance surface bonding capacity. (VTT, 2015; Holik, 2006; Gräsbeck, 1983)

Surface strength of coated paperboard can be measured with two different picking test technologies: wax method or IGT printability tester. Wax method is based on applying melted waxes (with different degree of hardness) to the surface of paperboard and as cooled wax is pulled away. Then the damages of the paperboard surface is checked. The surface strength is determined based on the wax that did not harm the surface of the board. The resulted value is based on the degree of hardness of the wax. (VTT, 2015; Holik, 2006)

The other method for picking test is based on the pressure applied to the test samples in printing with different speeds. The force, which is applied to the sample surface, is proportional to the speed of printing and ink stickiness. After printing, the first damages of the surface are searched and the printing speed in this point is the measure of surface strength [cm/s]. IGT measurement method is used at Ingerois mill. (VTT, 2015; Holik, 2006)

### **3.5 Dusting**

Dusting of the paperboard surface depends on the bonding strength of the surface. Weak adhesion of the coating layer, which could be due to the lack of binding agent, is the reason for detaching of coating. Poor adhesion of the coating can be a cause of dirt between the base paperboard and the coating. Too high temperatures at the beginning of the dryer section causes paperboard grades, which consist mechanical pulp, to stick to the cylinders. This leads to dusting. Surface sizing has a positive effect on dusting. With only a small amount of size on the surface of the paperboard can reduce the dusting problem significantly. Size bonds the dust particles to the surface of the board. (VTT, 2015; Mattelmäki, 2004)

One way to measure dusting is to determine the amount of loose fibers on back layer of the board. Test method is called "Tape test" and is an internal method in Stora Enso. Three board samples are cut from the machine roll and then samples are placed to table as a pile back layer upwards. The second sample (middle in the pile) is used for measurement. Tape is placed carefully on the back layer of the board and roller is run over the tape without pressing it. After rolling, tape is carefully ripped from the board. Tape is then scanned and evaluated with VveScan-program. The program identifies fibers and other loose particles in the tape. (Stora Enso, 2016)

### **3.6 Curl**

Curl of paperboard means exceptional structure of the paperboard sheet that is caused by asymmetric dimensional changes of paperboard. Usually curl is noticed after sheeting and it causes problems usually in finishing processes. If sheets are too curly they cannot be handled and transferred as they should. Curl occurs in the sheet only if there is two-sidedness of the in-plane strains. There are different reasons for two-sidedness of paper or board. First, two-sidedness can be a cause of the manufacturing process when the curl is detected at fixed moisture content.

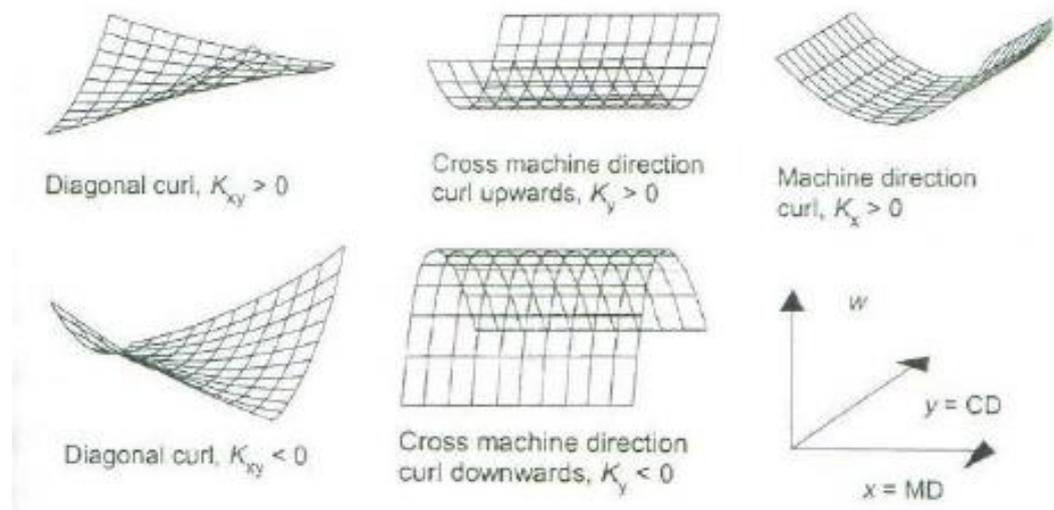
The drying of the paperboard during the manufacturing process has a major impact on curl. Especially, the final drying phase is important because the drying stresses are generated in that phase. When temperature of lower cylinders of the drying group is increased the board curl towards the bottom side of the web. Top side of the web is faced towards bottom cylinders so the curl turns opposite direction in relation to the hot side of the web. Explanation to this phenomenon is that when the top side of the web is dried more than the bottom side it cannot shrink freely because the bottom side stays unchanged. When the moisture from the drier top side is removed the bottom side dries and shrinks in proportion to the top side. (Kajanto, Niskanen, 1998; VTT, 2017)

Curl can occur as a consequence of the changes in moisture content and two-sidedness in the hygroexpansivity. The paperboard curls naturally to the direction of the last moisturized layer. As hygroexpansive two-sidedness of the paperboard occur ( $\beta_{\text{top}} \neq \beta_{\text{bottom}}$ ) curl appears. Typical cause of curl is two-sidedness of fiber orientation because it influences on the hygroexpansivity of the web. If stronger orientation on the wire side occurs it leads to lower hygroexpansivity on the top side and the increase of moisture content that causes the curl  $K_y$  rise. Dimensional changes occur bigger in CD than in MD and for that reason CD is prevalent. Dimensional changes can also occur also as a combination, which leads to a saddle-shape form of the board. Fiber orientation of individual board layers may vary independently. (Kajanto et al., 1998)

Wetting of the dried paperboard causes curl if it is not wetted through the whole thickness of the board structure. If the top layer of the board is moisturized the sheet turns first away from the moisturized side but later turns back. This phenomenon is caused by the expansion that happens first when the other side is moisturized and then the moisture profile in the thickness direction is balanced. Curl that is originated from re-wetting the board is permanent because the moisturized top side expands when the dry backside of the board is causing compressive stress. Curl is influenced by the asymmetric coating, for example one-sided coating, because the structure transforms to two-sided. (Kajanto et al., 1998)

Magnitude of curl is characterized with three curl components: MD, CD and diagonal curl. Typically, paperboard sheet curl occurs only in the form of

cylindrical surface and its axial direction is defined as the axis of curl. Magnitude of curl is defined with the opposite radius of curvature. Curl is described with  $K$  and its unit is  $[K] = 1/m$ . If the absolute value  $K$  is increased it means that also curl increases. Positive values indicate that sheet is curled upwards and negative values that sheet is curled downwards. Diagonal curl can be also called twist curl because the curl does not resemble the basic cylinder. In Figure 11 dimensions of curl are presented. (Kajanto et al., 1998)



**Figure 11** Magnitude of curl is described in three dimensions: CD, MD and diagonal direction. (Kajanto, Niskanen, 1998)

There is no standardized measurement method for board curl. Often the best method in practice is the direct determination of the out-of-plane deviation of sample sheet by comparing samples to reference pieces or by measuring the deviation with specific measurement unit. At Ingerois mill curl is measured in the control room right after the samples are cut from the machine roll sample. Sample sheet is cut from the whole width of the machine roll and smaller sample pieces, in which curl is measured, are then cut from the both sides (tending and driving side) of the cross directional sheet. In the control room, the sample is placed on the measurement box (Figure 12) and the value of curl is then read from the measurement scale. The normal values ranges between 5 mm and 7 mm because it is desirable that board has little curl toward the backside of the board. The difficulty concerning the measurement of curl is the humidity as the curl changes when the relative humidity changes. (Kajanto et al., 1998; VTT, 2015)



**Figure 12** Curl is measured with "Measurement box" at Ingerois mill. The board is placed against the white measurement scale at the bottom of the box where the curl value is read.

VTT has invented a measuring device, named OPTICUM, which measures curl of the round cut samples. Device measures the curl in three dimensions and the result describes how many millimeters the edges of the samples vary from the middle level of the sample disc. Samples are placed in a chamber where the humidity is set to the required level of the measurement. There are two versions of the OPTICUM meter: OPTICUM1 and OPTICUM2. OPTICUM1 can be used after wetting and other treatments when the OPTICUM2 measures the curl in specified humidity. The practical problem related to the use of OPTICUM is that the moisture equilibrium of the paperboard needs time to set in chamber. (Kajanto et al., 1998; VTT, 2015)

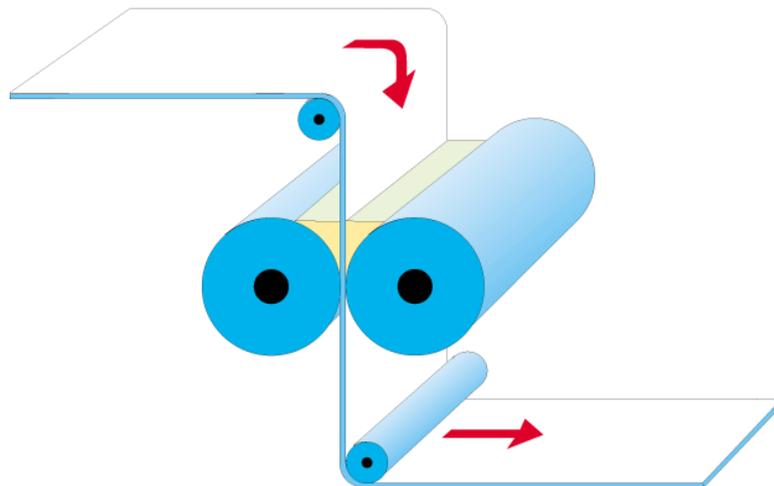
#### **4 SURFACE SIZING**

In surface sizing process, sizing agent is applied to the surface of the paperboard with size or film press to create a strong surface layer and to bind the fibers into surface. The main purpose of surface sizing is to improve surface and strength properties of the paperboard. Surface sizing improves both surface strength and internal strength of paperboard. When solids content of surface sizing agent is high the drying requirement of size decreases and energy savings can be achieved. Surface sizing is often placed before coating to seal the surface of the board but it can also be as its own process with no coating applied on top of the size. In the following chapters different coaters, application technologies and drying of the

surface size are presented. (Biricik, Sonmez, Ozden, 2011; Lipponen, Grön, Bruun, Laine, 2003)

#### 4.1 Pond size press

In conventional pond press, application of surface size is executed with nip of two rolls and a pond filled with size. The web goes through the nip of two rotating rolls and the pond is located in between the web and rolls (Figure 13). Advantages of pond size press are a good penetration of size to the sheet structure, its reliability because of the low maintenance need, the steady quality of paperboard, and runnability at lower machine speeds (under 800 m/min). High speeds result to high amount of web breaks. Weak spots of web are formed because of hydrodynamic forces in the pond and ruptures of these weak spots can cause web breaks. Excessive amount of size packs to the holes of the web and after some time the size sticks to the top roll, which then tear up the web. In addition, excessive wetting in the pond might cause reduction of web strength in the after-dryer section. Weakness of pond size press is the adjustment of the size amount to the web. The moisture content of web is high (40% to 50%) after exiting the pond press. This lowers the strength of the web and increases the drying demand. (Hägglom-Ahnger et al., 2001; Grön, Rantanen, 2000)

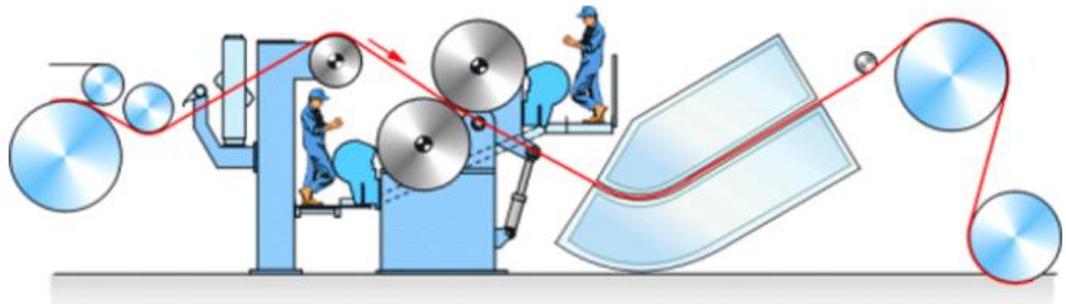


**Figure 13** The pond size press forms a size pond between the rotating rolls. The size amount is difficult to control with size press. (VTT, 2015)

#### 4.2 Film size press

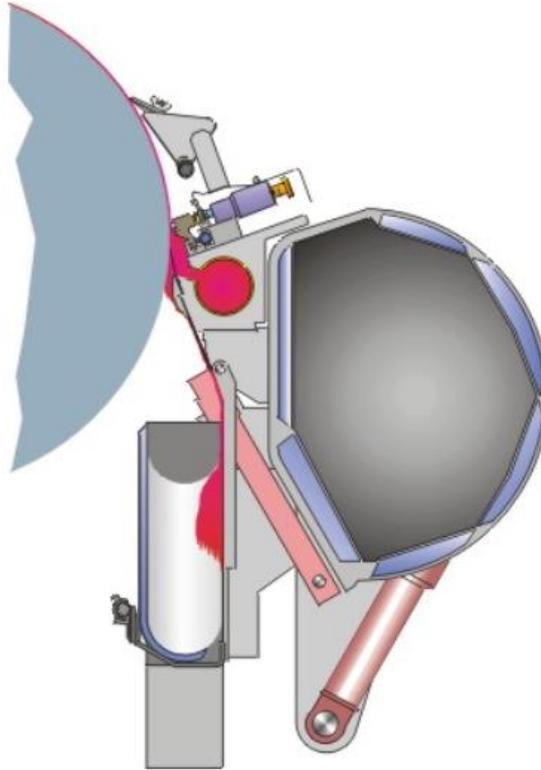
The film size press uses different techniques for application of surface size compared to the pond size press. The conventional film size press (Figure 14) is

consisted of two rolls that form a nip in which the web goes through. First, surface size is applied to the roll surface and from there it is transferred to the paperboard surface in the nip. The nip pressure is between 20 and 40 kN/m. Due to high nip pressure water of the size is penetrated to the board structure. This penetration leads to higher solid content of surface size between the paperboard and roll. The size penetration depends on the base paperboard openness and the properties of size. Size amount is easier to adjust with the film size press than with pond size press as the wanted amount is preapplied to the roll surface with metering unit such as blade or rod. The drying requirement of surface size decreases if size is metered properly. In addition, there is no speed limit when there is no pond. It is possible to apply two different sizes for the top and back layer of the web because the size application is done individually for both rolls. Film size press has one considerable weakness compared to pond press: the size does not penetrate to paperboard web as well as in pond size press which decreases the bonding strength. (Grön et al., 2000; Häggblom-Ahnger et al., 2001)



**Figure 14** A conventional film size press. (VTT, 2015)

The most common technology in film size presses is a preapplying film size press that utilizes short-dwell type of metering. In many cases, blades or rods are used as metering elements. Especially rods are used in surface sizing (Figure 15). The size film amount is formed based on the cross-sectional area that is formed between the rod and the roll. Factors that influence on the amount of size are elastic modulus of roll coater, rod load and form of groove profile. (Grön et al., 2000)



**Figure 15** Short-dwell size applicator where grooved rod is used for metering. The amount of surface size can be easily controlled by changing the parameters of a metering element. (VTT, 2015)

Rod can be either grooved or smooth. Metering principle with grooved rod is volumetric which means that the thickness of the size is determined by the open area between the rod and the roll. The profile of the rod is chosen based on the wanted size amount and size properties. Concern with grooved rods is the rapid wear that influences the uniformity of the film amount. The wear of the rod can be minimized by choosing softer cover for applicator roll and by reducing the rod load. Rod has to be changed when film cross profiles are not acceptable anymore. One risk in surface sizing is that roll becomes size-repellent because of deposits. This may lead to skipping in small scale. The reasons for deposit forming can be unstable defoaming agents in the size, water-repellent agents and dispersions that are mechanically or chemically unstable. (Grön et al., 2000)

Grooved rod with large diameter was developed to reduce the rod wear and scratching of the roll covers. By increasing the wear time of the rod, streaks in the size film decreases. Because of the hydrodynamic forces the large-diameter grooved rod acts more like a smooth rod. The contact area between rod and roll cover increases when diameter of the rod is increased. Larger contact area

decreases the specific pressure between the rod and cover roll, which leads to longer wear time. In longer nip (larger diameter of the rod) higher hydrodynamic pressure is formed and the size film smoothens. Longer nip prevents streaks and scratches better than short nip because impurities goes through it more easily. (Grön et al., 2000)

Smooth rod is acting on hydrodynamic principle. The hydrodynamic force is created between the rod and roll cover in the converging nip. The film amount is determined with the rod loading pressure, rod rotation speed, rod diameter and surface size properties such as solids content and viscosity. The hardness of the roll cover has to be considered as it also influences the amount of the size. (Grön et al., 2000)

### **4.3 Application techniques of coating or size**

#### **Applicator roll**

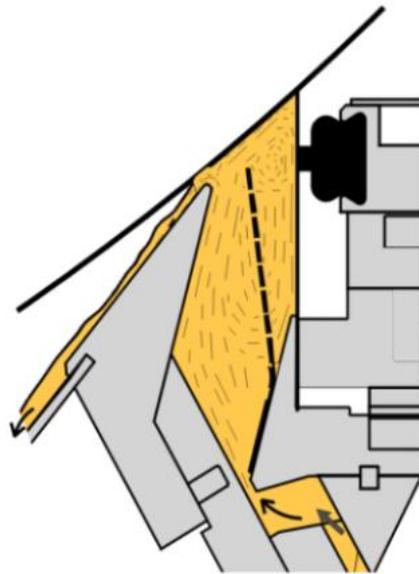
Applicator roll is consisted of the applicator pan, backing roll and smaller application roll (typically 30% - 40% of backing roll diameter) which is located underneath the backing roll. Bearings of backing roll are typically fixed to the support frame or straight to the coating machine. Instead, the bearings of the applicator roll are installed on levers that can be adjusted. The applicator roll is rotating to the same direction as the web is running but with the speed of 15% to 25% of the speed of the machine. Factors that influence on the amount of coating that stays on the web are the gap between the rolls, the diameters of the rolls, the speed of the applicator roll and the properties of the size or coating. The applicator roll is partly submerged into the size of the applicator pan during the operation. When the pan is full of size, applicator roll transfers size to the roll nip where it is applied to the web. The pressure in the application nip is high (maximum pressure 0.5 – 4 bars) which provides even and uniform first layer of the size. When web exits the nip, part of the size remains on the surface of the web and part goes back to the pan as a return flow. With high machine speeds (increased turbulence in the nip entry), the returning part of the coating can splash and cause air entrapment that leaves uncoated spots to the web. Board web travels around the backing roll to the metering element after size application. Before the web reaches the metering element it takes up the moisture from the size layer and at the same time

the size is dewatering. Metering of color or size can be done with rotating rod, blade, or air jet. (Linnonmaa, Trefz, 2000)

Long dwell time is both an advantage and a disadvantage. With a long dwell time and high amount of coating/size high coat weights are easier to reach. The strength properties of the paperboard weaken if the liquid amount penetrating to the fiber structure is too high. The dewatering phenomenon influences on the maximum solids content of the coating because part of the coatings liquid phase is absorbed to the base paperboard. As the coating layer reaches the metering element it might be so thick that it causes the metering element to crack or bend. Diluting of coating color solves the problem but drying requirement grows higher. This effect makes it difficult to apply lower amounts than  $6 \text{ g/m}^2$  of coating to the paperboard. (Linnonmaa et al., 2000)

#### **Short dwell time applicator**

In the short dwell application, coating or size is placed in an application chamber in which it is fed through an opening in the bottom of a chamber. One side of the chamber is the moving web that causes movement and mixing of coating/size in the chamber. The return flow of coating/size influences the mass equilibrium of the process and the removal of excess air coming to the chamber has to be executed carefully. Return flow goes back to the machine circulation from the return gap which is the space between the web and regulation edge (Figure 16). (Linnonmaa et al., 2000)



**Figure 16** Short dwell time applicator. The coating is fed to the chamber from the bottom of it and the return flow to the machine circulation flows between the web and the regulation edge. (VTT, 2015)

The main benefit of short dwell applicator is that it does not weaken the web as much as the applicators with longer dwell time. Stronger web has lower amount of web breaks. Weakening of web is due to the too much dewatering of the coating. Dewatering causes the coating color thickening in the machine circulation because of the loss of water to the base paper or board. Harmful dewatering can be avoided with short dwell applicator due to the short dwell time and low pressure in the coating chamber. The fibers of the paperboard do not have time to swell before metering (short dwell time) which causes the fibers swell after metering. When fibers swell after metering surface of coated/sized board is rougher than with applicators with longer dwell times. (Linnonmaa et al., 2000)

### **Jet applicator**

There are two different types of jet applicators: Premetered jet applicators and free jet applicators. In premeter applicator technology a thin coating film is premetered to the web and after that the layer is metered again with metering blade. This leads to no splashing in the dwell area, lower amount of coating and uniform coating film. The pressure in the coating chamber has to be adjusted precisely to achieve good runnability. The pressure can be controlled with overflow baffle blade and the pumping rate to the applicator. This requires a careful adjustment of applicator

head, which is placed under the backing roll. Because of difficult placing of applicator head the adjustments are complicated to execute. Use of two separate metering elements in one backing roll cause problems when the web becomes loose between the elements and the web tension cannot be controlled. Web loosening can be avoided by having two separate rolls where there is only one metering element in roll. Downside of two rolls is that more space and investment are required. (Linnonmaa et al., 2000)

The free jet applicator contains a nozzle (nozzle gap is 0.6-2 mm) which is placed under the backing roll. The jet of coating color is adjusted with the shape of the nozzle. The speed of the jet is adjusted by the nozzle gap and the flow rate of the coating. The speed of the web needs to be higher than the velocity of the jet to achieve good impingement of the jet of coating color to the web. This speed difference stretches the jet and forms a uniform layer of coating. Benefit of the free jet application technique is that the application of coating does not cause film split patterns or overflow. Compared to other application technologies free jet applicator has two major advantages: there are no speed limitations to the coating station and applicator does not have contact to the web or the backing roll. (Linnonmaa et al., 2000)

#### **4.4 Drying**

During drying process of surface sizing, the excess water from the size is removed. The most common drying methods for coated or surface sized paperboard surface are cylinder drying, air-drying and infrared drying. Drying system can be a combination of different methods. Drying process of size is described as a four-stage system, which starts when size is applied to the paperboard surface. First water is absorbed from the size to the paperboard and fibers are starting to swell. Evaporation does not occur in this phase. This first phase, which ends to the first dryer, should not last too long especially if the temperature of the web is high or the paperboard absorbs a lot of water. The second phase contains the process from the first dryer to the beginning of an immobilization of the surface size. Non-immobilized size or coating layer is damaged if it suffers any mechanical contact in this stage. This needs to be considered in the machine layout and choose a non-contact drying method. During second phase, the web is heated strongly to reach the evaporation temperature and

water of the surface size continues absorbing to the paperboard. The third phase of drying, which influences on the product quality critically, includes the immobilization of the surface size layer. Immobilization point describes the moment when the binders stop moving. In addition, absorption of water to the base paperboard decreases. If drying is too powerful in the third phase, it leads to print mottling. The final moisture content is defined in the fourth phase. The quality of size layer does not change significantly in the final phase of drying process. (Hägglom-Ahnger et al., 2001; Heikkilä, Rajala, 2000)

Drying of the coating or sizing influences to the curl phenomenon of the paperboard. As described in Chapter 3.6, curl is changed when the moisture content of the board structure is altered. One-sided treatment (such as surface sizing) changes the moisture content of the paperboard asymmetrically and causes curl. The curl of the paperboard can be adjusted by controlling the drying section. (Kajanto et al., 1998)

### **Air-dryer**

Air-dryers are divided into two different types: air flotation dryers and single-sided air impingement hoods. In air flotation dryers, air cushions or air jets support the web on both sides. The most important part of air dryer are the air nozzles that blow the hot air to the web. Nozzles control the evaporation and runnability of the web. The distance between nozzles and web depends on the nozzle type. The distance, where the maximum heat transfer rate is achieved, is about 2-10 mm with every nozzle type. Two-sided air flotation dryers can be used at all web speeds and basis weights because of their stable web runnability. Single-sided impingement dryers are used when the web has to be dried only from one side. Typically, rolls from the other side support the web. This is advantageous especially with low web speeds and high web strengths. Dwell time of web in drying section depends on the machine speed. With typical production speed of board machine dwell time is <1 second. (Hägglom-Ahnger et al., 2001; Heikkilä et al., 2000)

The evaporation of water is executed by blowing hot air to the web surface. For energy-saving reasons 80-90% of the air is circulated back to the dryer and 10-20% of the air is removed as exhaust air. The hot air is usually heated with in-line

gas burners or steam coils. The heat source selection is based on the required air temperature, price of energy and investment costs. When steam-heated dryer is used the available steam pressure defines the maximum impingement temperature. Typical operating pressures is 10-15 bars which leads to the impingement temperature of 170-185°C. The designed operating temperature in gas-heated dryers is almost twice higher (300-350°C). All air-dryers are manufactured so that impingement velocity can be controlled but the velocity is typically 40-60 m/s. Advantages of the air-dryers are high specific evaporation rate and energy efficiency. Compared to infrared dryers drying efficiency is lower but the drying process is gentler and the risk of overheating the web is lower. (Hägglom-Ahnger et al., 2001; Heikkilä et al., 2000)

### **Infrared dryer**

Thermal radiation is electromagnetic radiation that is independent from the medium. The wavelength area of thermal radiation in the electromagnetic spectrum is 1-100 µm. As thermal radiation meets the web some of the radiation goes through the web, some reflects back and some absorbs to the web and transforms to heat. The heat flow is dependent mostly on two factors: temperature and emissivity of the object. Emissivity of the object is at highest level at wavelengths between 1.0 µm and 1.4 µm.

Infrared dryers usually consist of several individual elements that can be controlled separately. Due to individual control of different elements, drying efficiency and the cross-directional moisture content of the web can be optimized. Dwell time of web in IR drying section is <1 second with typical machine speed (500-900 m/min) in board production. Advantages and disadvantages of infrared (IR) dryer are listed in Table II. (Hägglom-Ahnger et al., 2001; Heikkilä et al., 2000)

**Table II** Advantages and disadvantages of IR dryer technology. (Hägglom-Ahnger et al., 2001; Heikkilä et al., 2000)

<b>Advantages</b>	<b>Disadvantages</b>
Little space demand	High operation costs (especially electrical IR dryers) compared to other dryers
Effective increase of web temperature (especially electrical IR dryers)	Risk of fire in web breaks
Fast controllability	High energy density restricts the amount of rows that can be used
Possibility to direct the infrared radiation to certain area	Risk of overheating the web

Radiative material can be heated in two ways: electric current or gas flame. Electrical IR radiators are usually halogen lamps with tungsten filament. The radiation to the web is controlled by the reflector behind the lamp. Reflector can be a big plate that is used to reflect the energy from all of the lamps or then every lamp can have their own reflector. Reflectors are coated with material that has good emissivity. The heat energy of gas-heated IR radiators derives from the burning of gas-air mixture. Produced combustion energy keeps the radiator temperature at the level which heat radiation is emitted. Typically premixing of gas and air is placed before the burning area and then the mix is transferred to the burner through nozzles or porous material. The adiabatic temperature of burning process is 1940-1970°C and the produced energy is transferred partly convectively to the emitter. Temperature of the emitter is kept between 800°C and 1100°C. In the front of this emitter, which is made of ceramic or metallic material, is a ceramic or metal screen that operate as a radiator. This plate is also heated with the combustion energy. The distance between IR dryer and the web is few centimeters and profile should be as uniform as possible. (Hägglom-Ahnger et al., 2001; Heikkilä et al., 2000)

Wavelength of electrical IR dryer range is 0.8-2  $\mu\text{m}$  and the emitted wavelength of gas-heated IR dryer is 1.5-2  $\mu\text{m}$ . The wavelength area of electrical IR dryer is better considering the optimal wavelength area of the emissivity. The web temperature can be raised higher with electrical IR dryers than with the gas-heated dryers. Typically, IR dryer is the first dryer after coating process and the web

temperature has to be increased quickly to the level in which the water evaporates and coating consolidates. The radiation efficiency of electrical IR dryer is 80%-85% when the efficiency of gas IR dryer is only 28%-55%. (Hägglom-Ahnger et al., 2001; Heikkilä et al., 2000)

### **Cylinder dryer**

Cylinder drying cannot be utilized before the coating layer is immobilized because the web does not withstand mechanical contact before that. Cylinders are typically used in the end of the drying section of coated board. Cylinders do not only dry the web but also moves it forward. There is usually only 2-6 cylinders in a coating machine but the amount can be higher in double sized film press coating. Dwell time in cylinder drying (3 drying cylinders) is approximately 1-2 seconds in typical machine speeds of board production. Cylinders in the end part of the drying can alternatively be used for cooling of the web. The cooling of cylinders is accomplished with cold water that is sprayed inside of the cylinder wall. Water is removed by using compressed air and pressurizing the cylinder with it. (Heikkilä et al., 2000)

Felting is used to press the web against cylinder surfaces. Felting can be executed with different geometries. Felted system with two fabrics (the other at the top cylinders and the other at the bottom cylinders) is called double felting. Double felting is efficient for drying but it has poor runnability in high web speeds. Bottom fabric causes difficulties in cleaning and removal of broke. Cleaning problem can be solved by removing the bottom fabric but it decreases significantly the drying capacity of the bottom cylinders. Slalom felting geometry is utilized in machines with high speeds to achieve good runnability. The fabric presses the web against the top cylinders and on the bottom cylinders fabric isolates the web from the cylinders. The web is supported through the whole process but the drying capacity of bottom cylinders is poor. In addition to felted cylinders, unfelted cylinders are used. Problem with unfelted cylinders is the air film that is formed between the web and cylinder. The air film is formed because the web tension alone is not enough to create a close contact between cylinder and web. This problem occurs especially with low weight paper grades. Unfelted cylinders can be a good option when heavier grades are dried because of higher web tension and lower speed of the web. (Heikkilä et al., 2000)

Open hood structure is simple and it is often used in coating machines. Cylinder group is covered with open hood. The ends of the hood, tending side and sometimes the drive side, are opened to allow an easy access to the cylinders. Disadvantage of open hood structure is the large amount of steam energy which is used for heating the exhaust air coming from the machine room. This energy loss can mostly be avoided with a closed hood that covers the whole cylinder group. Access for maintenance is made possible with lift doors on the tending side. Closed hoods are used in case of large size cylinder groups or when the evaporated water volume is high. (Heikkilä et al., 2000)

#### **4.4 The effect of web properties**

The base of paperboard in surface sizing should create the best possible conditions for the runnability of the machine to avoid breaks. The properties of the base board has to be such that size is easy to apply on it. Web should consist as little mechanical flaws (such as holes, wrinkles and streaks) as possible. Factors that influence on the absorption of the size are hydrophobic sizing capacity, porosity, roughness and moisture content of the base paperboard. Even a little amount of hydrophobic size reduces absorption of surface size significantly because the absorption of the size is highly dependent on hydrophobic sizing capacity. (Pylkkö, 2000; VTT, 2015)

Porosity and roughness of paperboard are determined based on the quality requirements of certain paperboard grade. For this reason, these properties are not often used as control parameters in sizing. Although not used as control parameters in the process, porosity and roughness of the base paper or board have to be monitored and maintain in the certain level. In practice, as the pore size of the board increases the absorption of size increases too. (Pylkkö, 2000; VTT, 2015)

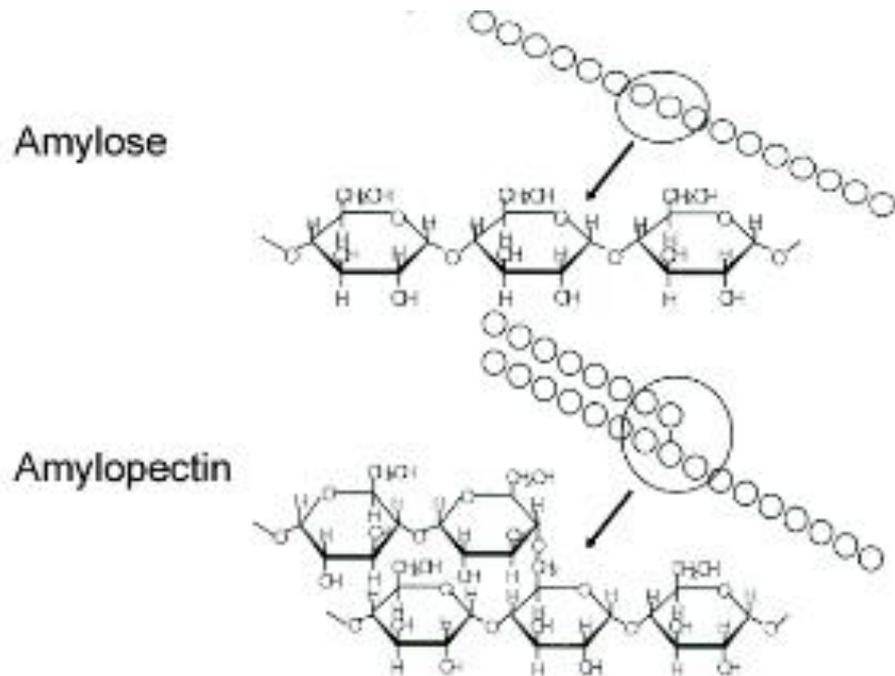
The optimal web moisture level of the paperboard depends on the pulp that is used. When mechanical pulp is used, the moisture content level is high. In general, lower moisture content of paperboard increases the amount of absorbed size. Important factor considering moisture content level is an even moisture profile to avoid uneven consistency. (VTT, 2015)

#### **4.5 Surface sizing agents**

Starch is the most common surface sizing agent. Starch contains high amount of hydroxyl groups that forms hydrogen bonds with water and fibers. This leads to enhanced paperboard strength and stiffness. The use of starch improves also dimensional stability and offset printability. One advantage of starch is its low price compared to other sizing agents. There are also other less used surface sizing agents such as carboxymethyl cellulose (CMC) and polyvinyl alcohol (PVA). (Radosta, Kiessler, Vorweg, Brenner, 2016; Ketola, Andersson, 1999; Häggblom-Ahnger et al., 2001)

##### **Starch**

Starch is a glucose polymer and its anhydroglucose units are joined with 1,4- $\alpha$ -D-glucosidic bonds. Starch is consisted of two main components (Figure 17), straight-chained amylose and branched-chained amylopectin. The amount of amylose varies between 20% and 25%. Especially amylose fractions have a tendency to retrograde which means forming of complexes of naturally occurring free fatty acids and amylose. These complexes are called Retrograded Amylose Particles (RAPs). RAPs cause problems by forming sediment to the storage tanks and by accumulating in size press. Runnability of the paperboard machine suffers because of the variation in the sizing solution. RAPs cause also dusting during drying and decrease in paperboard strength. RAPs are formed more easily in certain conditions. The storage time should be kept short (less than 2.5 hours) and storage temperature under 70°C for the starch paste. When pH of starch is adjusted over seven risk of having RAPs decreases. In practice, if large areas of RAPs are formed the whole surface sizing system has to be emptied and cleaned before starting again. (Cargill, 2013; Ketola et al., 1999; Voigt, 1990; Mäkelä, 2000)



**Figure 17** The main components of starch are amylose and amylopectin. The difference between components is the structure of the starch chains: Amylose is straight-chained component and amylopectin is branched component. (VTT, 2015).

Because of the organized, hydrogen bonded structures of starch its granules are not soluble in cold water or alcohol but in higher temperature starch becomes soluble to water. When the temperature is high enough starch granules start to swell and open, and some of the polymer fractions of lowest amylose come out from granules. These changes in the chemical structure of starch lead to rise in viscosity. This physical starch modification is called gelatinization. Temperature that is required for gelatinization is dependent on the source of starch. (Annushko, 2013)

Starch is an optimal polymer to be used as a surface sizing agent because of its ability to form hydrogen bonds like cellulose. The straight-chained amylose has a tendency to form tight bonds to the surface of chemical pulp fibers. The branched amylopectin forms bonds with fibers and starch molecules. These bonds result to 3-dimensional network of bonds. The hydrogen bonds of starch are not particularly strong in water solution because of the tendency of water to form competitive hydrogen bridges. Strong hydrogen bonds are formed between starch and the other components when the starch size is applied on the surface of the board and water is removed in drying. (VTT, 2015)

Starch can be extracted from different raw materials such as potato, wheat and corn. Differences between native starches are density, size, composition and shape of starch. Components that are causing problems, like foaming, are proteins and fats. The low content of these components in potato starch is one reason that makes it popular (Table III). Nowadays, proteins and fats are separated from the starch, not only because of the problems in paperboard production, but they are also very valuable components in other industries. For example, separated maize oil is used in food industry. Wheat and corn are becoming very popular in forest industry because the harmful components can be separated very efficiently. In addition, wheat and corn starches are not as expensive as potato starch. (Cargill, 2013)

**Table III** Chemical compositions of starch raw materials. (Ketola et al., 1999)

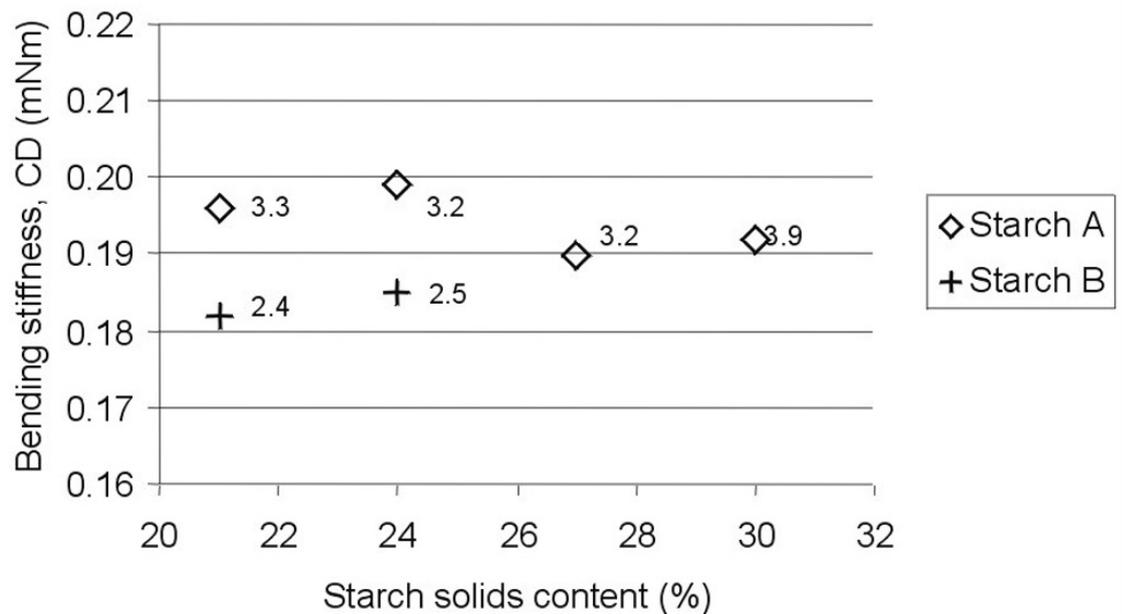
	<b>Potato</b>	<b>Corn</b>	<b>Barley</b>	<b>Wheat</b>	<b>Tapioca</b>
<b>Moisture, %</b>	70-85	7-23	13	10-13	60-75
<b>Gelatinization temperature, °C</b>	60-85	75-80	80-85	80-85	65-70
<b>Starch, %</b>	13-25	54-66	56-64	54-65	20-30
<b>Protein, %</b>	1-4	7-12	7-10	9-15	1
<b>Fat, %</b>	0.02-0.1	3-5	2-3	1-2	*
<b>Fiber, %</b>	0.2-3	2-3	10-13	2-4	2
<b>Minerals, %</b>	0.4-2	1-3	2	2	*
<b>Sugars, %</b>	1	1-3	1	1	*

\* In total 3%.

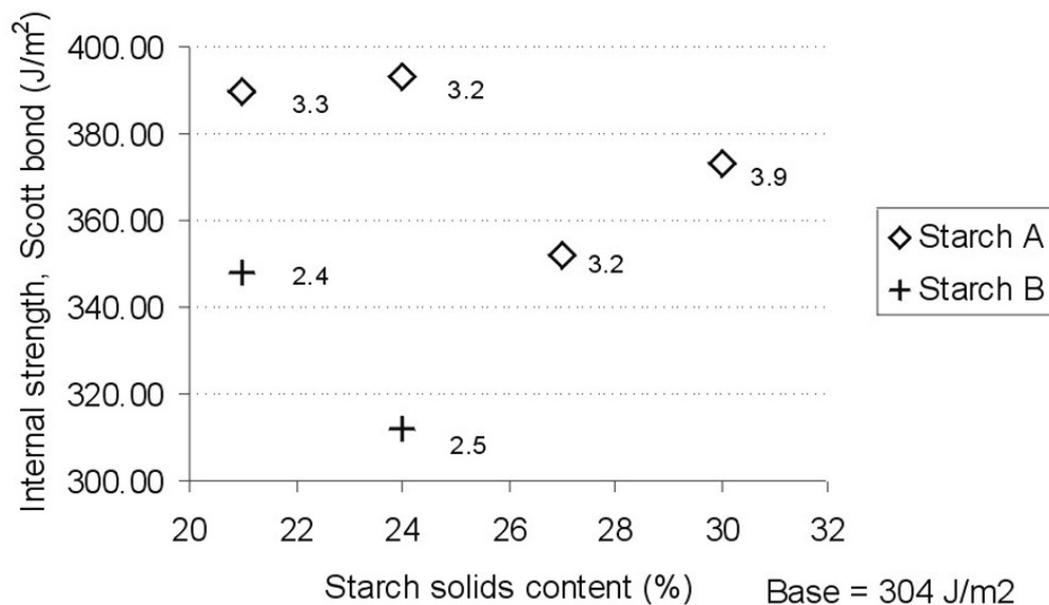
Starches from different sources differ with their gelatinization temperatures. As can be seen from the Table III potato has the lowest gelatinization temperature. This needs to take into consideration in the manufacturing process. Weak cooking causes highly swollen and weakly dispersed starches which results to paste that resembles salve. Cooking should neither be too rough because it results to starch paste acting like hydrocolloid. The effect of the starch on the board properties or on the acting in the surface sizing process is influenced more of the starch solids content and viscosity than the raw material of starch. (Cargill, 2013)

The effect of solids content of starch influence the penetration of the size to the paperboard in surface sizing. Lipponen et al. has investigated the effect of the solids content of starch solution increase from 8% to 21-30% in surface sizing. Trials were performed for uncoated fine paper with metered size press. 10 mm

smooth rods were used in film metering and cover of the roll was made of polyurethane with hardness of 36 P&J. Two starches with different viscosities were studied. Viscosity of starch A was 20 mPas and viscosity of starch B was 100 mPas. In this study was found that by increasing solids content starch penetration was decreased and more starch solution stayed on the surface of the paper. When compared starch solution with 21% and 30% solids content no big differences in surface strength or bending stiffness were found. The results of bending stiffness during trial are presented in Figure 18. Instead, internal strength (Scott bond) decreased due to the smaller amount of penetrated starch solution in to the paper (Figure 19). Starch B stayed more on the paper surface than starch A that had lower viscosity. The decrease in Scott Bond values was bigger with starch B because of the higher viscosity. (Lipponen et al., 2013)



**Figure 18** Bending stiffness was investigated when solids content was raised from 21% to 30%. The amount of starch is presented next to the measurement points. (Lipponen et al., 2013)



**Figure 19** Scott Bond strength was monitored when solids content was raised from 21% to 30%. The amount of starch is presented next to the measurement points. (Lipponen et al., 2013)

In addition to surface sizing, starch is used as a dry-strength additive which is added to the wet end. Starch influences mainly to z-directional strength values. Scott Bond strength and IGT printability test indicate the influence of starch on delamination strength. Typically, as the amount of starch increases Scott Bond strength increases. The improvement (increase) in Scott Bond strength is almost linear during increase of cationic starch. When cationic starch was added to the surface layer of the board Scott Bond strength increased but also elastic modulus and IGT values were increased. IGT surface strength improved more during surface sizing with high amount of starch (3.5 g/m<sup>2</sup>) than by adding cationic starch as a dry-strength additive. (Ketola et al., 1999; Nybolm, 1991)

#### **Other surface sizing agents**

Carboxymethyl cellulose (CMC) is a synthetic component that is produced in a reaction of alkaline cellulose and monochloroacetic acid or its sodium salt. It is sold as a powder form and delivered in bags to the customer. CMC controls the water retention and oil adsorption in surface sizing but it is also used as a binding agent and thickener. CMC forms lumps at a temperature under 50°C which means that it has to be dispersed in warm water. Lumps cause problems in the proceeding process by blocking screens. The concentration of CMC is limited because of its

radical increase of viscosity. CMC improves the elasticity of surface and abrasion resistance. (VTT, 2015; Mäkinen, 2000)

Polyvinyl alcohol (PVA) is composed of hydrocarbon chain that has hydroxyl group in every other carbon. PVA is sold as a powder form and is delivered in big bags to a paperboard mill. Solid PVA is produced in a two-stage process: Radical initiated polymerization of vinyl acetate and alcoholysis of polyvinyl acetate. Properties of PVA are formed based during these processes. The batch cooking process of PVA is performed the same way as the batch cooking of starch. PVA is added to the vessel with an efficient agitator and inlet tube for the heating steam. Steam is then leaded to the vessel. PVA slurry mixing has to be strong to avoid lumps. Temperature of the slurry is raised up to 90-100°C and kept high for 20-30 minutes. The maximum viscosity of PVA is in temperature between 65-75°C. When PVA solution clears it is stored to the storage vessel where the temperature is kept warm to avoid changes in viscosity of solution. The most important properties of PVA considering surface sizing are its total solubility to water, its film-forming properties and its adhesion to fibers and fillers. These properties improve significantly dimensional stability and surface strength of paperboard. PVA can be used individually or as a combination with starch. Viscosity needs to be controlled carefully when PVA is combined with starch. (Hentzschel, 2000; Mäkinen, 2000)

#### **4.6 Surface size additives**

Effect of surface sizing can be boost by adding specific compounds to starch. Hydrophobic polymers are added to starch to improve printing result. Hydrophobic polymers can be either film formers or non-film formers. SMA is water-soluble compound which forms an even film on the surface of the paperboard. Polyurethane and styrene acylate are formed as little balls in water dispersion. When this dispersion is added to starch they form a matrix with hydrophobic spots and hydrophilic starch areas.

In addition, some internal sizes such as ASA (Alkenyl Succinic Anhydride), AKD (Alkyl Ketene Dimer) and rosin size can be added to the starch. ASA and AKD are synthetic, water-soluble neutral sizes that react with hydrogen groups of cellulose. AKD and ASA form film matrix including hydrophilic areas of starch and areas of hydrophobic dots of AKD or ASA. AKD was the most used neutral

size at the beginning but the popularity of ASA has grown. ASA is cooked entirely during the manufacturing process and there is no need for excess drying before surface sizing. Cooking of AKD continues during storage. The required pH area is more restricted with AKD (more than 6.5) than with ASA (pH 4.5-8). ASA does not cause slippery as AKD and ASA can be easily removed with caustic. One significant disadvantage of ASA compared to AKD is the fast reaction between ASA and water. Due to reactivity with, ASA has prepare just before it dosing it. ASA reacts with water of emulsion, loses part of its capacity, and forms accumulation to the process. ASA requires good retention and accurate dosing to avoid hydrolysis. ASA does not provide as good hydrophobicity as AKD, which makes it suitable for printing and writing paper production. (Hägglom-Ahnger et al., 2000; VTT, 2015)

Rosin sizes are derived from natural oleo-resin of softwood. The most common source of rosin is Northern pinewood. Rosin size is water insoluble so it has to be converted before using as an internal size. After converting rosin size is either as a form of soap or as a form of dispersion. Rosin size is attached to the surface of the fiber with the help of alum or cationic alumina floc. The use of aluminum compounds requires well-monitored wet end chemistry. The biggest advantages of rosin size compared to neutral sizes are the low price of rosin size, low reactivity with water and good tolerate of size storage time. The disadvantages of rosin sizes compared to neutral sizes have influenced to the growing popularity of neutral sizes. The required amount of size is more than twice higher with rosin size than with neutral sizes. The higher amount of size decreases the strength values of the board. Rosin size is not reacting well in neutral and alkaline conditions or with high pulp temperatures. (Hägglom-Ahnger et al., 2000;VTT, 2015)

Conductivity of paperboard can be increased with 0.2-0.5% salt addition to the surface size. Especially in office papers the salt addition is important. The risk of sheeted office papers sticking to each other with electrostatic forces is decreased. This sticking causes problems in printing and copy machines. Too high salt addition causes problems electrical graphics development in the copy machines. In addition, the foaming in size circulation can be controlled by salt addition. (VTT, 2015)

To improve optical properties of the paperboard surface pigments can be added to the starch paste. Pigment particles fill the paperboard pores and by that pigmentation influences porosity of the surface. Typically, dispersion contains 50% of pigment and the other half of starch. Elastic modulus of the pigments differ in a range of 3-25 GPa depending on the pigment and its properties. Elastic modulus is increased when the elastic modulus of pigment-starch dispersion is higher than elastic modulus of the board. (VTT, 2015; Häggblom-Ahnger et al., 2001; Kajanto, 1995)

## **5 MODIFICATION OF NATIVE STARCH**

Native starch has to be modified before using in surface sizing because of its high viscosity. Viscosity of surface size has to be optimized in a level that strength improvement can be achieved. Low viscosity of starch indicates that starch chain is degraded too much which decreases bonding ability of starch. In addition, viscosity and concentration of starch solution influence to the machine runnability and starch penetration. Modification can be carried out in several different methods. These modification methods are categorized into two groups: degrading and substituting. Substituting of starch means chemical modification such as etherification and esterification. For example, changing the nature of starch to positively charged by cationization dewatering properties improve and retrogradation is prevented. (Bruun, 2000)

Starch degradation can be performed either on-site or at the starch modification plant. On-site degradation is performed usually either with thermochemical oxidation or with enzymatic degradation. At the starch modification plant, the degradation of starch is executed by utilizing oxidants such as hypochlorite, hydrogen peroxide or acid hydrolysis. Difference between on-site and pre-degraded starch is the stability of the product quality. This is caused by the different conditions in the processes. On-site process conditions have to be adjusted in a way that viscosity is low enough. Conversion is typically exaggerated to make sure that viscosity is not increased too high. This exaggeration leads to variation in properties of product. Starch modification plant has milder conditions compared to on-site degradation that provides more equal properties for the product. (Bruun, 2000)

### **5.1 Thermochemical oxidation**

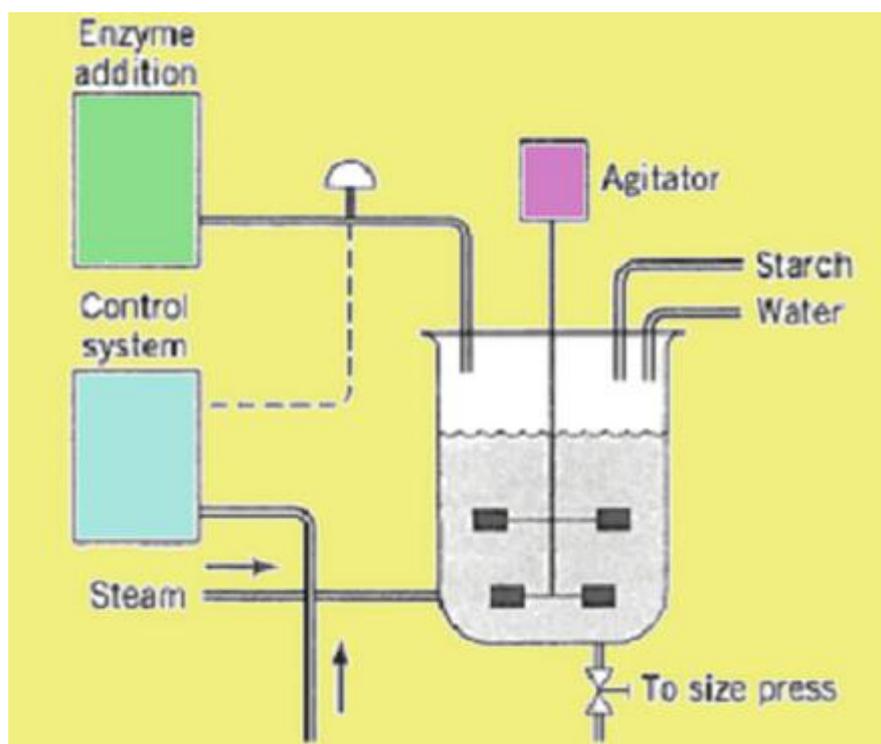
During thermochemical oxidation process unmodified starch is degraded to the wanted viscosity and its homogeneity improves. Modification of starch slurry takes place in jet cooker in the presence of the oxidizing agent. The operating temperature is about 150°C. Addition of steam to the jet cooker has to be performed carefully to achieve proper steam condensation and pasting. Steam addition influences on the retention time in the hold coil after the jet cooker. Retention time has to be constant to achieve uniform quality of starch. The viscosity of starch is adjusted by changing the amount of soluble oxidant. (Bublitz, Kenneth, Craig, 1990)

pH of the oxidizing agent is about 7 or a little higher but typically the starch slurry is acidic. pH has to be adjusted with caustic to the level of 8.0-8.5 when exiting the converter to prevent the precipitation of amylose. The amount of oxidant is based on the starch weight and is described here in percentages. The oxidant level is typically between 0.05% and 0.3% but if ammonium persulfate is used the range varies from 0.05% to 0.6%. Besides ammonium persulfate, potassium persulfate, hydrogen peroxide and sodium hypochlorite are used as an oxidizing agent. (Bublitz et al., 1990)

### **5.2 Enzymatic modification**

Degradation of starch can be executed enzymatically. The most common enzymes used for conversion of starch are amylases that are found from many sources such as plants, microorganisms and animals. Alpha amylase is typically used in the conversion process of starch. During hydrolyzing of starch,  $\alpha$ -D-(1,4) glucosidic bonds are broken randomly.  $\alpha$ -D-(1,4) (1,6) glucosyl oligosaccharides and maltose are formed as depolymerization products. Enzyme cannot hydrolyze  $\alpha$ -D-(1,6) bonds in amylopectin so limiting dextrans are left in the product. The optimum pH area for functioning of alpha-amylase is between 6.3 and 6.8. Inactivation of enzymes can be executed with pH below 4 or above 9. The required amount of enzyme is 0.1-0.2% inactivated agent that is based on the starch amount. Enzymes are often sold to the factory based on the potency or strength of enzymatic activity per unit weight/unit volume. Potency needs to be taken carefully into consideration because the same amount of enzymes with different potencies bring different enzyme activity to the process. (Bajpai, 2012)

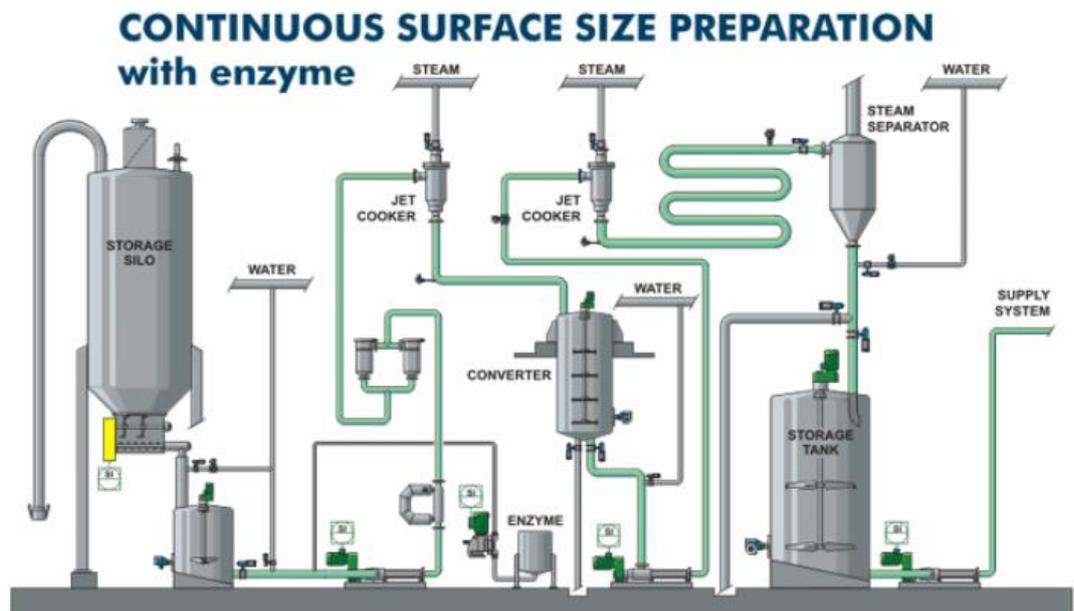
Enzymatic starch modification can be executed in two types of reactor systems: batch and continuous systems. In batch system (Figure 20) native starch is dispersed in water in a cooker. Cooker has an agitator inside which makes sure that slurry is mixed continuously. Heating of the cooker is started when enzyme is added to the cooker. Temperature is kept in 70-80°C for 10 to 15 minutes for gelatinization reaction to occur. When reaction time has passed more steam is released to achieve temperature (95°C) which deactivates the enzymes. High temperature is kept in a cooker about 5-7 minutes to deactivate the enzymes. When enzymes have been deactivated dilution water is added to ensure that the required solid concentration level is maintained. Solution is cooled down to 60°C and the needed solids content level is adjusted. Cooled and diluted starch solution is lead to storage tank. From storage tank starch goes through a vibratory screen to service tank where it is ready to be pumped into the size press. The temperature of the pumped slurry is 45-50°C. (Bajpai, 2012; Lovin, Wheeler, 1990)



**Figure 20** Principle picture of batch conversion system. (Tolan, 2002)

The continuous systems are typically used in larger starch users. In Figure 21 modern continuous enzymatic degradation process is presented. Enzymatic degradation process consists storage tank for native starch, small storage tank for enzyme, two jet cookers, converter, steam separator and storage tank for the

processed starch. Viscosity of the starch is adjusted with the residence time of the converter (converting time) and the amount of enzyme. The enzymatic cooking process starts when water-diluted starch and enzymes are combined and pumped into the first jet cooker where enzymes start to degrade the starch. This phase is called pre-cook and the temperature in cooker is between 70°C and 90°C. From the pre-cook, solution continues to the converter where enzymes continue to degrade the starch. Degradation of starch decreases the starch viscosity. Solution stays 10-20 minutes in the converter and then it continues to post-cook to the second jet cooker. Enzymes are deactivated with a high temperature (120-140°C) during post-cook. The temperature is kept high in the pipeline after the jet cooker because deactivation of enzymes has to be ensured. When the starch solution arrives to the steam separator all the enzymes should be deactivated. After steam separation, starch is diluted with water to wanted solids content and then it goes to storage tank where it is ready to take in use. (Lovin, Wheeler, 1990; Bajpai, 2012)



**Figure 21** Continuous enzymatic degradation process of starch. (VTT, 2015)

## EXPERIMENTAL PART

Experimental part of thesis includes a short introduction of Ingerois Board Mill and the board machine. Production process of folding boxboard is described from the drying section to the reeling as the parameters of wet end are not included to

the thesis. The new INVO Coater and the start-up of the new surface sizing process is described more detailed as their own chapters.

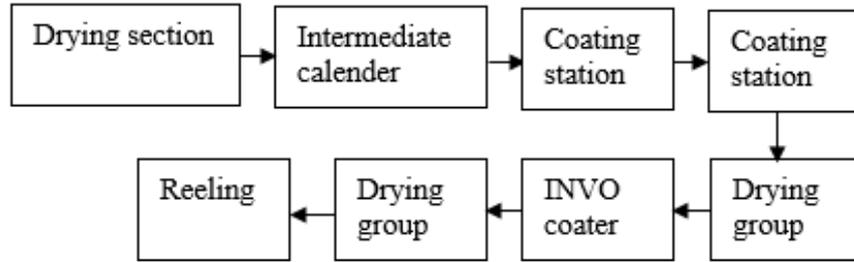
Trials are divided into three sections: Enzymatic cooking process, Properties of the surface size starch, Drying and Curl. The trials of the first section consider the optimization of the enzymatic cooking process parameters. In the second section, the effect of starch properties on quality of board is determined. Third part determines how the moisturizing unit and two drying groups influence on the curl of surface sized board. In addition, the drying requirement of the last drying group was determined as the moisturizing unit was taken out of use.

## **6 INGEROIS BOARD MILL**

Ingerois board machine 4 produces two different brands of folding boxboard named Tamfold and Tambrite. Produced folding boxboard is used in consumer packaging such as pharmaceuticals and food. Tamfold and Tambrite differ with the amount of coating layers. Tamfold has one coating layer on top side and Tambrite has two. Both brands are uncoated from the backside. The basis weight of the product varies between 190-335 g/m<sup>2</sup> (top and back layers 20-40 g/m<sup>2</sup> and middle layer 110-290 g/m<sup>2</sup>). Produced folding boxboard consists of different pulps in three layers. Top and back layers are consisted of bleached chemical pulp. The middle layer is consisted of broke and bleached groundwood pulp. (Stora Enso)

### **6.1 Production process from the drying section to the reeling**

Considering this thesis, it is important to describe the process from the drying section to the reeling of the produced paperboard (Figure 22). The drying section consists of six drying groups before intermediate calendering and two drying groups after the calendering. One drying group is placed after the first two coating stations and the last one is placed after the third coating station. Intermediate calendering smoothens the surface of the board and by that uniform coating layer is easier to achieve.

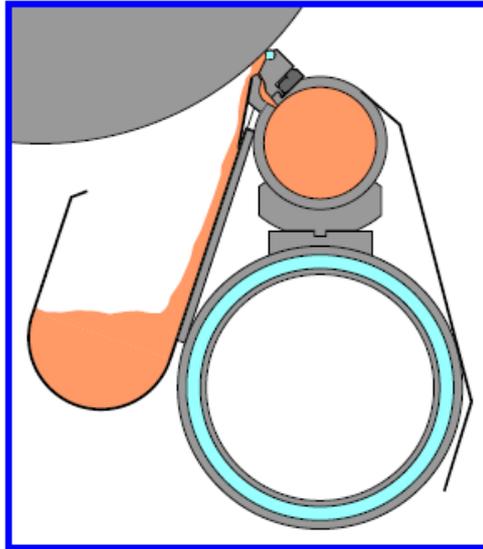


**Figure 22** Block diagram of the production from drying section to the reeling.

From calendering the web continues to the coating stations. Process consists of three coating stations in which two first ones are used for coating of top side of the board. In both of these coating stations blade application technology is used. The third coating station (INVO coater) is installed during this project on January and it is used for surface sizing of the backside of the board. The third coating station (INVO coater) is described more detailed in the next chapter 6.2. Drying of coating and size are carried out with gas IR dryers and air-dryers. From the coating section web continues to the ninth drying group and then to the reeling.

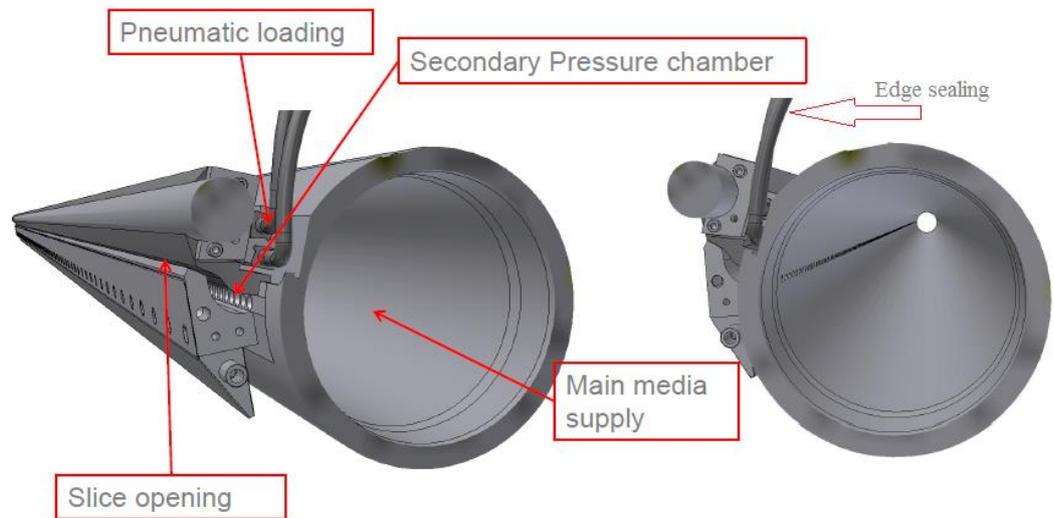
## 6.2 UMV INVO Coater

The INVO Coater (Figure 23) is a jet coating applicator in which two different metering elements, rod and INVO Tip, can be utilized. Smooth rod is used as a metering element during surface sizing. The application and metering of size is executed with short dwell technique that provides a good lubrication in the metering area. INVO coater is consisted of backing roll, rod, rod bed, edge sealings, applicator pipe, water nozzles, support beam and chamber where the excess size flows.



**Figure 23** Metering of surface size is executed with smooth rod. The excess surface size is returned back to machine circulation. (UMV, 2017)

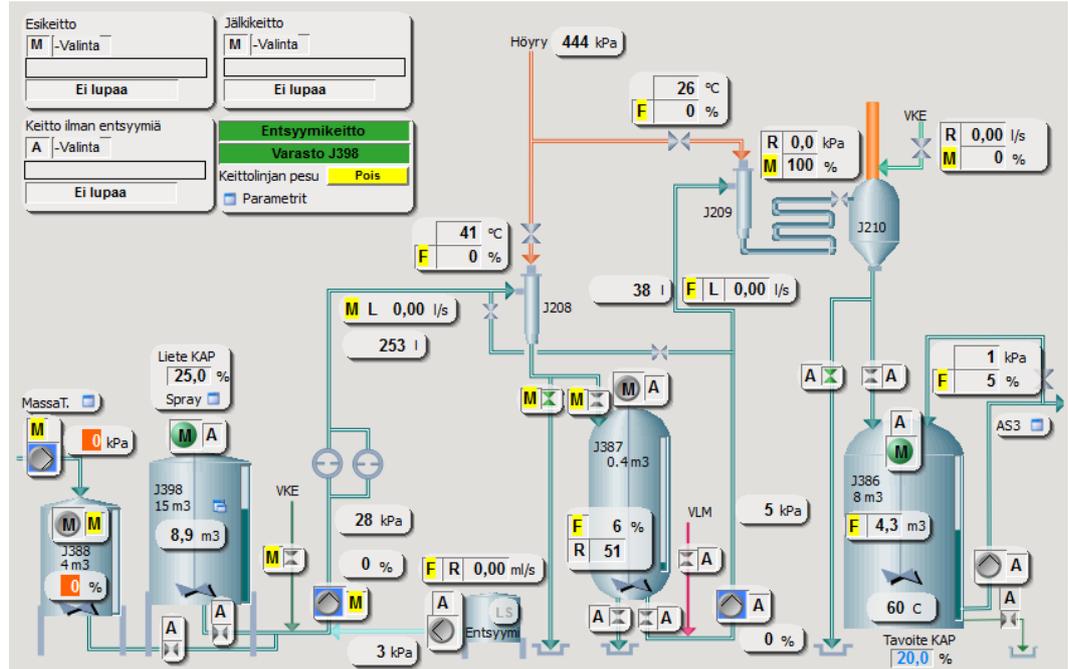
As the surface sizing is started the applicator pipe fills with surface size and the size flows to the small secondary pressure chamber (Figure 24). Surface size flows from the slice opening on to the moving web. After size is applicated to the web it is metered with smooth rod. The excess size flows to the return chamber and from there it returns to the machine circulation. The width of the surface size film is metered with edge sealings that are placed on both sides of the coater. The uncoated edges of the web have to be sprayed with water to prevent breaking of rod and rod bed. Water nozzles are placed on the backside of the backing roll to keep the edges of the roll wet. The parameters considering the rod are the rotation speed and the rod pressure. Rod pressure describes the pressure that rod applies to the web/backing roll. Modification of rod parameters is not included to the experimental part of the thesis but the parameters were modified during normal production.



**Figure 24** Application unit details of INVO Coater. (UMV, 2017)

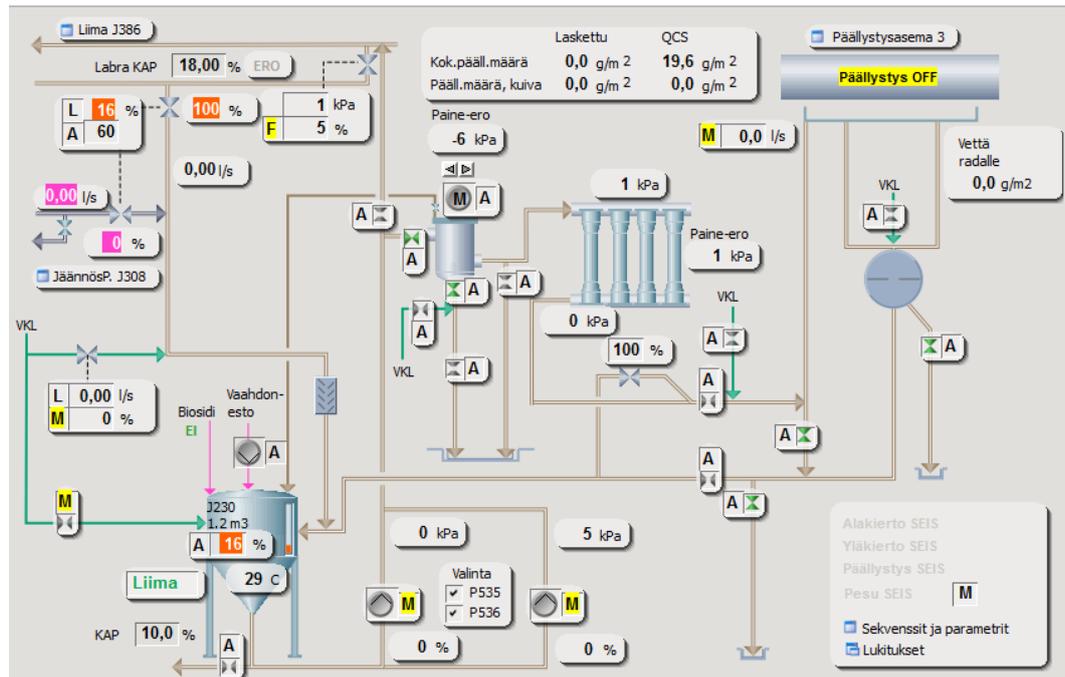
### 6.3 Enzymatic cooking process

Enzymatic cooking process consists of starch sludge tank, two jet cookers, converter, steam separator, storage tank of enzyme and storage tank of cooked starch. The cooking process (Figure 25) starts from the storage tank of starch sludge (J398). Starch sludge is diluted into wanted solids content (20% -30%) and the diluted sludge is led to the line where it is united with enzyme. The mix of enzyme and starch continues to the first jet cooker (J208) where the cooking is started in temperature of 90°C. Solution continues to the converter where the cooking continues. Temperature is kept high in the converter. The time in converter (J387) is one of the parameters that influences on the viscosity of the starch. The longer the starch stays in the converter the lower the resulting viscosity is. Converting time varies usually between 10 and 20 minutes depending on the starch and the wanted viscosity. When the converting time is passed starch continues to the second jet cooker (J209) where the enzyme is deactivated by rising the temperature up to 130°C. The temperature is kept high in the pipeline that leads to steam recovery (J210) to make sure the enzyme is properly deactivated. After steam recovery the starch flows to the storage tank (J386). Dilution water is led to the pipeline that leads to the storage tank from the steam separation.



**Figure 25** The process picture of the enzymatic cooking process.

From the storage tank, starch is directed to the machine tank (J230) where it is diluted to its final solids content. Antifoaming agent is fed continuously to the size from the installed pipeline to the machine tank. Antifoaming agent prevents foaming of the size. The excess microbial growth is controlled by adding biocide to the machine tank where it is mixed to the starch. The additions of antifoaming agent and biocide are assumed to have no effect on trial results. The process picture of the machine circulation is presented in Figure 26. Surface size is directed to the coater through screening unit. The amount of surface size and water are calculated based on the size flows between storage tank and machine tank. Calculation considers also the set solids content of starch in the machine tank.



**Figure 26** The process picture of the machine circulation.

## 7 START-UP

This chapter describes not only the start-up day but also the few weeks period when the coater was monitored more carefully and the parameters of the enzymatic cooking process and starch were set. Start-up plan considering surface size was to apply  $0.5 \text{ g/m}^2$  of dry starch to the back layer of the paperboard. The solids content of surface size was adjusted low to make sure that enough water was applied to back side of the web. The board has a tendency to be straightened or to be curled towards the top layer if there is not enough water applied to the back layer. The solids content of starch was adjusted to 5% with two dilution water lines.

Level of the size viscosity after the cooking process was hard to predict with the low starch solids content. It could be predicted that viscosity would be much lower than in the pre-trials (Chapter 8.1) because the starch solution was diluted to significantly lower solids content after the cooking. Due to this prediction, viscosity was observed during the start-up in case of unexpected rise in its level but if problems did not occur the parameters of the cooking process were kept constant. Viscosity of the starch was modified after the start-up in controlled trials.

Start-up plan with the enzymatic cooking process was to start with defined parameters and to keep them constant during the start-up if there were no problems. Parameter values of the enzymatic cooking process were chosen based on the pre-trials: enzyme dose 2 kg/t and converting time 20 minutes. With these parameters the resulting starch viscosity varied between 20 mPas to 30 mPas that was, as predicted, much lower than in the pre-trials.

The solids content was raised quite quickly from 5% to 8% and finally to 10%. The amount of starch on the back layer rose from 0.5 g/m<sup>2</sup> to 0.8 g/m<sup>2</sup>. The solids content of starch was not set higher for continuous production until it had been tested with short trials that the bigger amount of starch did not cause any problems when it got to the client. The viscosity of starch rose to 30-45 mPas when solids content was increased to 10%.

## **8 THE ENZYMATIC COOKING**

The most important process parameters of surface sizing agent are viscosity and solids content. They influence the most on the quality of the size film and web runnability at coating station. Viscosity of the surface size is modified with enzymatic cooking process parameters, converting time and enzyme dose. The results of the all trials considering enzymatic cooking can be found in Appendices I and II.

Viscosity of all trial samples was measured with Brookfield viscometer. The measurement equipment consist of the viscometer and spindle. Spindle is chosen based on the nature of the sample. Spindle is attached to viscometer and then dipped into the sample solution. The motor of viscometer rotates the spindle with a speed of 10 rpm. Viscosity is calculated based on the resistance to rotation. Temperature of the samples influences significantly on their viscosity level so the temperature of the compared samples was the same during measurement.

### **8.1 Pre-trials**

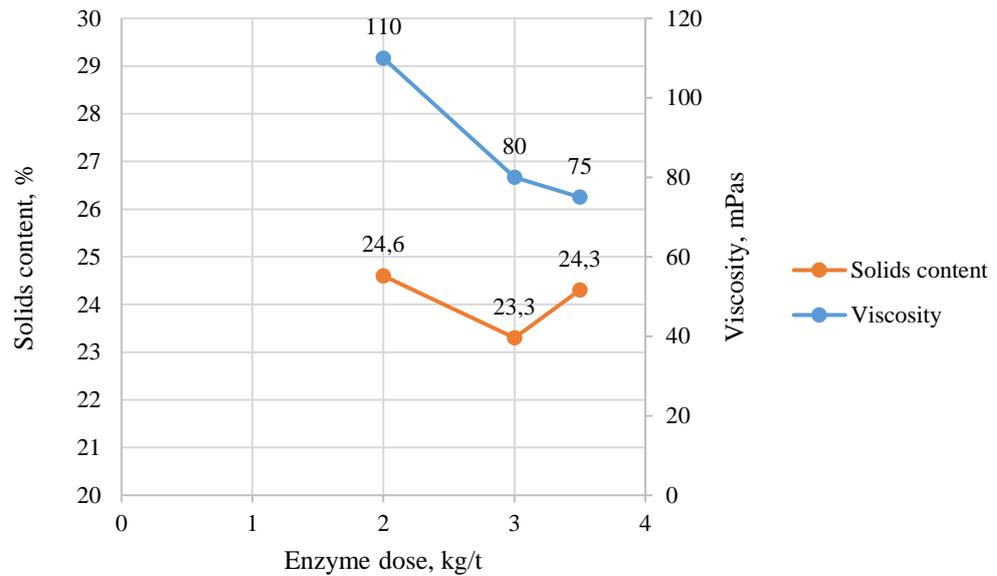
In the pre-trial effect of the enzyme dose and converting time were investigated when the solids content of starch sludge (before cooking) was over 30%. Enzyme activity was kept constant in trial. Pre-trials were executed before the installation of the coater to make sure that the enzymatic cooking process worked as planned. Aim of the trials was to gain knowledge of the influence of cooking parameters on

the starch quality. The parameters of the cooking process for the start-up was determined based on pre-trials. In Table IV measurement points of the pre-trials are described.

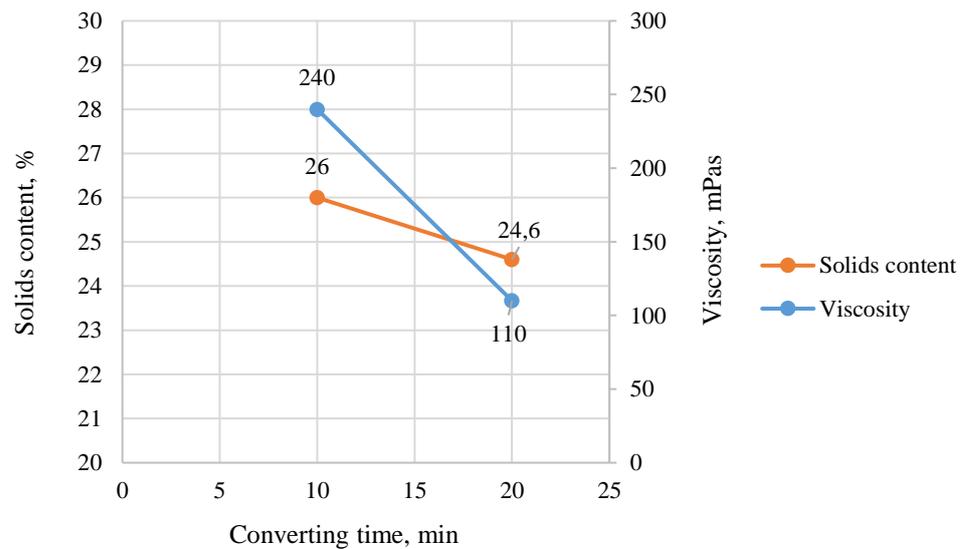
**Table IV** In the pre-trials effect of enzymatic cooking process were investigated.

Measurement point	Enzyme dose, kg/t	Converting time, min
1	3.5	20
2	3.0	20
3	2.0	20
4	2.0	10
5	1.5	10
6	2.0	15
7	2.0	20

In Figures 27 and 28 can be seen that the effect of the variables did not have any significant influence on the solids content of starch paste. Solids content of the starch is controlled with dilution water. Instead, both parameters influenced on viscosity of starch. Based on the Figures 27 and 28 viscosity decreased under 100 mPas if the enzyme dose was much over 2 kg/t. The desired viscosity level of starch is 100-150 mPas. Lower viscosity level indicates typically that starch is degraded too much and bonding ability of starch is decreased. Viscosity of starch influence the starch penetration into the board structure. When starch has low viscosity it penetrates into the board structure. To achieve improvement in bending stiffness and surface strength starch has to stay on the surface of the back layer. Based on these results enzyme dose of 3 kg/t or more is too high.



**Figure 27** The effect of the amount of enzyme used in the cooking process. The converting time is 20 minutes.



**Figure 28** The effect of the converting time in the cooking process. The enzyme dose is 2 kg/t.

## 8.2 Effect of the enzyme dose and converting time

In pre-trials (Chapter 8.1) the effect of enzyme dose and converting time on the starch viscosity were tested before the start-up of the new process. Based on the pre-trials the start-up parameters were chosen, enzyme dose of 2 kg/t and converting time 20 minutes. With these parameter values, the final viscosity of the starch in the start-up was 20-30 mPas with starch solids content of 5%. Viscosity was significantly lower compared to pre-trials that was due to the much lower solids content of starch at the start-up (in pre-trials about 25%). After the start-up

enzymatic cooking parameters were studied more to gain knowledge how much enzyme and how long converting time were needed to achieve the desired viscosity level. In general, if there is a possibility to lower the amount of raw materials used in the process the costs of raw materials decrease. It is more beneficial considering raw material costs to lower the enzyme dose than the converting time when the viscosity of the starch has to be increased.

Trial was executed in two days, in the first trial the effect of enzyme dose was tested and in the second trial the converting time was modified. On both days, the samples in which the viscosity and solids content were measured were taken from the pipeline that lead to the storage tank of cooked starch. In this way the starch had not yet been mixed with the starch in the storage tank. The viscosity of storage tank was also monitored during the trial to see if the viscosity level increases too much. The goal was to keep the viscosity of the storage in a range of 100 mPas to 150 mPas. Before the trial, the amount of cooked starch in storage tank was about 5 m<sup>3</sup> and the viscosity of that starch was 70 mPas. The solids content of starch was kept constant during trial (10% in machine tank).

### **Enzyme dose**

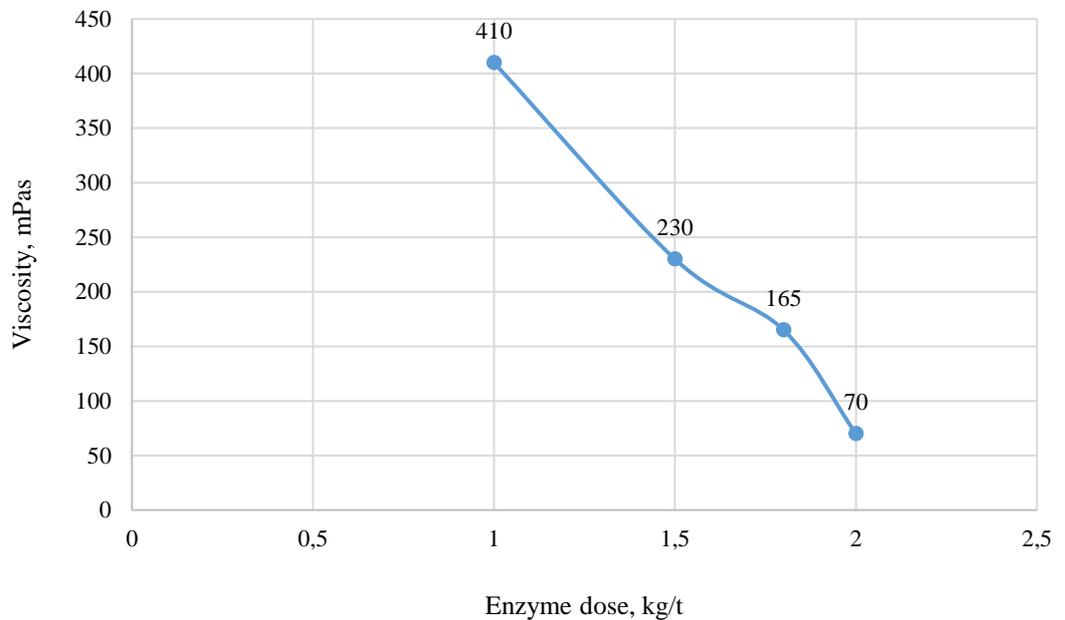
The aim of this trial was to optimize the enzyme dose of the cooking process in a way that the viscosity after cooking set about 100-150 mPas and by that the viscosity in the storage tank stays under 150 mPas. Measurement points of the trial are presented in Table V. The first row of Table V is the reference point that shows the parameters that were used in the enzymatic cooking process before the trial. The converting time was kept constant during the trial.

**Table V** The measurement points and the reference point of the trial.

<b>Measurement point</b>	<b>Enzyme dose, kg/t</b>	<b>Converting time, min</b>
Reference point	2	20
1	1	20
2	1.8	20
3	1.5	20

At the first measurement point enzyme dose was decreased in half which was the minimum amount that supplier of the process recommended to use. In smaller amounts the enzyme is not able to degrade starch enough. As can be seen from Figure 29 viscosity increased to over 400 mPas. Due to big increase of viscosity,

the enzyme dose was raised to 1.8 kg/t at the next measurement point. Although the decrease of enzyme dose at the second measurement point compared to the reference point was only 0.2 kg/t it increased the viscosity almost 100 mPas. Based on the results even a small decrease in enzyme dose has a significant effect on the degradation of starch. As a conclusion, enzyme dose modification can be used when viscosity of the starch has to be changed considerably but for small adjustments it cannot be used as a variable. Viscosity at the storage tank increased from 70 mPas to 120 mPas during the trial. Enzyme dose was decided to keep in 1.8 kg/t after the trial as with that dose the viscosity was after cooking 165 mPas and in the storage tank 120 mPas as desired.



**Figure 29** Enzyme dose was decreased from 2 kg/t to 1 kg/t during trial. The lowest enzyme dose (2 kg/t) is the reference point. Converting time was 20 minutes. The desired viscosity level is 100-150 mPas.

The viscosity in the machine tank was measured by the process workers once in an 8-hour shift. During enzyme dose trial the viscosity rose from 40 mPas to 50 mPas. The rise was only marginal because the solids content of starch was set low and the high amount of dilution water lowered the viscosity.

### Converting time

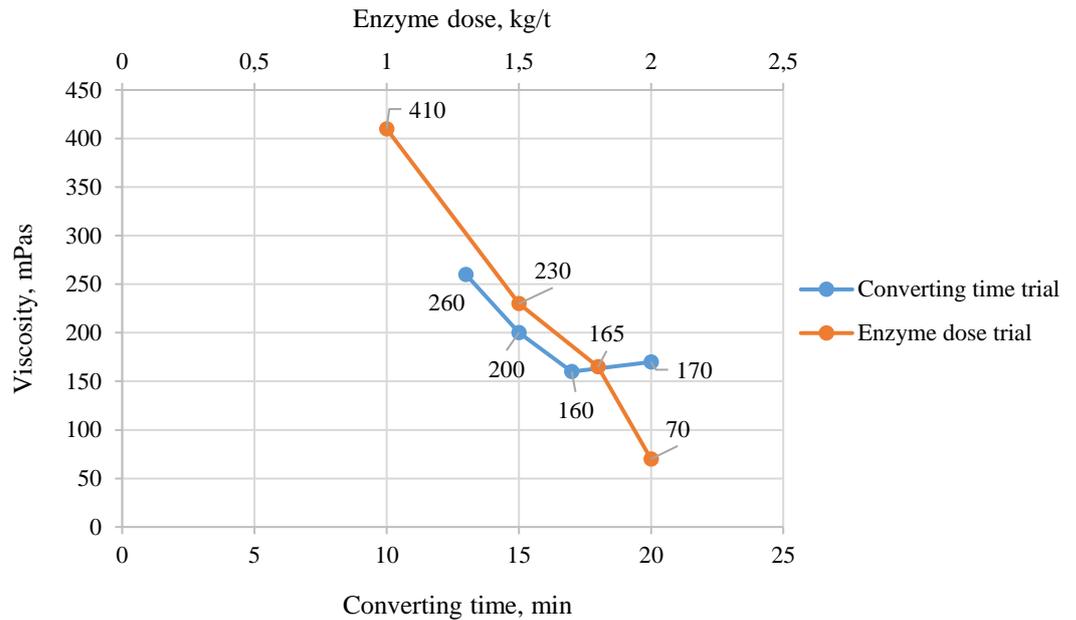
The aim of this trial was to optimize the converting time of the enzymatic cooking process and to determine how much does the changes in converting time influence on the viscosity of starch. The minimum converting time supplier recommended

to use was 10 minutes because in shorter converting times the enzyme has not enough time to degrade starch sufficiently. The aimed viscosity level after cooking was approximately 150 mPas and in the storage tank in a range of 100 mPas to 150 mPas. The measurement points and the reference point of the trial are presented in Table VI. The enzyme dose was kept constant during trial.

**Table VI** Measurement points of the converting time trial.

Measurement point	Converting time, min	Enzyme dose, kg/t
Reference point	20	1.8
1	15	1.8
2	13	1.8
3	17	1.8

Effect of converting time on starch viscosity was much milder compared to the effect of the enzyme dose (Figure 30). Converting time can be used as a variable when viscosity requires only fine adjustment. The viscosity of starch with converting time of 17 minutes is almost the same as with the converting time of 20 minutes. This indicates that enzyme do not degrade the starch during the last three minutes at all. Due to this result there was no reason to increase the converting time back up to 20 minutes again. The converting time was decided to keep in 17 minutes until the viscosity needed to be adjusted. The viscosity of starch in the storage tank increased during the trial from 120 mPas to 150 mPas and in the machine tank from 55 mPas to 57 mPas.



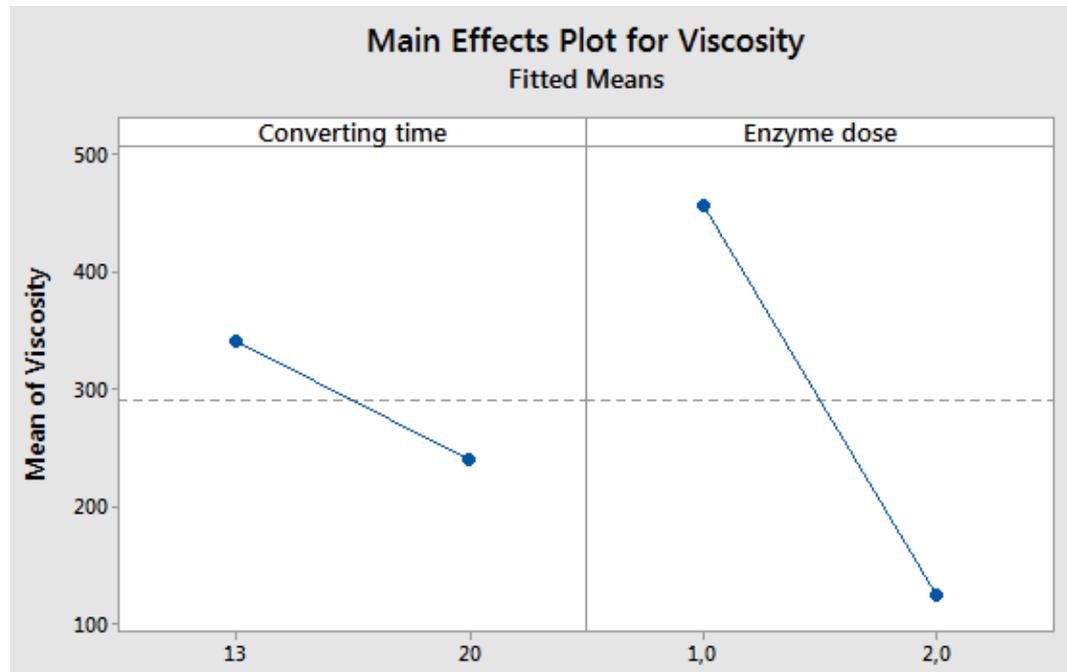
**Figure 30** The results of enzyme dose trial and converting time trial. Converting time during enzyme dose trial was 20 minutes and enzyme dose during converting trial was 1.8 kg/t.

### Analyzing results with Minitab Statistical Software

The results of the enzyme dose and converting time trial were analyzed by using Minitab analyzing tool. Minitab Statistical Software is a statistical software package that can be used for wide data analyzing. Minitab Statistical Software is provided by Minitab Inc. In this chapter two types of analyzes were used: Main effects plot and Cube plot. Main effects plot describes the difference between level means for factors. Main factor can be determined when factors influence on the response differently. The response is graphed for every factor level and they are connected with line. Cube plot presents the relationship between factors. The analysis predicts values for every factor level combination. Minitab analyzes the data with two to eight factors and plots square (with only two factors) or cube model based on the fitted means of factor combinations.

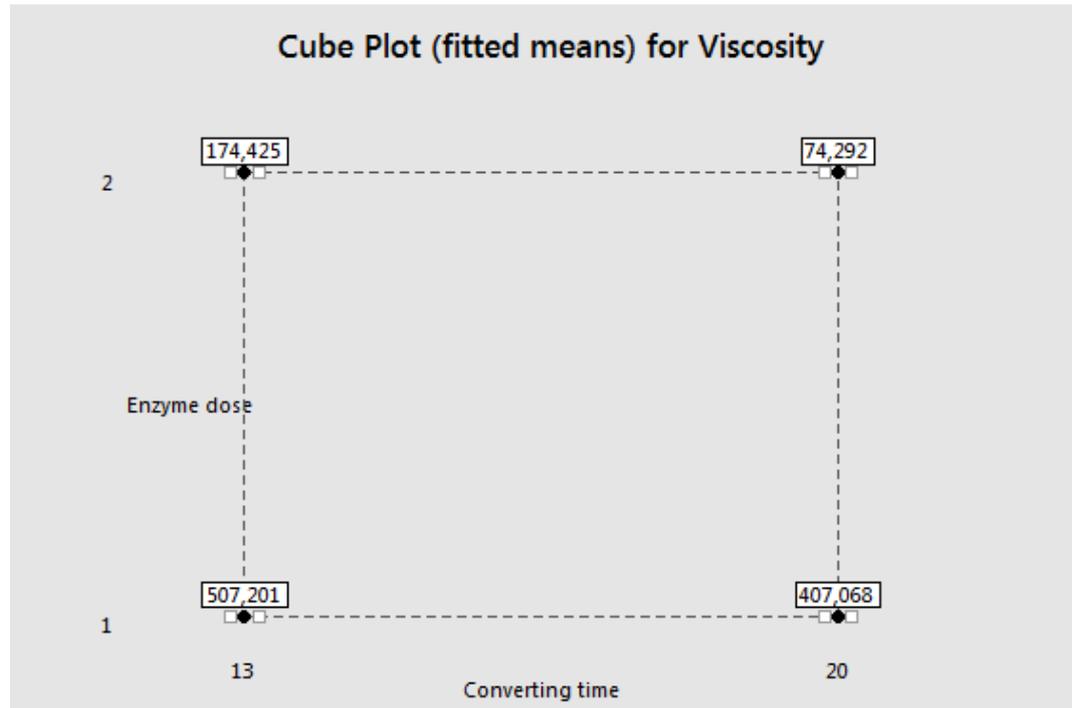
As mentioned in the previous chapter changes in converting time has much smaller effect on viscosity than changes of enzyme dose. This is shown in Figure 31 in which the effect of converting time and enzyme dose on starch viscosity are analyzed by using Main effects plot. The line, which describes the effect of the enzyme dose is much sharper than the one that describes the effect of the

converting time. The sharp line indicates that enzyme dose influences on starch viscosity more effectively than converting time.



**Figure 31** The effect of enzymatic cooking process on the starch viscosity is described with lines. The sharpness of the line indicates how big impact it has on viscosity.

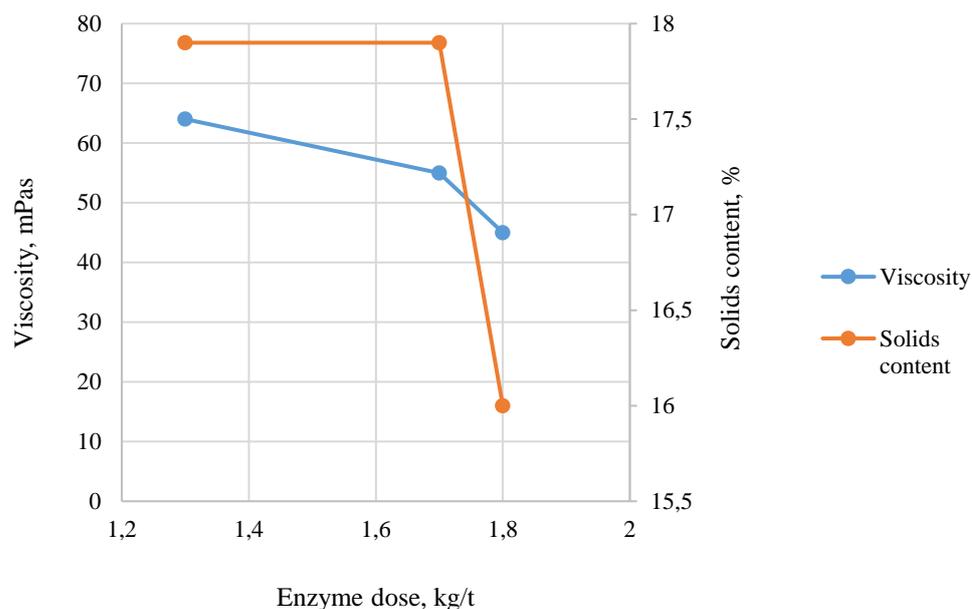
In Figure 32 the fitted means of viscosity based on the trial results are calculated with Minitab and then plotted as a square with parameter values. Because the values in the plot are calculated and fitted to the plot they need to be evaluated as directive values. The Cube plot indicates that if the enzyme dose is at level of 2 kg/t the decrease in converting time does not increase the viscosity much over the desired viscosity level (150 mPas). Based on that decrease of converting time should not cause too high viscosity. Instead, the enzyme dose cannot be decreased in big amounts even when the converting time is 20 minutes because it influences on the viscosity level significantly.



**Figure 32** Cube plot is based on the trial result values that were used in Minitab to calculate the values for the plot.

### 8.3 The change of surface size starch

During trials of thesis the supplier of the surface size starch was changed. The starch “type” was not changed (native wheat starch) but there are some small differences between starches of different suppliers. For example, in a way that starch viscosity reacts to the enzyme during the enzymatic cooking process. For this reason, the enzyme dose had to be optimized again. The optimization was executed during normal production. The changes of the enzyme dose were done in small steps to see the response in viscosity level and to avoid runnability problems in case of too high viscosity. The optimization steps are presented in Figure 33. The samples were taken right after the storage tank before the dilution water line of machine tank. The viscosity of old starch with the enzyme dose of 1.8 kg/t was 120-150 mPas and with the new starch it was 45 mPas. This indicates that the new starch was more sensitive to enzyme than the old one. The viscosity of new starch was increased by lowering the enzyme dose. The converting time was kept in 17 minutes. Converting time is used as a variable when only small adjustments are required. In this case, the fine adjustment was not enough as the viscosity of starch had to be raised to the level of 100-150 mPas.



**Figure 33** Enzyme dose was optimized after the starch change. The new starch was much more sensitive to the enzyme so the enzyme dose of the cooking process had to be lowered. The viscosities of the samples are measured at the same temperature (65 °C).

Running parameters of the enzymatic cooking process were defined and they are presented in Table VII. Due to problems in board production optimization with “New” starch could not be completed during the time limit of thesis. The objective was to achieve viscosity level more than 100 mPas so the optimization of the enzyme dose continues after this thesis by lowering the enzyme dose. As can be seen the running parameters are very different with different starches. Difference is due to different reactivity towards the enzyme. In this case, the enzyme degraded more efficiently the “New” starch and the enzyme dose had to be lowered significantly.

**Table VII** Running parameters of the enzymatic cooking process. Running parameters are defined both to the “Old” starch and to “New” starch.

Starch	Enzyme dose, kg/t	Converting time, min	Viscosity, storage tank, mPas	Solids contents, storage tank, %
“Old” starch	1.8	17	120	18.9
“New” starch	1.3	17	64	18.2

## **8.4 Conclusions**

During the enzymatic cooking process trials the enzyme dose and converting time of the cooking process were optimized. The parameters influence degradation and the viscosity level of starch. As a result of the trials, running parameters of the cooking process were defined. During trials the supplier of the surface size starch changed and the parameters had to be defined again. The optimization of the “New” starch was not completed during the time limit of this thesis due to other problems in the production process. Based on the trial results the viscosity level is primarily adjusted by changing the enzyme dose because its effect on viscosity is more significant than the effect of converting time.

### **The optimized parameters**

The desired viscosity level of surface size in the storage tank was 100-150 mPas with solids content of 18-20%. This level was achieved with “Old” starch with enzyme dose of 1.8 kg/t and converting time of 17 minutes. The enzyme influenced the “New” starch more efficiently and with the optimized parameters of the “Old” starch viscosity level decreased to 45-55 mPas (solids content 17.3%). The viscosity level of the “New” starch was increased by lowering the enzyme dose. With enzyme dose of 1.3 kg/t the viscosity was about 65 mPas which is only about a half what was the aimed viscosity. Increasing of viscosity was continued after this thesis by lowering the enzyme dose.

## **9 EFFECT ON BOARD QUALITY**

The effect of starch properties, solids content and viscosity, on board quality were examined in four trials presented in chapters below. Objective of trials was to find running parameters that provide the wanted quality improvements such as higher bending stiffness or better delamination strength of the board. In addition, curl of the board was monitored during trials. The specific results of the solids content trials and viscosity trial can be found in Appendices III-V.

Wedge process diagnostic tool is used in result analyzing. Wedge is Matlab based online process diagnostic system that collects process data from the manufacturing process. Data can be collected to an analysis from specific period such as time of the trials. (Savcor Oy, 2017)

### 9.1 Solids content

The solids content of starch was set to 10% after the start-up. With 10% solids content and 30-50 mPas viscosity the amount of starch applied to the back layer varied between 0.7 g/m<sup>2</sup> and 0.9 g/m<sup>2</sup>. The amount of starch describes the amount of dry starch that is applied to the web. Higher amounts of starch were run in controlled trials that are described in following sections. Monitoring of starch amount is based on the calculation in the process automation system Damatic DNA. Factors that influence on the calculation are the solids content of starch in machine tank and the starch flow between storage and machine tank. The solids content value that is used in calculation is not measured value; it is a value that process worker sets in DNA and which the system is trying to reach. The starch amount calculation is reliable when the solids content value is kept constant during normal production. When the value is adjusted in trials laboratory measurements have to be done more frequently to have an estimation of the actual starch amount. For example, when the measured solids content of starch is lower than the set value it indicates that the calculated value of starch amount is higher than the actual amount is. The drying parameters of surface size was not changed during the trials as the moisture content of board did not change when the amount of size to the back layer increased.

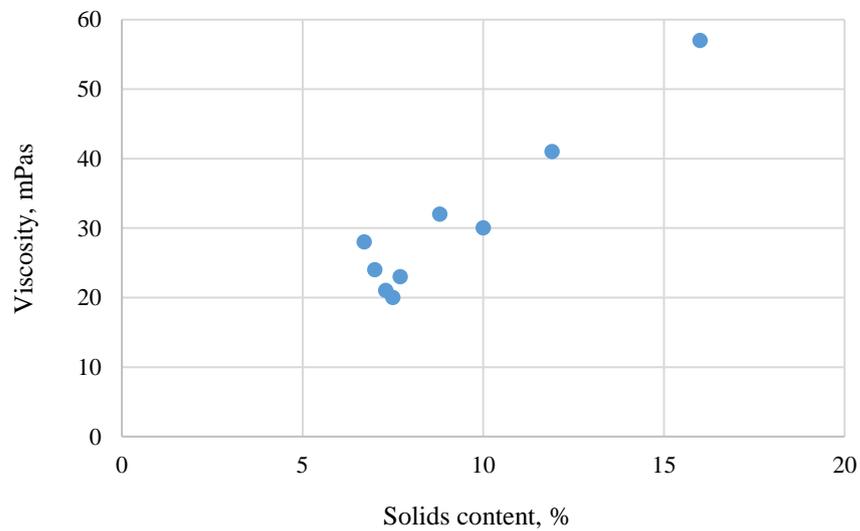
#### First trial

The first trial was ran quite fast after the start-up of the coater. Aim was to gain knowledge how the higher solids content of surface size influence on the surface sizing process and would it require adjustments in the process. The target amount of starch was 1.5-2.0 g/m<sup>2</sup> during the trial. In Table VIII the measurement points of the trial are presented. Solids content values describe the values that are set to the “monitoring system” DNA not the measured values.

**Table VIII** Measurement points of the first solids content trial. Solids content values describes the set values on the DNA not the measured values.

Measurement point	Solids content, machine tank, %	Solids content, storage tank, %
Reference point	8	20
1	10	20
2	13	20
3	15	20

During the first trial it was noticed that there was a lot of foam coming out from the machine tank. Foaming agent was not added to the starch during the first trial. Line for the foaming agent was built after foaming problem occurred but was not ready during the trial. The calculation of the starch amount was not reliable because of the overflow of the size from the machine tank. The calculation is based on the flows between storage tank and machine tank of starch. The calculated starch amount in the highest solids content of starch was  $1.4 \text{ g/m}^2$  but due to foaming it probably was not as high as calculated. Starch flow from the storage tank to machine tank is based on the consumption of surface size. In this foaming “situation” consumption was a sum of the starch which board applied at the coater and the overflow of starch foam from the tank. The starch amount calculation was reliable when the foaming agent was started to fed into the machine tank after the trial. In the next trials the amount could be monitored. The solids content of starch increased during the trial as desired. Viscosity increased almost linearly during increase of solids content (Figure 34). When the trial was finished the solids content was set back to 10% in the machine tank.



**Figure 34** Viscosity and solids content of starch in the machine tank were monitored during trial.

### Second trial

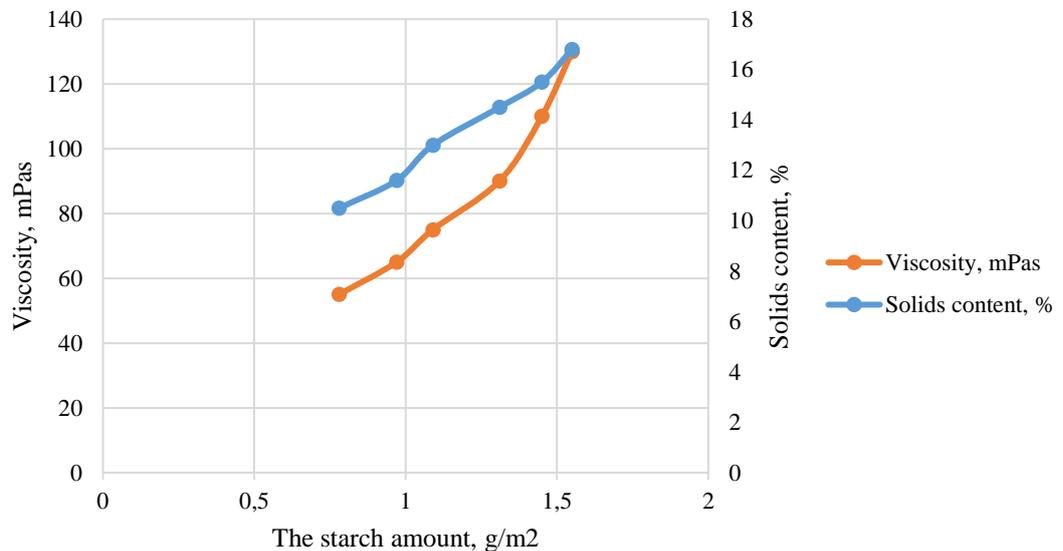
The objective of the second trial was to run similar trial as the first one because the foaming problem during the first trial cause unreliable results of the applied starch amount. The solids content was risen in two steps to the level of 18% in machine tank and the amount of starch was predicted to be about  $1.5\text{-}2.0 \text{ g/m}^2$ .

The solids content in storage tank was kept constant during the trial. The measurement points of the second trial are presented in Table IX. The produced board was double coated Tambrite with basis weight of 240 g/m<sup>2</sup>.

**Table IX** Measurement points of the second solids content trial. Solids content values describes the set values on the DNA not the measured values.

Measurement point	Solids content, machine tank, %	Solids content, storage tank, %
Reference point	10	18
1	14	18
2	18	18

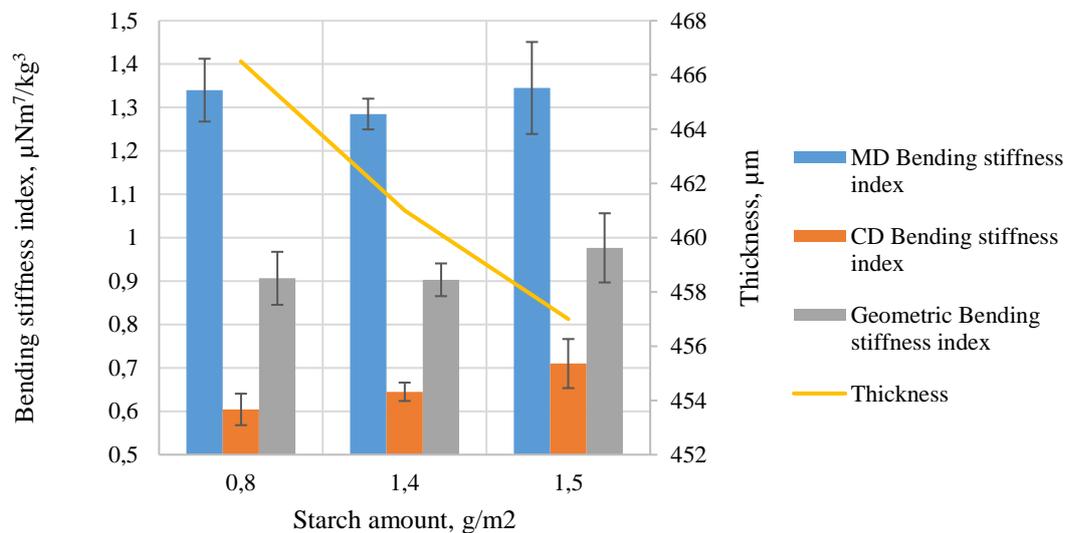
During trial starch amount increased quite linearly with the increase of solids content and viscosity of starch (Figure 35). The amount of water applied to the web increased also but less than 20% (8.5 g/m<sup>2</sup> to 10 g/m<sup>2</sup>) as the starch amount doubled during the trial. The viscosity of starch increased during trial due to lower amount of dilution water to the machine tank. The parameters of enzymatic cooking process were kept constant.



**Figure 35** The target solids content of starch was 18% during trial. The process parameters of enzymatic cooking process were kept constant.

One of the observed quality properties of the board during the trial was bending stiffness. Bending stiffness of the board is viewed as bending stiffness index in this thesis. Bending stiffness index is calculated by dividing bending stiffness with third power of basis weight. Bending stiffness index values of boards with different basis weight are comparable. In Figure 36 the averages of MD, CD and

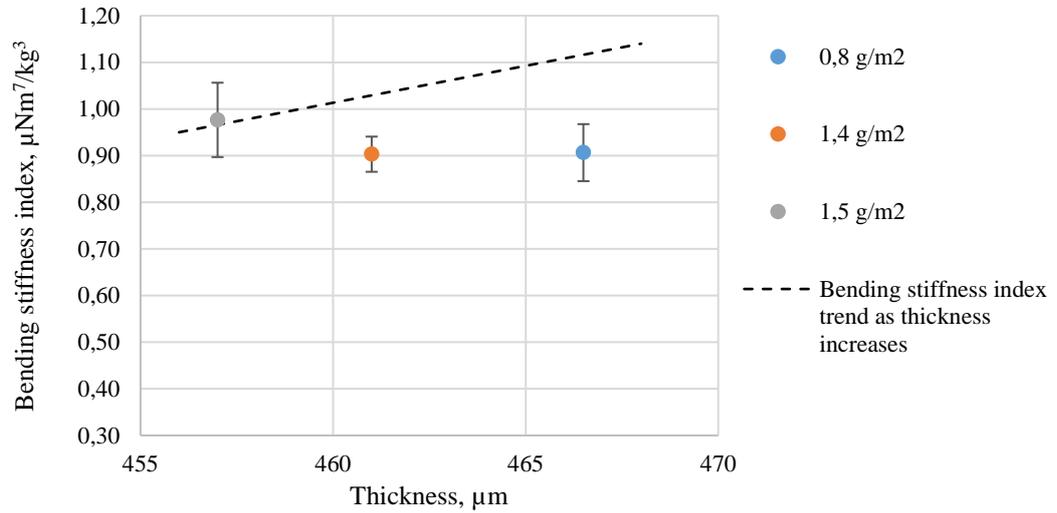
geometrical bending stiffness index of samples are presented in three points in which the amount of starch is different. The CD bending stiffness index increased when starch amount increased but MD bending stiffness index remained almost the same. Geometrical bending stiffness is calculated with Equation 8 (Chapter 3.2 Bending stiffness) and it presents the resultant of the CD and MD bending stiffness. Geometrical bending stiffness index increased about 8% during trial. In addition, the deviations of results in each measurement point are presented. Standard deviations in trial points were high (4-8%) especially in high starch amounts. Normal deviations of bending stiffness indexes are 4-5%. Normal deviations are calculated based on the data of one-year period of bending stiffness measurements (Wedge).



**Figure 36** Bending stiffness index as machine directional, cross directional and geometrical bending stiffness index. Black lines describe standard deviation of the results.

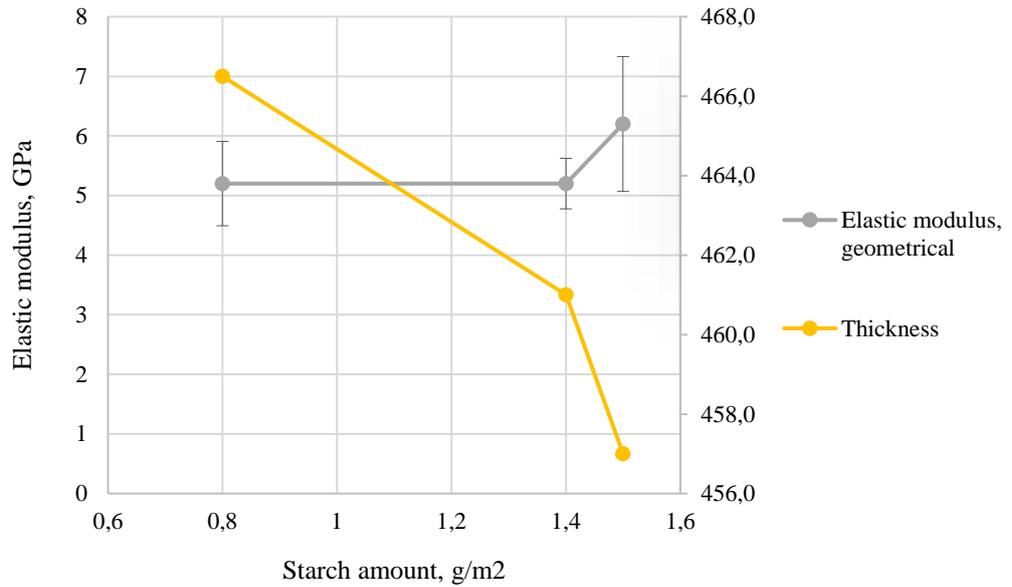
Thickness decreased more than 10  $\mu\text{m}$  during the trial. Decrease of thickness influences the bending stiffness index significantly as bending stiffness is dependent on the third power of thickness. Geometrical bending stiffness index values during trial are presented as a function of thickness in Figure 37. There are variation in both bending stiffness index and thickness of the board samples. The highest bending stiffness index is achieved with the highest amount of starch although the thickness is the lowest of trial in that point. Based on Figure 37 bending stiffness index increases due to higher starch amount not thickness. Could

be predicted that if the thickness had been constant during trial bending stiffness index would have increased more in the measurement point of the highest starch amount. Trend line in Figure 37 describes how bending stiffness index should increase when thickness increases over 10  $\mu\text{m}$ .



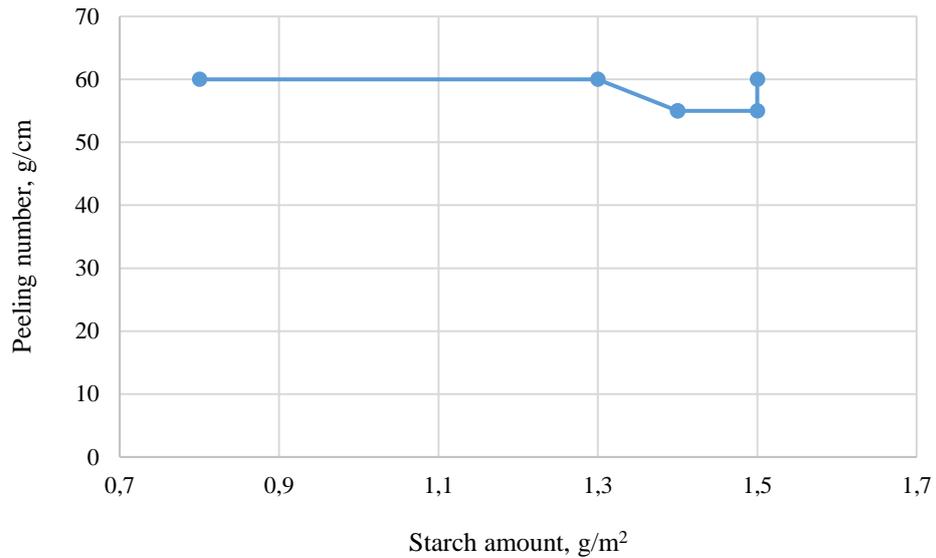
**Figure 37** Bending stiffness index is presented as a function of thickness to see the relation between the bending stiffness index improvement and thickness. Trend line describes how bending stiffness index should theoretically increase when thickness increases.

Based on theoretical knowledge surface sizing influences elastic modulus of the layer it is applied on. Bending stiffness increase is predicted to be a cause of the increased elastic modulus of the back layer. Elastic modulus of the back layer is estimated with calculation model that is based on connection between bending stiffness and elastic modulus, and on 8-month period of quality measurements at Ingerois mill. Elastic modulus of back layer of the board is iterated based on measured bending stiffness of trial samples. Geometrical elastic modulus in different starch amounts are presented in Figure 38. Geometrical elastic modulus increased 19% when the low starch amount (0.8 g/m<sup>2</sup>) and the high starch amount (1.5 g/m<sup>2</sup>) samples are compared. Deviations of results are significant due to deviation of bending stiffness results.



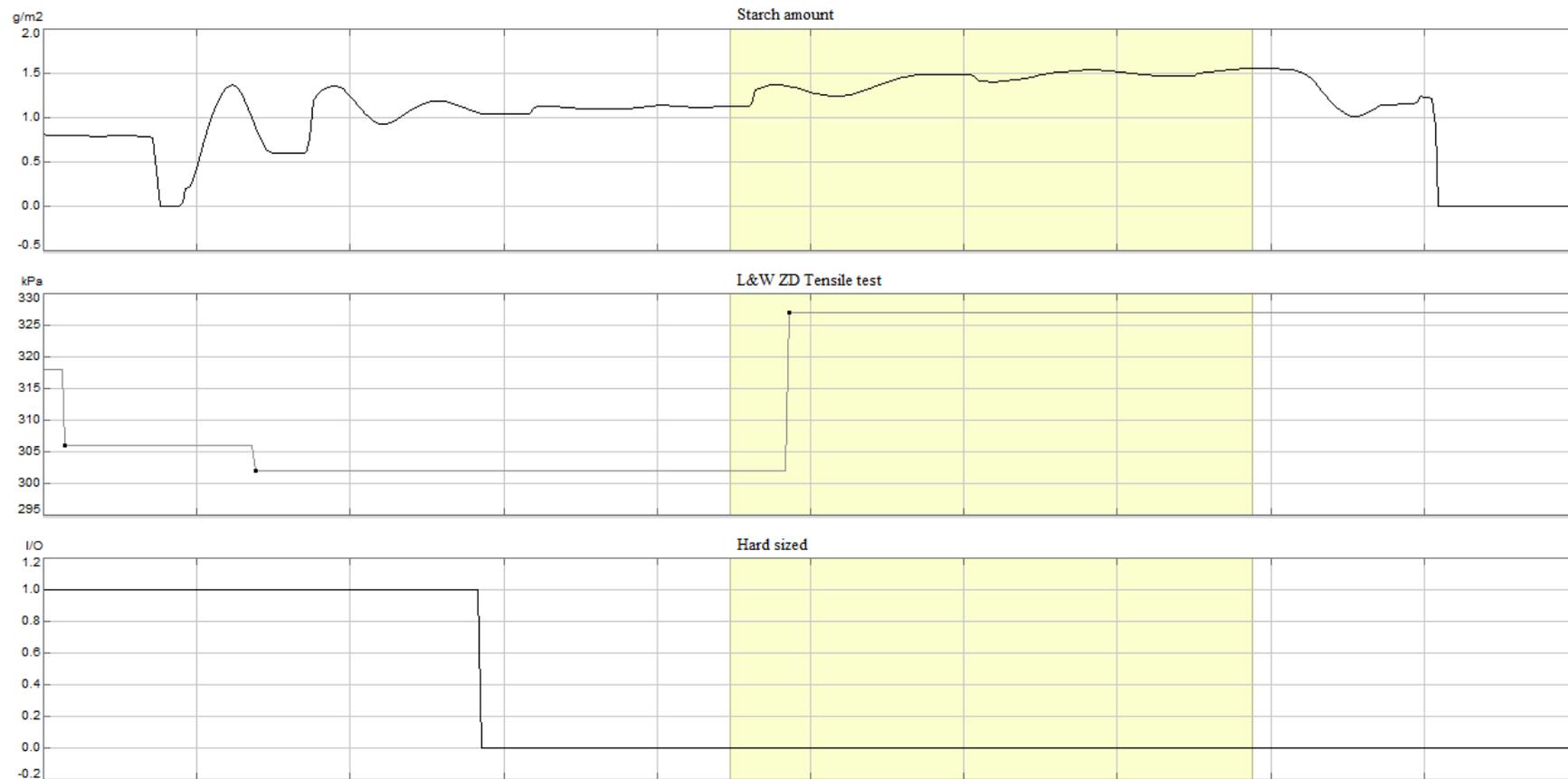
**Figure 38** Geometrical elastic modulus is estimated with calculation model. Standard deviations of results are presented with black lines.

In Figure 39 and 40 the trends of delamination strength are presented during the trial. Delamination strength is measured in two different ways: “Peeling test” and L&W ZD Tensile test. Peeling test describes the delamination strength between back and middle layer as the measurement was executed to the back layer of the board. Peeling test values are presented in Figure 39. Delamination strength of the board did not improve during trial according to Peeling test results. This was predicted because the starch viscosity increased during trial and starch penetration into the board structure typically decreases when the viscosity of starch increases.



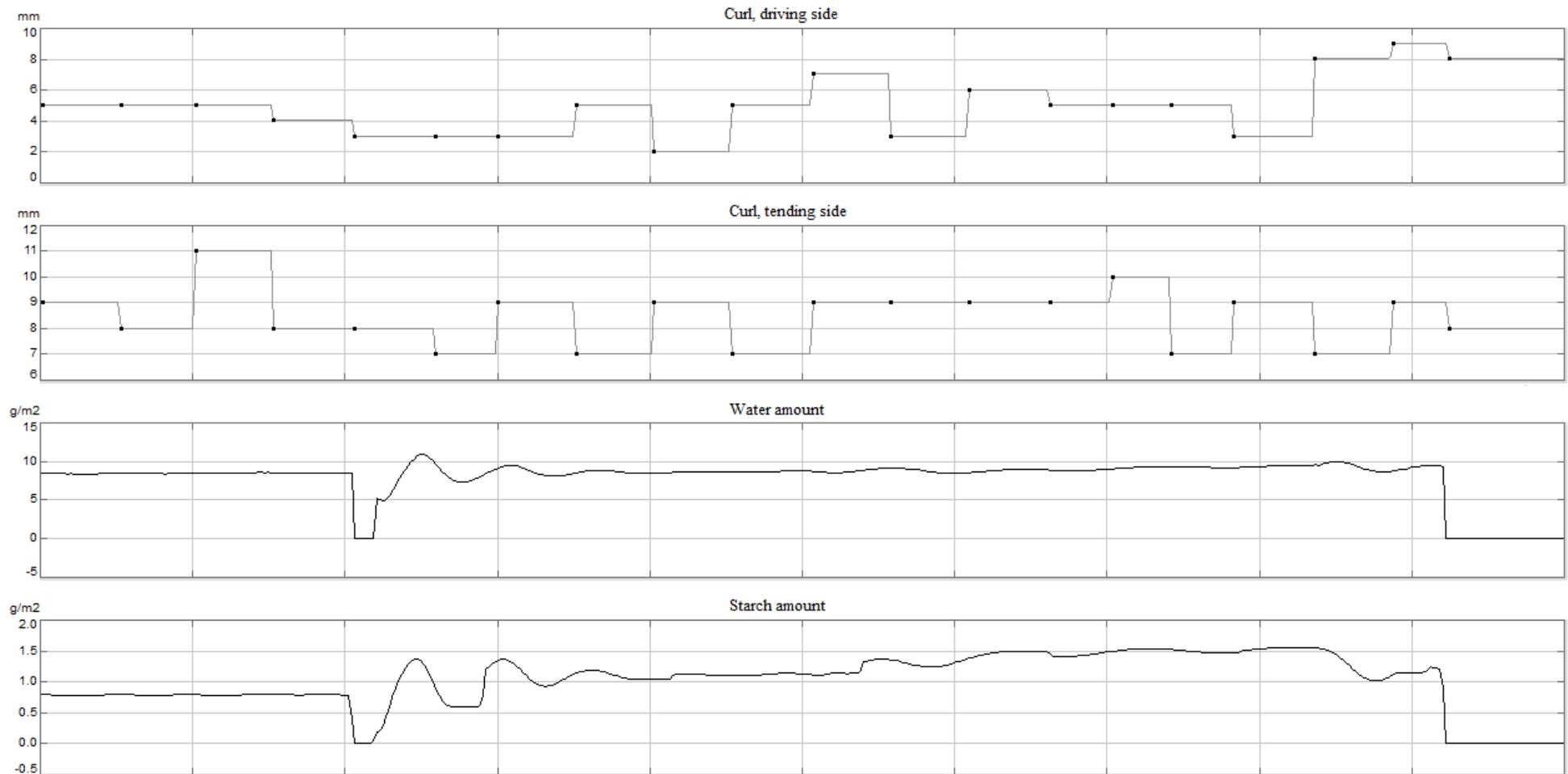
**Figure 39** The effect of high starch amount on delamination strength is estimated based on the "Peeling test".

Delamination strength was also measured with L&W ZD Tensile Tester. The L&W ZD Tensile test results indicate increase of delamination strength during trial that is contrary to the results of Peeling test (Figure 39). Factor which influence delamination strength of the board is internal sizing. Typically, internal size decreases delamination strength of the board. At the start of the trial, the "hard sized" brand was run to the customer that means increased addition of internal size ASA during production of that brand. In Figure 40, can be seen that increased addition of ASA ("Hard sized") is stopped in the early part of the trial. It takes few hours before excess ASA is totally removed from the process water. The increase of delamination strength (L&W ZD Tensile test) during the trial sets to the same time as could be predicted that the effect of increased addition of ASA has ended. The increase of delamination strength is more probably a consequence of removal of excess ASA from the process water than the increased starch amount.



**Figure 40** Print of Wedge data. Delamination strength was measured with L&W ZD Tensile Tester two times during trial (black dots of line). The time interval in the Figure is seven hours. The “Hard sized” trend describes the excess addition of ASA. Yellow “area” describes the time when the starch amount calculation was accurately more than 1 g/m<sup>2</sup>.

As can be seen in Figure 41 the increased solids content of starch and a little higher amount of water applied to the web did not influence on cross directional curl of the board. Curl of the board was not controlled with pressure difference of drying cylinders. Based on trial results controlling of curl is independent of the solids content of the starch if the water applied to the back layer is not lowered. The water amount that web absorbs during surface sizing has been at least  $8 \text{ g/m}^2$  during the process. Curl of the board has to be controlled other ways such as pressure difference of drying cylinders.



**Figure 41** Print of Wedge data. Cross directional curl of the board during the trial. Time interval in Figure is 8 hours.

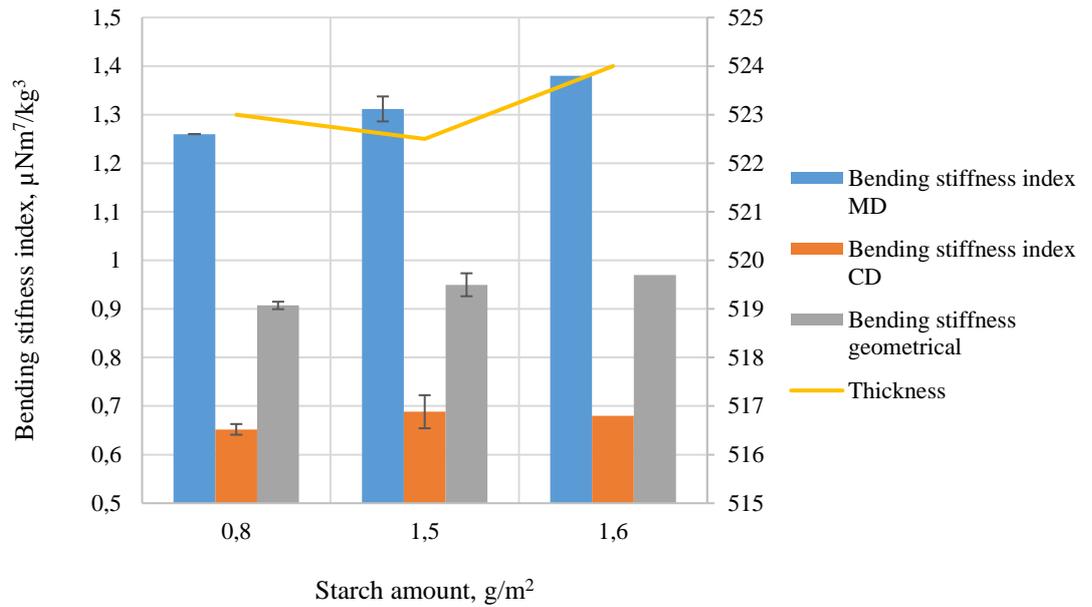
### Third trial

The objective of the trial was to see if the results of third trial was similar compared to the second trial. The enzymatic cooking process parameters were kept constant during the trial so the viscosity changed only due to the lower amount of dilution water as the solids content was increased. The produced board was double coated Tambrite with basis weight of 270 g/m<sup>2</sup>. The measurement points of the third trial are presented in Table X. In the earlier two trials the solids content was increased in steps but in third trial the solids content was set to the maximum point right at the start. In this way the wanted solids content was achieved faster.

**Table X** The measurement points of the third trial.

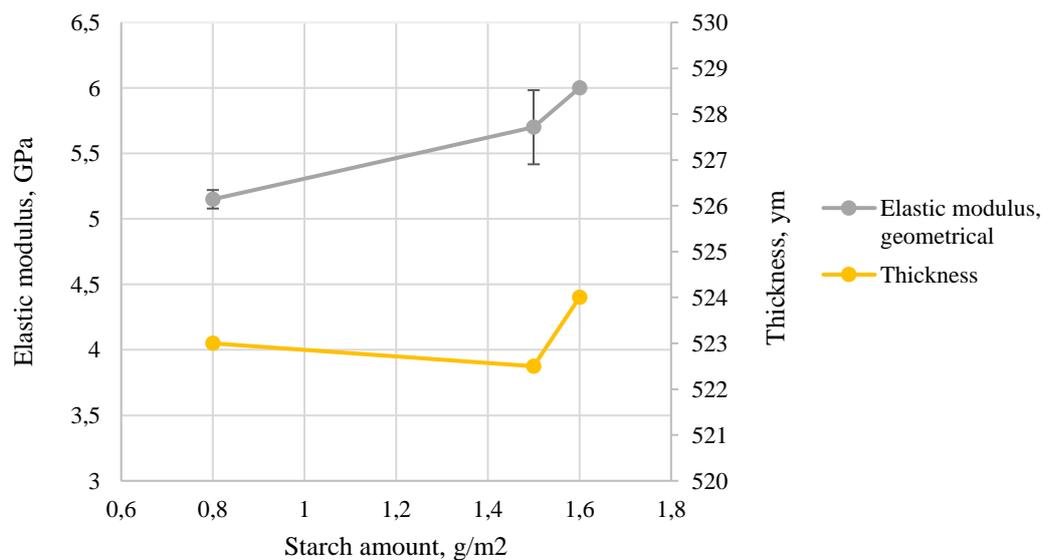
Measurement point	Solids content, machine tank, %	Solids content, storage tank, %
Reference point	10	18
1	18	18

Effect of the increased starch amount on bending stiffness is determined by calculating bending stiffness index to make the results comparable with other trials where different basis weight board was produced. Bending stiffness index increased in both cross direction and machine direction more than 6% (Figure 42). Variation of thickness was very small between measurement points so it did not cause big variation to the bending stiffness index results as in the second trial. As the thickness has not increased significantly during the trial can be assumed that the improvement in bending stiffness is caused by the increased starch amount. Standard deviations of bending stiffness results were 2-5%, which is the normal range for bending stiffness values at Ingerois mill.



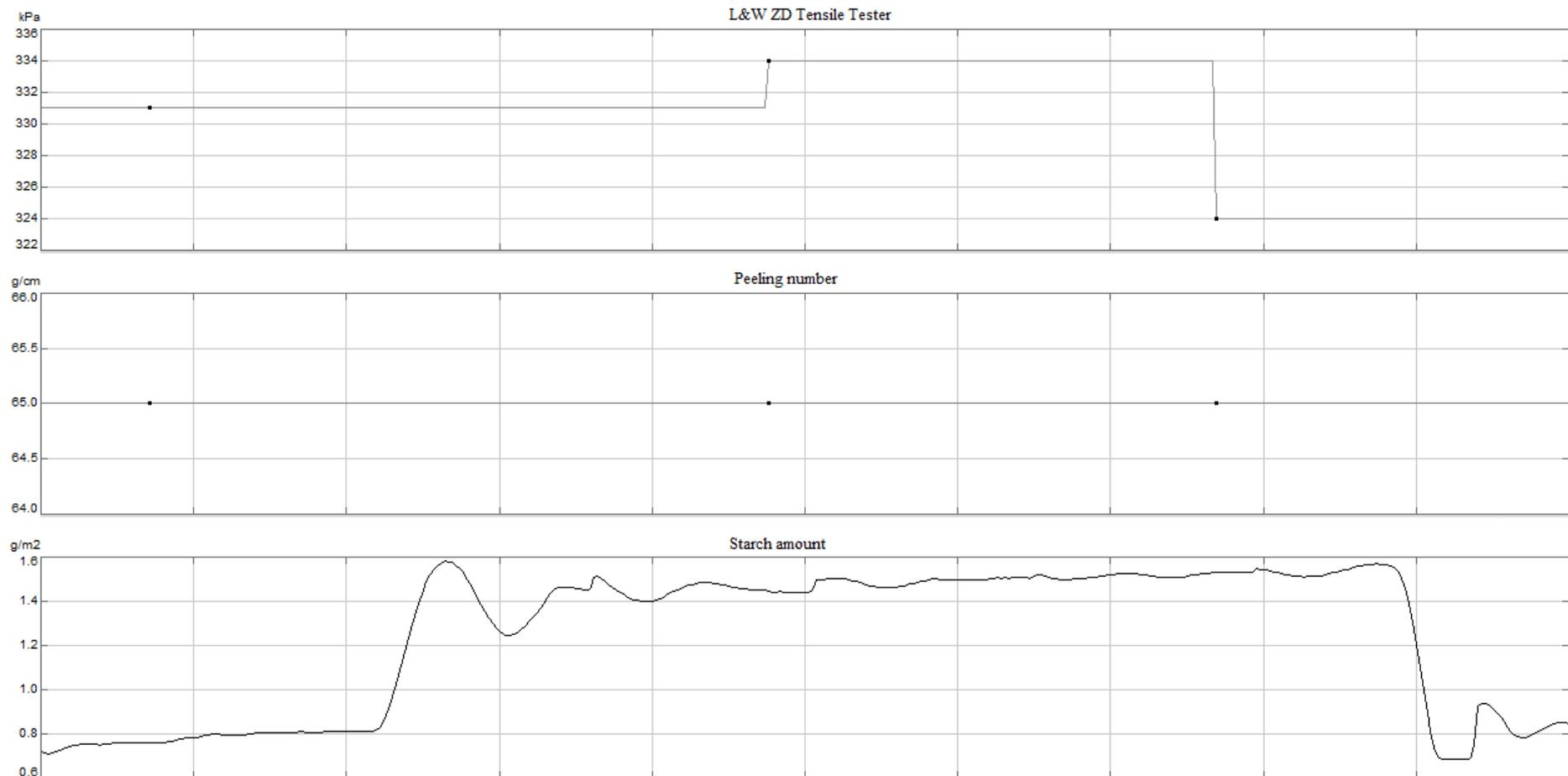
**Figure 42** Cross directional, machine directional and geometrical bending stiffness index during trial. Yellow line describes thickness of the board.

The theoretical improvement of elastic modulus of the back layer is calculated based on the calculation model. The basis weight was the same in every trial points so the bending stiffness results did not changed due to differences between basis weights of samples. During trial the geometrical elastic modulus increased about 17% that is about the same increase as in second trial. Results are presented in Figure 43. The trial results are compared in more detail in the next chapter.



**Figure 43** The estimated geometrical elastic modulus of the back layer as a function of starch amount. Black lines describe variation between sample results.

As the effect of starch on delamination strength of the board left unclear in second trial it was monitored during third trial also. The excess ASA was not added to the board during third trial so it did not influence the results as in second trial. Instead, spray starch was added to the back layer to improve ply-bond strength between back and middle layer. The amount of spray starch was kept constant during the trial so it did not cause variation to delamination strength results. In Figure 44 results of delamination strength according to Peeling test and L&W Tensile test are presented. Peeling test results indicate that higher amount of starch applied to the board does not improve or weaken the delamination strength. The results of L&W ZD Tensile test are both increased and decreased during trial. The starch amount was almost the same during the increase and decrease in delamination strength trend. This suggests that the increased starch amount is not the factor that influenced the delamination strength variation during trial. Based on the results the increased delamination strength values during the second trial was more of the cause of the ASA removal from the process water than the increased starch amount.



**Figure 44** The effect of high starch amount on delamination strength is estimated based on the "Peeling test" and L&W ZD Tensile test. Tests were executed once before the trial and two times during trial (black dots of line). The time interval in Figure is seven hours.

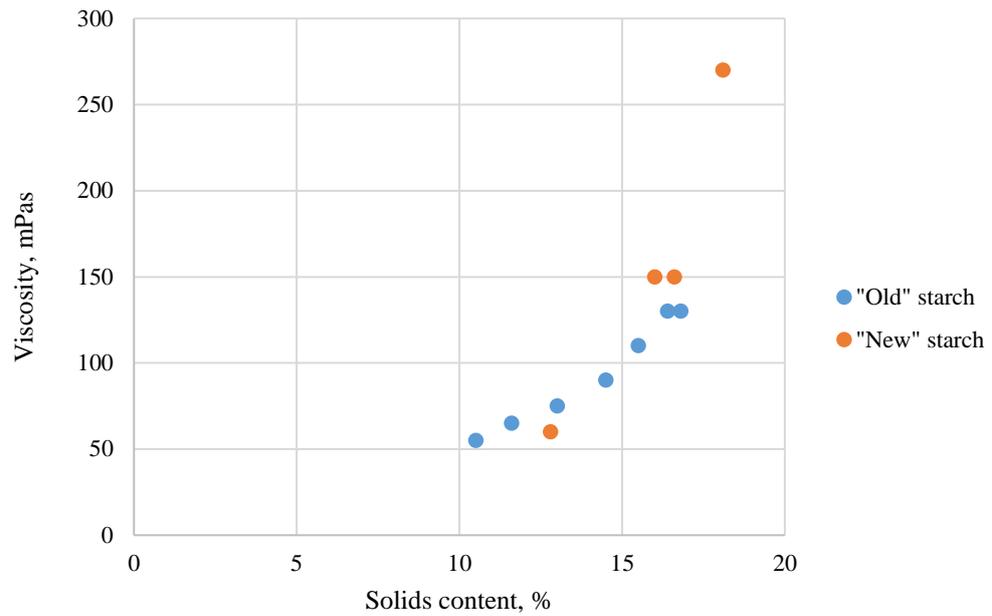
## 9.2 Viscosity

The objective of viscosity trial was to raise the viscosity of the surface size higher than in the previous trials and examine how the high viscosity influences on the quality properties of the board. The produced board during trial was double coated Tambrite with basis weight of 270 g/m<sup>2</sup>. The trial plan was to raise the solids content of starch high and then decrease the enzyme dose to increase the viscosity higher than in previous trials. During the increase of solids content of starch viscosity increased so high (270 mPas) that enzyme dose could not be decreased. Higher viscosity due to lower enzyme dose could have caused problems at the coater. The trial plan is presented in Table XI in which the second measurement point could not be carried out.

**Table XI** The measurement points of the trial. The second measurement point was not executed due to unexpected increase of viscosity during the first measurement point.

Measurement point	Solids content, %	Enzyme dose, kg/t
Reference point	10	1.4
1	18	1.4
2	18	1.2

As described in chapter 8.3 the supplier of the surface size starch was changed during the thesis trials. For this reason, the enzyme dose level is lower than in previous trials. Viscosity of the “Old” and “New” starch during trials are compared in Figure 45. The “Old” starch describes viscosity during the second solids content trial in which the “Old” starch was used and enzyme dose was kept in 1.8 kg/t. The “New” starch describes viscosity in this trial when the enzyme dose was kept in 1.4 kg/t. The viscosity of “New” starch in machine tank raised unexpectedly high when the solids content was increased in the first measurement point. When the solids content was set higher the dilution water flow to the machine tank decreased but nothing in the enzymatic cooking process did not change. Although every starch sample was taken from the same place in a same way the sample might not have been presentative. For example, there might have been thickened starch in the sample line that end up mixing to the sample.

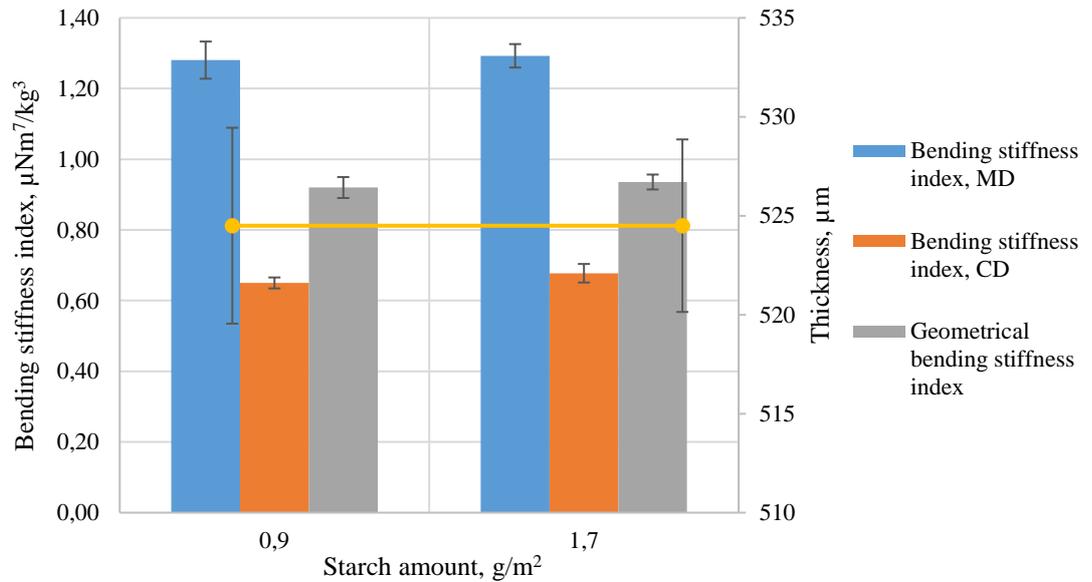


**Figure 45** The trends of the "New" and "Old" starch as a function of solids content of starch. The data of "Old" starch is based on the second trial measurements. Measured samples are taken from the machine tank.

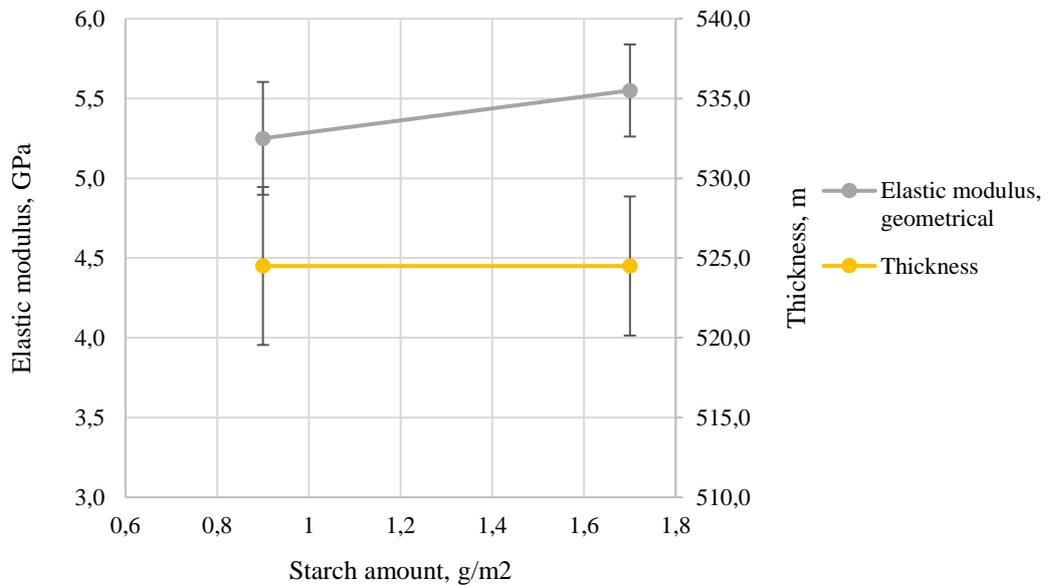
The bending stiffness index is presented in two trial points in Figure 46. In the trial point "Starch amount 1.7 g/m<sup>2</sup>" the solids content of starch was 16.5-18% and the viscosity was 150-270 mPas. The solids content was in the same level as in previous trials but the viscosity was raised higher to see if higher viscosity would give higher bending stiffness. Based on the results in Figure 46 geometrical bending stiffness index increased only about 2%, which is lower than the standard deviation of geometrical bending stiffness index results at Ingerois mill. Increase of bending stiffness index in second solids content trial was 8% and in the third trial it was 7%. Thickness, which can cause variation to the bending stiffness index, varied significantly during trial but the average thickness of measurement points and the deviation between thicknesses of samples were almost the same.

The hypothesis was that higher viscosity of starch would increase bending stiffness index because starch would stay on the surface of the layer and would not penetrate into the board structure. Hypothesis was not proven to be accurate based on the trial results. Difference compared to other trials was that the "New" starch was in use during trial. "New" starch reacted differently towards the enzymatic treatment than the "Old" starch which was used in other trials. Enzymatic cooking influences bonding ability of starch so the different starch is

one probable reason for the different bending stiffness index results compared to other trial results. The increase in theoretically estimated geometrical elastic modulus of the back layer was only 6%. Increase is significantly lower compared to second and third solids content trials due to the minor increase of bending stiffness (Figure 47).



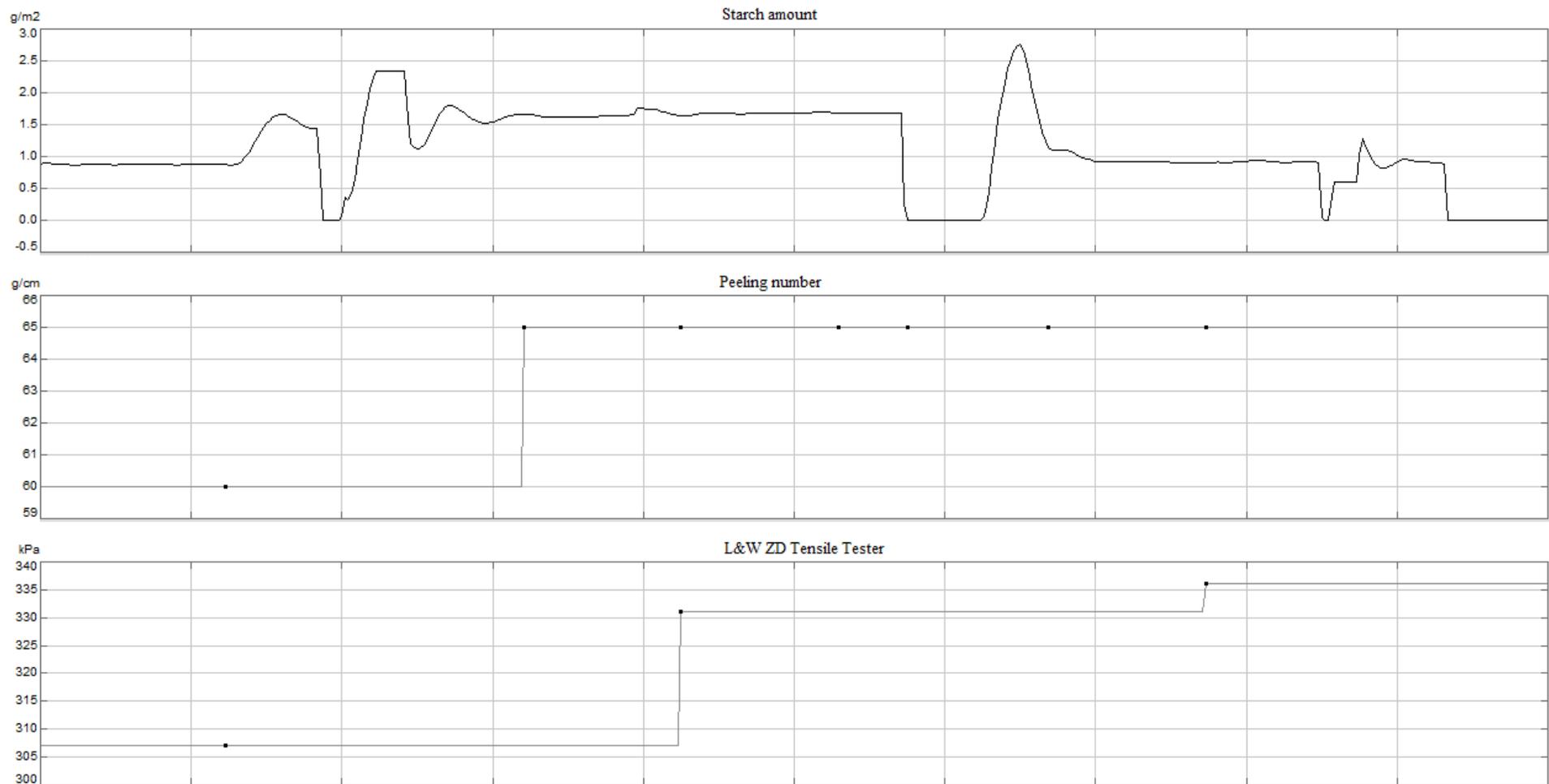
**Figure 46** Machine directional, cross directional and geometrical bending stiffness index in two measurement points. Black lines describe deviation of sample results in each measurement point.



**Figure 47** Calculated geometrical elastic modulus of the back layer as a function of starch amount during trial.

In Figure 48 the trends of delamination strength of the board are presented during the trial. Delamination strength was measured in two methods as in previous

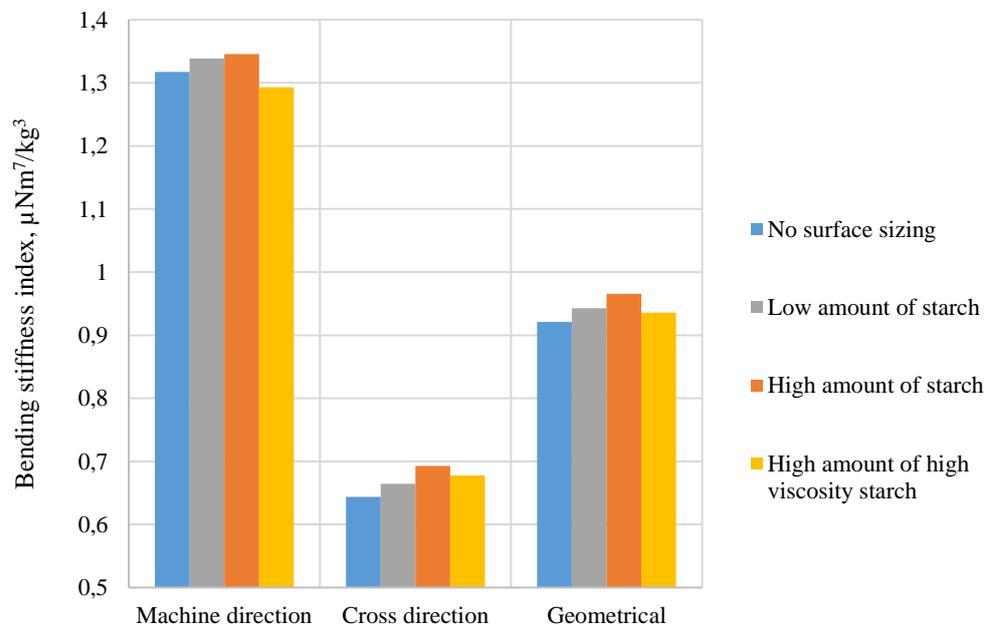
trials: “Peeling” test and L&W ZD Tensile test. Opposite to previous trials, delamination strength values were improved as the solids content and viscosity of starch increased which indicate that starch has penetrated into the board. This result do not follow the theory that starch with higher viscosity penetrates less into the board structure. The other factors that influence delamination strength were studied more carefully and two influencing factors were changed during trial: Quality of groundwood (middle layer of board) and beating level of chemical pulp (top and back layers). Shopper Riegler value of groundwood pulp was increased which indicates that pulp was refined more and the bonding ability of fiber increased. In addition, the beating of chemical pulp had been increased which improves the bonding ability of chemical pulp fibers. The increase of delamination strength was probably more of a consequence of the changes considering pulps than increase of starch.



**Figure 48** Peeling test was executed three times during trial and L&W ZD Tensile test was done once (black dots of line). Time interval in the Figure is eight hours.

## 9.4 Conclusions

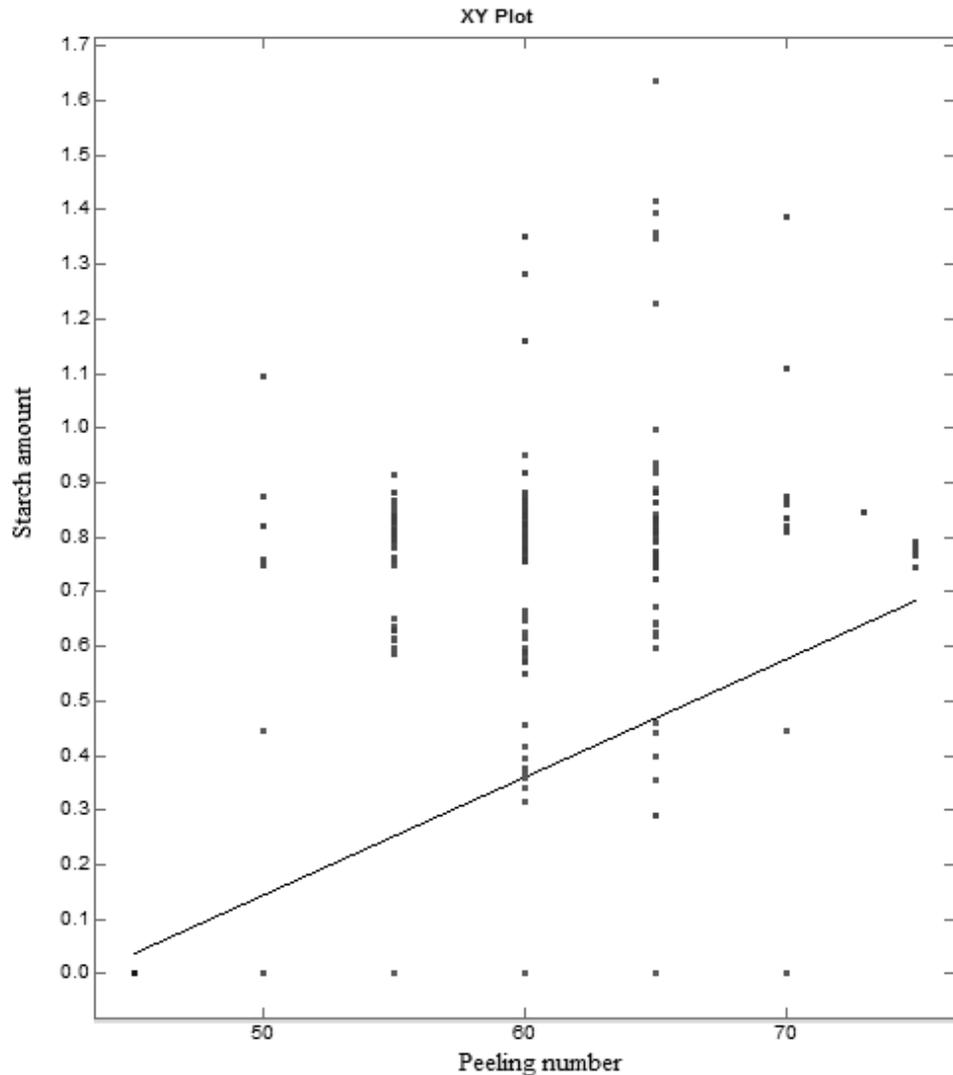
In this chapter, the results of the solids content and viscosity trials are compared. Objective of all these trials was to determine how the change of starch properties influence bending stiffness, delamination strength and curl of the board. Based on the results running parameters of starch could be defined. In Figure 49, the effect of starch amount on bending stiffness index is presented by using data from process diagnostic tool Wedge and from the results of the trials. Data that represents board without surface sized back layer is collected from four-month period before the coating station was installed. Low amount of starch represents the time when surface size amount was 0.5-0.9 g/m<sup>2</sup>. The data for the “High amount of starch” and “High viscosity” sections bases on the trials that are presented above. Figure 49 indicates that the higher amount of starch influences bending stiffness index of the board positively but higher viscosity does not cause any excess benefit. Factor that has to be taken into consideration is the change of starch before the viscosity trial. Based on trial the “New” starch influences on bending stiffness of the board differently than the “Old” one. The change of starch is probable reason for differences between bending stiffness index results of trials.



**Figure 49** “No surface sizing” bar represents data from four-month period before surface sizing process was installed. “Low amount of starch” represents amounts of 0.5-0.9 g/m<sup>2</sup>. Data is collected from Wedge. “High amount of starch” and “High amount of high viscosity starch” base on the trials.

Based on theoretical knowledge surface sizing improves bending stiffness of the board by increasing elastic modulus of the layer it is applied on. For this reason, elastic modulus of the surface sized back layer is estimated based on the measured bending stiffness values. Estimation of elastic modulus is executed with calculation model that is formed at Ingerois mill. Based on estimation, elastic modulus of the back layer increased approximately 4% with low starch amount. Comparison is executed based on data when low surface size amount was applied on back layer and data of 4-month period before the surface sizing was started. The increase of elastic modulus of the back layer with high amount of starch (1.5-1.6 g/m<sup>2</sup>) is 11% compared board without surface size. Increase is lower compared to trial results because bending stiffness of trial reference samples were lower than the average level of bending stiffness. During second and third solids content trial, elastic modulus of the back layer increased 17-19% when amount of starch increased from 0.8 g/m<sup>2</sup> to 1.6 g/m<sup>2</sup>.

One of the predicted benefits of surface sizing with starch of low solids content and viscosity was the improvement of delamination strength between middle and back layer. For this reason, the effect of starch amount on delamination strength was monitored during trials. There was neither positive nor negative effect on delamination strength as the starch amount was increased from 0.8 g/m<sup>2</sup> to 1.5 g/m<sup>2</sup>. Result was logical as the increase in starch amount was executed by increasing the solids content and viscosity of surface size. In Figure 50, the effect of surface size amount is presented as a function of peeling number that represents the delamination strength between middle and back layer. Only the data from the low starch amounts are presented. The peeling number increases with the increase of starch amount. This indicates that when the objective is to improve delamination strength of the board the starch solids content and viscosity have to be adjusted low. Solids content of starch in machine tank is about 10% and viscosity is 40-60 mPas when the starch amount varies between 0.7-0.8 g/m<sup>2</sup>.



**Figure 50** Peeling number as a function of starch amount. Peeling number indicates delamination strength between middle and back layer of the board.

The curl of the board was monitored during the second solids content trial. The increased starch or water amount that layer applied had no visible effect on board curl during trial. The curl was not influenced by the changes of starch properties during the other trials either which indicates that the curl cannot be modified by changing the solids content or viscosity of starch. The curl of the board stayed in target range during surface sizing. If the board straightens curl have to be controlled in other ways such as pressure difference between specific drying cylinders or by drying the top layer more after the two first coaters.

Process have to be viewed in two different ways when the running parameters of surface sizing process are defined. First, when the objective is to apply low amount of surface size on the board. The solids content of starch should be 8-12%

to achieve surface size amount between 0.6-0.9 g/m<sup>2</sup>. The viscosity of surface size is determined in enzymatic cooking process by adjusting the enzyme dose and converting time (Chapter 8). As explained in the Chapter 8 it is important that viscosity is not decreased under 100 mPas (viscosity after cooking) as the starch chain loses its bonding ability if it is degraded too much. With low solids content (8-12%) surface size is diluted before the machine tank, which lowers the viscosity in machine tank to a level of 40-60 mPas. By applying surface size with low solids content and viscosity delamination strength is increased but bending stiffness index is not changed significantly. Based on Figure 49 geometrical bending stiffness index increased about 2% when low surface size amount is applied on the back layer of the board.

When objective is to apply high amount of surface size to the board solids content of starch has to be increased. Based on the solids content trials, the solids content of starch has to be at least 16% in the machine tank to achieve 1.5-2.0 g/m<sup>2</sup> surface size amount. The viscosity in machine tank varies between 100 mPas and 150 mPas (at temperature 40-50°C) depending on the viscosity of the storage tank. The viscosity in the storage tank should be over 100 mPas at temperature 60-70°C and solids content higher than the aimed solids content in machine tank. The need for excess drying compared to the low surface size amount is not required based on the trials. With higher amount of surface size bending stiffness index increased more than with low starch amount. Based on the Figure 49 geometrical bending stiffness index increased about 5% compared to the board that is not surface sized. Data in Figure 49 is based on the calculated average from all of the trial results. Delamination strength improvement that was achieved with low amount of starch was also achieved with high starch amount. To see the improvements of these properties in longer term the higher amount of starch should be applied to the board for longer than only the trial time. In Table XII the starch properties for low and high amount of surface size are presented. The quality improvements that are achieved with each parameters are presented with symbols “+” (positive effect), “-” (negative effect) and “0” (no effect).

**Table XII** Running parameters for low and high amount of surface size.

<b>Starch amount, g/m<sup>2</sup></b>	<b>Solids content, Storage tank, %</b>	<b>Viscosity, Storage tank, mPas</b>	<b>Solids content, Machine tank, %</b>	<b>Viscosity, Machine tank, mPas</b>	<b>Quality improvements</b>
0.5-0.9	20	100-150	10	30-60	<ul style="list-style-type: none"> <li>• Delamination strength +</li> <li>• Bending stiffness +/-0</li> <li>• Curl +</li> </ul>
1.5-1.7	20-25	100-150	16-20	100-150	<ul style="list-style-type: none"> <li>• Delamination strength +</li> <li>• Bending stiffness +</li> <li>• Curl +</li> </ul>

## 10 DRYING AND CURL CONTROL

One of the objectives of the experimental part was to optimize the curl control and drying parameters considering the surface sizing process. The drying of surface size was planned to be executed with IR dryers and air dryers. During installation of the coating station it was noticed that there was rust in the air channels that made air dryers unusable. The surface size of the back layer was dried only by using IR dryers. The IR dryers were consisted of four sections which could be switched on and off individually. Amount of used IR sections varied from one to four sections during normal production because of troubles with lighting of the sections. Surface sized back layer of the web dried although only one section was in use. Based on process data the amount of IR sections used did not have significant effect on the moisture content of web or the drying requirement in drying section.

The drying group after the third coating station (ninth drying group) was used for drying when the moisturizing unit was in use. Moisturizing unit is placed under the first lower cylinder of the ninth drying group and it is the last drying group before reeling. After surface sizing process replaced the moisturizing unit as a moisturizer of the back layer the need for heating the web by using ninth drying group had to be determined.

Curl of the board could not be controlled specifically with surface sizing process. Board absorbed certain amount of water to the back layer and the solids content of starch did not cause significant variation to that amount. The ways to control the curl of the surface sized board had to be determine in trials. The objective of the trials in this section was to optimize drying parameters of the board by not using unnecessarily drying capacity and by that energy. The specific result values of trials of this section can be found in Appendices VI-VII.

### 10.1 Drying requirement of last drying group

Drying group that follows the new coating station and its infrared dryers is the ninth and last drying group of the board machine. Before the new coating station was in use the back layer of the board was sprayed with water. This prevented the board to curl towards the surface layer. The pneumatic moisturizing unit was placed under the first lower drying cylinder of the group. Because there are no other drying after the ninth drying group heating of the drying cylinders was necessary to achieve dry web before reeling. When coating station was installed drying of surface size was executed with infrared dryers, and the pneumatic moisturizing unit was taken off the use. Due to this change, the need for drying the web with the last drying group had to be determined again. In addition, the effect of the pressure difference between lower and higher cylinders of the ninth group on board curl was determined. In Table XIII the measurement points of the trial are presented.

**Table XIII** The measurement points of the ninth drying group trial. Cylinder pressures describe the pressures of the ninth group drying cylinders.

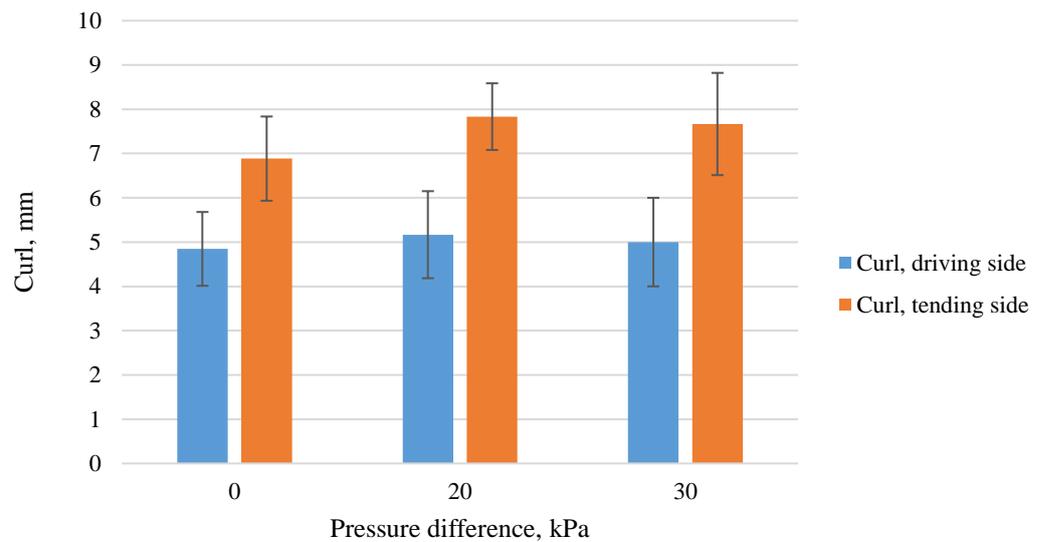
Measurement point	Lower cylinder pressure, kPa	Higher cylinder pressure, kPa
1	80	80
2	80	60
3	60	60
4	60	30
5	30	30
6	30	10
7	10	10
8	0	0
9	-10	-10
10	-20	-20

Produced board during the trial was double coated Tambrite and the basis weight was 260 g/m<sup>2</sup>. During trial the machine speed did not vary significantly. The pressure difference between lower and higher cylinders of seventh drying group was kept constant during trial. Seventh drying group is traditionally used for controlling curl. Pressure of the cylinders were lowered in small steps so the temperature of the cylinders had time to decrease and the effect of the certain pressure drop could be determined. The temperature of surface and back layer of the web were monitored carefully to make sure that the temperature stayed higher after the drying group than the temperature before the group. If it turned other way around humidity could start to condense on the measurement device placed before the reeling. Temperature of the web was measured by hand with infrared meter before and after the drying group but as more reliable measurement the on-line measurement device, which is placed before reeling, was monitored.

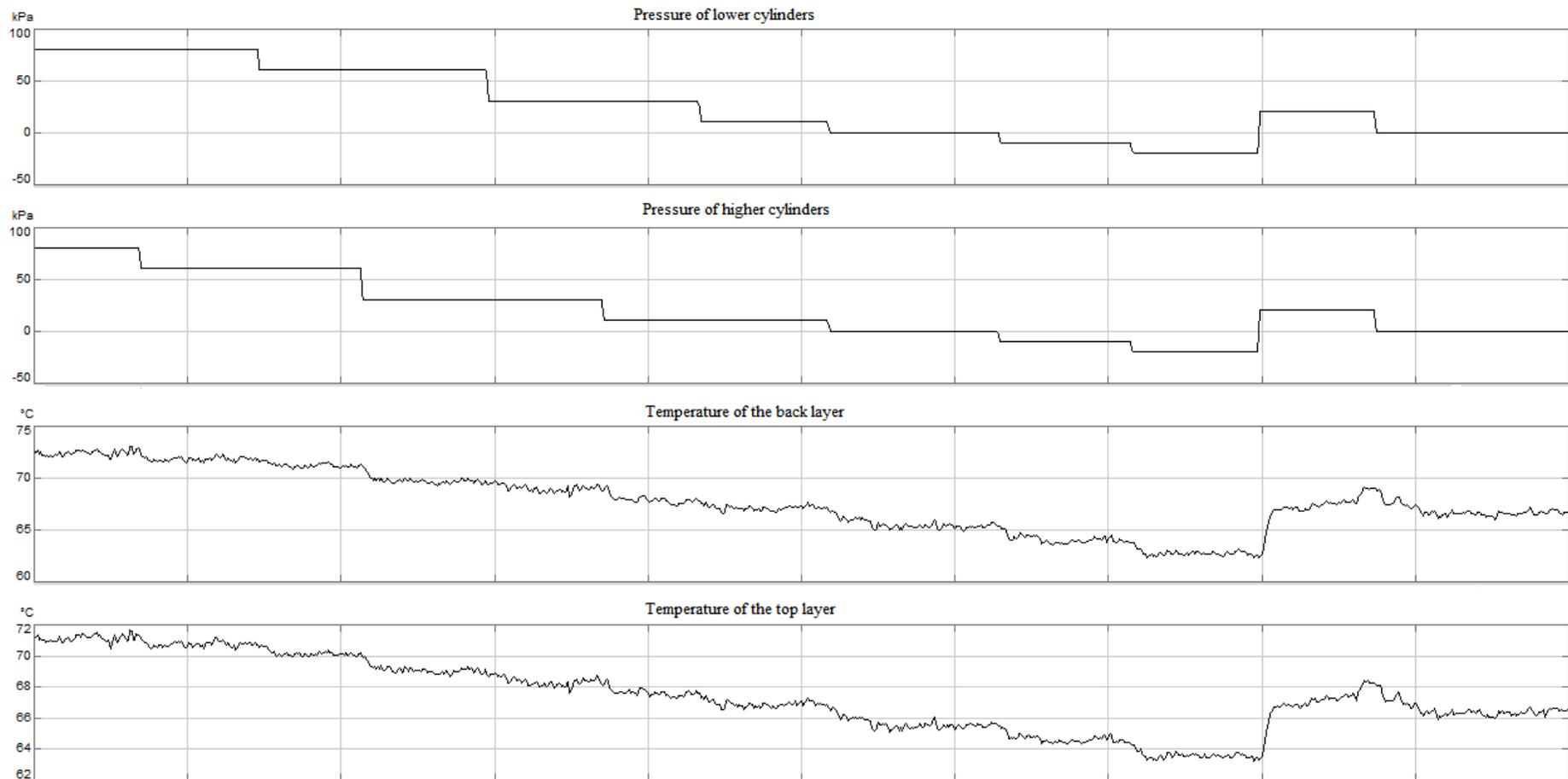
As a result of the trial described in Table XIII the temperature of the back layer decreased more than 10°C and the temperature decrease of the surface layer was about 8°C (Figure 52). Temperature of the web before the drying group stayed higher than the temperature after the group in every measurement point so there was no risk of moisture condensing into water drops. The moisture of the back layer increased 2%-units during the trial but stayed in tolerance. Results that are more specific can be found in Appendix VI. Based on the results of this trial can be said that web is heated unnecessarily with the last drying group when the moisturizing unit is not in use. As a conclusion, new instructions was made to keep the pressure of both upper and lower cylinders at 0 kPa to avoid unnecessary heating and use of energy.

The effect of pressure difference between the lower and higher cylinders in the final drying phase on curl of the paperboard was detected during the measurement points 2, 4 and 6. In these measurement points the lower cylinders had higher pressure which means that the surface layer was dried more. From every machine roll a cross directional sample was cut from the whole width of the machine. The curl was measured from the samples that are cut from the driving and tending side of the cross directional sample. These samples were taken into control room and the curl was measured right away with a specific measuring box (described more detailed in chapter 3.6 Curl). The positive result values means that the board is

curled towards the backside and the negative values means that the curl is towards the top side of the board. The results are presented in Figure 51. Based on this trial the pressure difference of lower and higher cylinders in the final drying phase, when the board is already coated and dried, do not have any effect on the curl. The measured values of curl had little variation in both directions that is considered normal with used measurement method.



**Figure 51** Board curl during trial. Black lines describe deviation between the measured curl values in each pressure difference point.



**Figure 52** Pressure of the cylinders of ninth drying group were lowered 100 kPa during trial. Time interval in Figure is 16 hours.

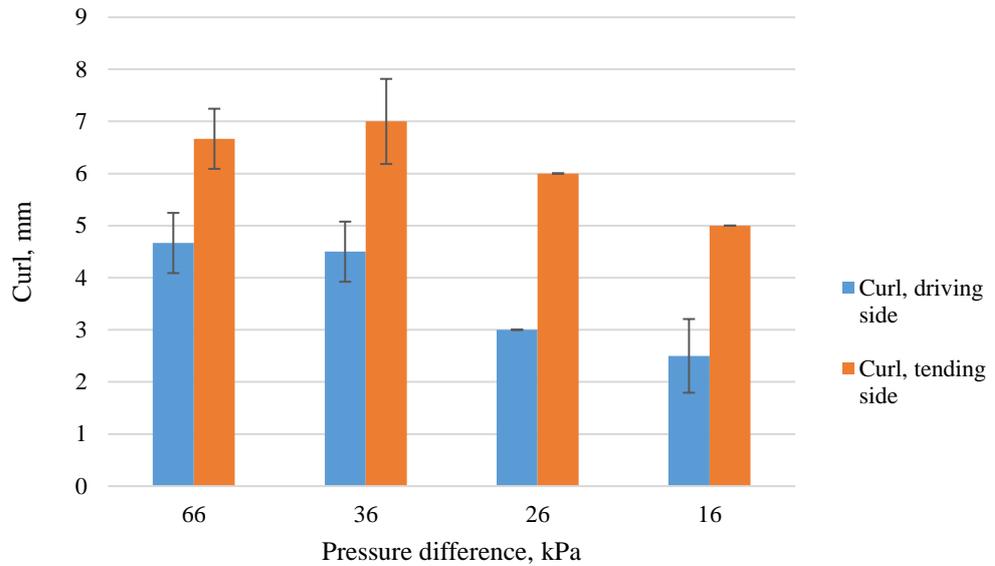
## 10.2 Curl control with the seventh drying group

Traditional way to control paperboard curl at Ingerois mill is done by adjusting the pressure difference of the lower and upper cylinders of the seventh drying group. Because the new surface sizing process changed the way of curl control, the effect of seventh drying group on curl was determined. The measurement points of the trial are described in Table XIV. When the pressure difference is positive it indicates that lower cylinders have higher pressure than upper cylinders and the top side of the board is dried more. During trial the machine speed was constant. Produced board during the trial was double coated Tambrite and the basis weight was 260 g/m<sup>2</sup>.

**Table XIV** Measurement points of the seventh drying group curl trial. Pressure difference describes the difference between lower and upper cylinders of the seventh drying group.

Measurement point	Pressure difference, kPa
1	36
2	66
3	16
4	26
5	36

Results of the trial are presented in Figure 53 where the bars describe the averages of curl values in each measurement point and the black lines describe the deviation of results. Based on trial results, the effect of pressure difference of seventh drying group on curl was the same as it was before the surface sizing process. Pressure difference of cylinders of the seventh drying group has a significant influence on board curl. Pressure difference was not lowered more because board started to straighten too much and trial was executed during normal production. When pressure difference of drying cylinders is used as a way to control curl it limits the drying capacity at high production speeds. For this reason, it is not beneficial to control curl only by changing the pressure difference of the drying cylinders.



**Figure 53** Cross directional curl of the board. The pressure difference of the seventh drying group upper and lower cylinders was decreased. Black lines describe the deviation of each measurement point results.

### 10.3 Moisturizing device

Before the surface sizing process, the curl of the board was controlled with pneumatic moisturizing device. The device is placed after the coating station and under the first lower drying cylinder of the last drying group. The effect of moisturizing device on surface sized board curl was determined during this trial. In addition, the moisture content of back layer was monitored during trial to avoid too high moisture content of the board. Cross sectional samples were taken from every machine roll and the curl of the board was measured in the control room right after taking the sample. The back layer was felt from the whole width of the sample to make sure that it did not feel wet or sticky. If the sprayed water amount is raised too high the starch on the back layer can start to wet again and the board layers stuck together in reeling. The trial plan was to raise the sprayed water amount in  $0.5 \text{ g/m}^2$  steps as long as the moisture content of the back layer stayed under 14% and the curl of the board could be controlled. The measurement points that were run are presented in Table XV.

**Table XV** Measurement points of the moisturizing device trial.

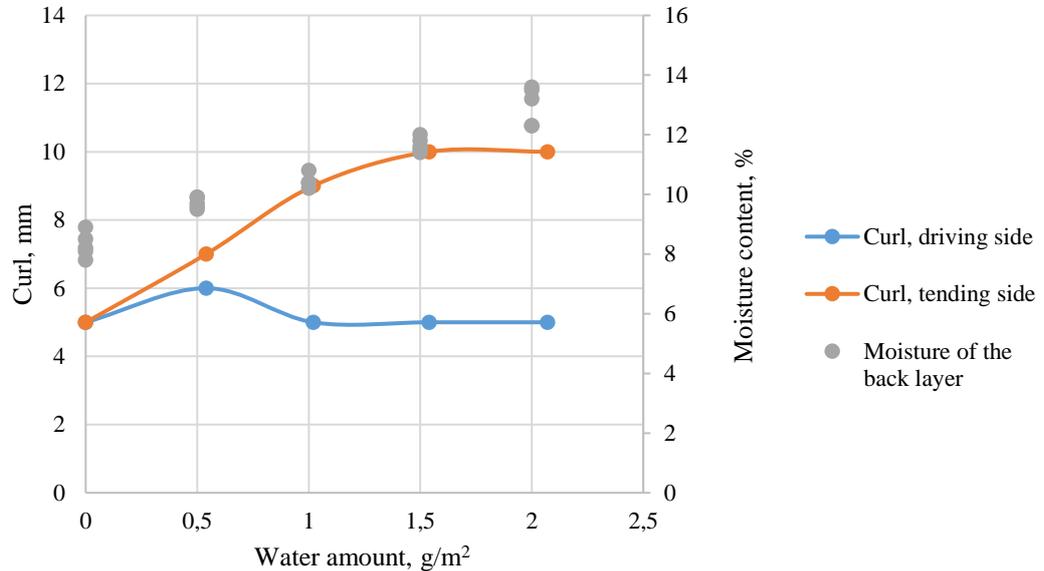
Measurement point	Water amount, g/m <sup>2</sup>
Reference point	0
1	0,5
2	1
3	1,5
4	2

The pressure difference between lower and upper cylinders of the seventh drying group was kept constant during trial as it is used as a parameter to adjust the curl. During trial the machine speed was constant. Produced board during trial was double coated Tambrite and the basis weight was 310 g/m<sup>2</sup>. The solids content and viscosity of surface size starch were kept constant to make sure the variation in curl was not influenced by surface size.

The water amount that was applied to the back layer before the surface sizing process varied between 3 g/m<sup>2</sup> and 4 g/m<sup>2</sup>. The last drying group is used for drying when moisturizing unit is used. During surface sizing back layer of board applies about 8-9 g/m<sup>2</sup> of water and the drying group is not used for drying of the web as it is dried with infrared dryers after the coater. During trial the pressure of last drying group cylinders was raised to 100 kPa which is the level in which the web was dried during the use of moisturizing unit.

In Figure 54 the effect of the excess water from the moisturizing unit on board curl is presented with trend lines. In addition, the moisture content of the back layer of the board before reeling is presented during the trial. The moisture content is monitored with online measuring unit that is placed before reeling. Online measuring unit measures the moisture content continuously and the whole trend is presented in Appendix VIII. The moisture content values in the Figure 54 are chosen from the continuous data in a way that for every measurement point there are five moisture content results with two to three minutes apart during each measurement point. Unexpectedly, the board curl changed only in tending side samples that is not logical because the water is sprayed from the nozzles that are placed to the whole width of the web. The curl on tending side curled towards back layer but driving side curl was not changed almost at all. After the trial it was noticed that big part of the nozzles from the driving side of the moisturizing

unit were clogged. This explains why the curl was changed only on the samples from the tending side.



**Figure 54** Cross directional curl of the board with increasing water amount of moisturizing unit.

Based on the results and the logical reason for asymmetric curl of the board can be said that excess water from the moisturizing unit curls surface sized board towards backside of the board. The water amount could be increased to 2 g/m<sup>2</sup> before the moisture content of back layer increased to almost 14%, which was the maximum limit that was set in the beginning of the trial. Although the moisture content increased during the trial the back layer did not feel sticky or wet which would have indicated that the surface size had started to wet again.

#### 10.4 Conclusions

One of the objectives was to optimize the curl control and drying parameters considering the surface sizing process. Drying and Curl Control section consisted three individual trials: Drying requirement of last drying group, curl control with seventh drying group and moisturizing unit trials. Drying of surface size with IR dryer and air dryer were not tested in trials as the air dryer turned out to be unusable and drying of the surface size was executed only with IR dryer. IR dryer is consisted of four sections and the amount of sections used during normal production varied between one to four sections. The moisture content of web or the drying requirement in drying section was not influenced by the lower amount of IR sections used after surface sizing.

The back layer of the board has to be moisturized to prevent its curl towards the top side. Before the new coating station was in use the back layer of the board was sprayed with water and the board was dried during the last drying group. After the moisturizing unit was taken off use and the surface sizing process was started the need of drying with last drying group had to be determined again. The temperature of the web was monitored carefully to make sure that the temperature stayed higher after the drying group than before it. Humidity condense on the measurement device before the reeling if the temperature decreased during the drying group. Based on trial the temperature of the back layer of the board was decreased more than 10°C and the temperature decrease of the surface layer was about 8°C as the pressure of the drying cylinders were decreased 100 kPa. The temperature stayed higher after the group than the temperature before the group and condensation of humidity did not cause any problems. The moisture of the back layer increased 2%-units during the trial but stayed in tolerance. As a conclusion new instructions was made to keep the pressure of upper and lower cylinders at 0 kPa (before the trial 80 kPa) to avoid unnecessary heating and use of energy.

In seventh drying group trial, the traditional way to control the board curl at Ingerois mill was determined with surface sized board. The pressure difference between lower and upper cylinders of seventh drying group was modified during trial and the behavior of board curl was monitored. Based on trial results surface sizing did not cause variation to the effect of pressure difference on board curl. Although pressure difference of cylinders can be used for controlling board curl it is not beneficial considering the drying capacity of the machine.

The objective of moisturizing device trial was to determine how board curl is influenced by excess water sprayed to the surface sized back layer by using pneumatic moisturizing unit. Based on trial results excess water from the moisturizing unit curls surface sized board towards backside of the board. The amount of water that was applied with moisturizing unit was 2 g/m<sup>2</sup> at the highest point. The trial was stopped in that point due to the increase of the moisture content of the back layer. Too high moisture content causes risk of surface size wetting which could lead to board layers stuck together in reeling.

## 11 FINAL CONCLUSIONS

Experimental part of thesis included trials considering enzymatic cooking process, surface size properties and their effect on quality of the board, curl control and drying after the surface sizing process. The parameters of the enzymatic cooking process determine the viscosity of the surface size. Running parameters of the cooking process were defined. As a result of the trials was found that it is more effective to adjust the viscosity by changing the enzyme dose than the converting time. The aimed viscosity level of surface size in the storage tank was 100-150 mPas when solids content was set to 18-20%. This level was achieved with enzyme dose of 1.8 kg/t and converting time of 17 minutes. During trials the supplier of the surface size starch was changed and the parameters had to be defined again. The effect of enzyme was much stronger with the “New” starch although the starch type was not changed (native wheat starch). The enzyme dose was lowered significantly to avoid too much degradation of starch. The optimization of the “New” starch was not completed during the time limit of this thesis.

The properties of the surface size starch was optimized in four trials. The objective was to find the running parameters that provide the wanted quality improvements and determine how the amount of surface size can be controlled. The behavior of the board curl was monitored during trials. Based on results the starch amount can be controlled by changing the solids content of starch. During normal production the solids content was kept low (8-12%) and the amount of surface size was between 0.5-0.9 g/m<sup>2</sup>. In trials the solids content was raised to 16-20% and the starch amount increased to 1.5-1.7 g/m<sup>2</sup>. The increase of viscosity as the solids content was already high (>16%) did not cause variation to the starch amount.

Results from all of the trials indicate that surface sizing of the back layer improves the bending stiffness and elastic modulus of the board. The increase of bending stiffness was more significant with high starch amount (1.5-1.7 g/m<sup>2</sup>) than with low starch amount (0.5-0.9 g/m<sup>2</sup>). Based on the viscosity trial, higher viscosity does not cause any excess benefit to the bending stiffness increase. Result is in contrary with theoretical knowledge. Factor that has to be taken into consideration in this comparison is the change of starch before the viscosity trial. The “New”

starch is one probable reason for low bending stiffness index increase because of different reactivity of “New” starch towards the enzymatic treatment. Enzymatic treatment influences bonding ability of starch.

The effect of surface sizing on delamination strength (especially between middle and back layer) was determined. The results indicated that there was neither positive nor negative effect on delamination strength as the starch amount was increased from 0.8 g/m<sup>2</sup> to 1.5 g/m<sup>2</sup>. Instead, when comparing the data of board without surface size and with low amount of surface size, delamination strength between middle and back layer (Peeling number) was increased with low amount of starch. The reason is logical because in low amounts of applied starch, the viscosity and solids content of starch are low (in machine tank). Regarding theoretical knowledge starch penetrates more into the board structure when its viscosity and solids content are low.

The increased starch or water amount that layer applied had no visible effect on board curl during trial. This indicates that the curl cannot be modified by changing the solids content or viscosity of starch. The curl of the board stayed in target range when the back layer was surface sized. Ways to control curl was determined in three trials: Drying requirement of the last drying group, Curl control with the seventh drying group and Moisturizing device trials. Based on the trials curl of the surface sized board can be controlled by adjusting the pressure difference between the lower and upper cylinders of the seventh drying group and by spraying water with moisturizing unit after the surface sizing process. The pressure difference between cylinders of the last drying group (ninth) did not influence board curl. Neither of these “effective” curl control methods are not very desirable as the pressure difference between cylinders limits the drying capacity (especially in high machine speeds) and use of moisturizing device requires more drying of the ninth drying group.

The drying requirement of ninth drying group was determined during trial because the moisturizing unit was taken out of use and surface size was dried by using IR dryer. Due to trial results web was heated unnecessarily with the last drying group when the moisturizing unit was not in use. The pressure of the drying cylinders were lowered 100 kPa and web temperature before reeling decreased almost 10°C during trial. As a conclusion, new instructions was made to keep the

pressure of the both upper and lower cylinders of ninth drying group at 0 kPa (80 kPa before trial).

Factors that require more research after this thesis are the “New” starch properties and drying parameters of the coater. The resulted quality improvements in trial where the “New” starch was used differed from the results of the other trials. For this reason, properties of the “New” starch have to be studied more after this thesis. The possibilities of air dryer could not be determined during trials due to their poor condition. The possibility of drying the surface sized web with IR dryer and then blow cool air from the air dryers to the web could be a possibility to lower the reeling temperature of the board. In addition, the effect of wet end parameters such as beating has to be determined. Based on theoretical knowledge, beating is a significant factor in penetration of the surface size into the board. Starch penetration influences the quality properties of the board.

The possibility of decreasing the amount of spray starch in the back layer or even leaving the spray starch off was discussed during thesis. The use of spray starch in the back layer causes web breaks when it is in continuous use. The starch starts to build up to the nozzles with time and finally starch clump releases and causes web break. Spray starch is added to achieve sufficient delamination strength of the board. As surface sizing improves the delamination strength of the back and middle layer there is a possibility to decrease the spray starch from the back layer.

## REFERENCES

- ABB Inc., 2016, Data sheet, L&W ZD Tensile Tester, Kista, Sweden, [Internet], Available at: [https://library.e.abb.com/public/d4fe543360cc4434a9d7d8ab80526e0c/285\\_LW\\_ZD\\_Tensile\\_Tester\\_v1.0.pdf](https://library.e.abb.com/public/d4fe543360cc4434a9d7d8ab80526e0c/285_LW_ZD_Tensile_Tester_v1.0.pdf), [referred to: 21.3.201].
- Annushko, A., 2013, Gelatin as an additive in bio-based barrier films, Master's Thesis, Lappeenranta University of Technology, Chemical and Process Engineering.
- Bajpai, P., 2012, Enzymatic Modification of Starch for Surface Sizing, Biotechnology for Pulp and Paper Processing, Chapter 16, Springer Science+Business Media, Thapar Research and Development Center Colony, Patiala, India, p. 317-325.
- Biricik, Y., Sonmez, S., Ozden, O., 2011, Effects of Surface Sizing with Starch on Physical Strength Properties of Paper, Department of Forest Product Chemistry and Technology, Istanbul University.
- Bublitz, R.H., Klem, R.E., Craig, K.A., 1990, Thermal and Thermal-chemical Conversion of Starch, Starch and Starch Products in Paper Coating, Kearney, R.L., Maurer, H.W, (Ed.), TAPPI PRESS, Atlanta, p. 109-122.
- Bruun, S.-E., 2000, Starch, Pigment Coating and Surface Sizing of Paper, Papermaking Science and Technology, Book 11, Lehtinen, E., (Ed.), Fabet Oy, Jyväskylä, p. 241-249.
- Cargill, 2013, How to avoid the formation of Retrograded Amylose Particles (RAPs).
- Fellers, C., Sören, Ö., Mäkelä, P., 2012, Evaluation of the Scott bond test method, Nordic Pulp and Paper Research Journal, vol 27, Stockholm, Sweden, [Internet], Available at: <http://www.innventia.com/PageFiles/7447/2012-27-02-p231-236-Fellers.pdf>, [referred to: 14.12.2016].
- Gräsbeck, L., 1983, Paperin pintaliimaus, Paperin valmistus, Osa 2, Arjas, A., (Ed.), Suomen Paperi-insinöörien Yhdistyksen oppi- ja käsikirja, Turku, Finland, p. 251-772.

Grön, J., Rantanen, R., 2000, Surface sizing and film coating, Pigment Coating and Surface Sizing of Paper, Papermaking Science and Technology, Book 11, Lehtinen, E., (Ed.), Fabet Oy, Jyväskylä, p. 489-537.

Heikkilä, P., Rajala, P., 2000, Drying of paper coatings and drying equipment, Pigment Coating and Surface Sizing of Paper, Papermaking Science and Technology, Book 11, Lehtinen, E., (Ed.), Fabet Oy, Jyväskylä, p. 543-564.

Hentschel, P., 2000, Polyvinyl alcohol, Pigment Coating and Surface Sizing of Paper, Papermaking Science and Technology, Book 11, Lehtinen, E., (Ed.), Fabet Oy, Jyväskylä, p. 277-286.

Holik, H., 2006, Handbook of Paper and Board, Sixta, H., (Ed.), WILEY-VCH, Germany.

Hägglom-Ahnger, U., Komulainen, P., 2001, Paperin ja kartongin valmistus, Kemiallinen metsäteollisuus II, Jyväskylä, Finland, p. 78-111.

Kajanto, I., 1995, Paperin taivutusjäykkyyden ja ryhdin perusteet, KCL, [Internal material].

Kajanto, I., 1998, Paper Physics, Structural mechanics of paper and board, Papermaking Science and Technology, Book 16, Niskanen, K., (Ed.), Fabet Oy, Jyväskylä, p. 193-219.

Kajanto, I., Laamanen, J., Kainulainen, M., 1998, Paper bulk and surface, Paper Physics, Papermaking Science and Technology, Book 16, Niskanen, K., (Ed.), Fabet Oy, Jyväskylä, p. 99-108.

Kajanto, I., Niskanen, K., 1998, Dimensional stability, Paper Physics, Papermaking Science and Technology, Book 16, Niskanen, K., (Ed.), Fabet Oy, Jyväskylä, p. 223-256.

Ketola, H., Andersson, T., 1999, Dry-strength additives, 1999, Dry-strength additives, Papermaking Chemistry, Papermaking Science and Technology, Book 4, Neimo, L., (Ed.), Fabet Oy, Jyväskylä, p. 270-275.

Kiviranta, A., 2000, Paperboard grades, Paper and Board Grades, Papermaking Science and Technology, Book 18, Paulapuro, H., (Ed.), Fabet Oy, Jyväskylä, p. 54-72.

Lindblad, G., Fürst, T., 2001, The Ultrasonic Measuring Technology on Paper and Board, Lorentz & Wettre, Kista, Sweden.

Linnonmaa, J., Trefz, M., 2000, Pigment coating techniques, Pigment Coating and Surface Sizing of Paper, Papermaking Science and Technology, Book 11, Lehtinen, E., (Ed.), Fabet Oy, Jyväskylä, Finland, p. 415-483.

Lipponen, J., Grön, J., Brun, S.-E., Laine, T., 2003, Surface Sizing with Starch Solutions at Solids Contents up to 30%, Journal of Pulp and Paper Science 30, number 3, pages 82-90. © 2004 Pulp and Paper Technical Association of Canada (PAPTAC).

Lorentzen & Wettre, 2013, L&W Handbook, Pulp and Paper Testing, L&W Bending Tester, Kista, Sweden, p. 155-157.

Lorentzen & Wettre, 2013, L&W Handbook, Pulp and Paper Testing, L&W ZD Tensile Tester, Kista, Sweden, p. 148-149.

Markström, H., Bending stiffness, Lorentzen & Wettre, [Internet], Available at: [http://www.lorentzen-wettre.com/images/stories/LorentzenWettre/QT\\_bilder/Measuring\\_bending\\_stiffness.pdf](http://www.lorentzen-wettre.com/images/stories/LorentzenWettre/QT_bilder/Measuring_bending_stiffness.pdf), [referred to 8.12.2016].

Mattelmäki, R., 2004, Factors affecting surface strength and fiber rising of SC and LWC paper-literature review, Stora Enso, Imatra, Finland.

Muhonen, P., 2013, Brittleness of paper, Master's thesis, Lappeenranta University of Technology, LUT Chemistry, Lappeenranta, Finland.

Mäkelä, M., 2000, Coating color preparation, Pigment Coating and Surface Sizing of Paper, Papermaking Science and Technology, Book 11, Lehtinen, E., (Ed.), Fabet Oy, Jyväskylä, Finland, p. 336-343.

Nevalainen, T., 2012, Kartongin taivutusjäykkyys ja sen määrittäminen, Kandidityö, Paperiteknikan laboratorio, LUT Kemia, Lappeenrannan teknillinen yliopisto.

Niskanen, K., Kärenlampi, P. 1998. In-plane tensile properties. Paper Physics, Papermaking Science and Technology, Book 16, Niskanen, K., (Ed.), Fapet Oy, Jyväskylä, p. 139-186.

Nybolm, I., 1991, Taivekartongin pintakerroksen optimointi II, KCL, [Internal material].

Pylkkö, J., 2000, Mechanical base paper, Pigment Coating and Surface Sizing of Paper, Papermaking Science and Technology, Book 11, Lehtinen, E., (Ed.), Fabet Oy, Jyväskylä, p. 29-44.

Pöllänen, S., 2008, Monikerroskartongin viiraosan liitoskosteuksien hallinta ja mittaaminen, Bachelor's Thesis, Tampere University of Applied Sciences.

Radosta, S., Kiessler, B., Vorweg, W., Brenner, T., 2016, Molecular composition of surface sizing starch prepared using oxidation, enzymatic hydrolysis and ultrasonic treatment methods, Starch – Stärke, Vol. 68, Issue 5-6, p. 541-548, Germany.

Savcor Oy, 2017, Wedge Process Diagnostic Tool, [Internet], Available at: <https://www.savcor.com/solutions/process-diagnostics/>, [referred to: 3.5.2017].

Stora Enso Official Website, Ingerois mill, [Internet], Available at: <http://renewablepackaging.storaenso.com/about-us/mills/ingerois-mill>, [referred to: 29.12.2016].

Stora Enso, 2016, Esittelymateriaali, [Internal website, WeShare].

Stora Enso, 2016, Pölymittaus, [Internal material].

TAPPI, 1996, Bending stiffness of paper and paperboard (resonance length method), [Internet], Available at: <https://research.cnr.ncsu.edu/wpsanalytical/documents/T535.PDF>, [referred to: 7.3.2017].

Testing Machines, Inc., Taber Stiffness Tester, [Internet], Available at: <https://www.testingmachines.com/product/79-05-taber-digital-stiffness-testers>, [referred to: 23.12.2016].

Tillmann, O., Paper and Board Grades and Their Properties, Handbook of Paper and Board, p. 446-466

Tolan, J., 2002, Enzymes, pulp and paper processing, Encyclopedia of Bioprocess Technology, Wiley, New York.

UMV, 2017, INVO Coater, [Internet], Available at: <http://www.umv.com/en/products/invo-coater/>, [referred to: 23.2.2017].

UMV, 2017, Presentation material, [Internal material].

Voigt, 1990, Dispersion of Starches, Starch and Starch Products in Paper Coating, Kearney, R.L., Maurer, H.W, (Ed.), TAPPI PRESS, Atlanta, p. 71-91.

VTT, Prowledge Oy, KnowPap 17.0 Learning Environment for Papermaking and Automation, [LUT Intranet], Available at: LUT, Intranet, signing in is required, [referred to 5.12.2016].

## **APPENDICES**

Appendix I	Pre-trials of enzymatic cooking process
Appendix II	Trials of enzymatic cooking process
Appendix III trial	Parameters and results of the second solids content
Appendix IV	Parameters and results of the third solids content trial
Appendix V	Parameters and results of the viscosity trial
Appendix VI	Parameters and results of the last drying group trial
Appendix VII	Parameters and results of the seventh drying group trial
Appendix VIII	Results of the moisturizing unit trial

**PRE-TRIALS OF ENZYMATIC COOKING PROCESS****Table I** Parameters during the pre-trials of the enzymatic cooking process and results.

<b>Enzyme dose, kg/t</b>	<b>Converting time, min</b>	<b>Temperature in the first jet cooker, °C</b>	<b>Temperature in the second jet cooker, °C</b>	<b>Dilution water, ml/s</b>	<b>Viscosity, mPas</b>	<b>Solids content, %</b>	<b>Temperature °C</b>
						31.8	
						30.7	
3.5	20	80	130	14	75	24.3	78
3.0	20	80	130	14	80	23.3	79
2.0	20	80	130	14	110	24.6	79
2.0	10	80	130	14	240	26.0	76
1.5	10	80	130	14	190	24.5	74
2.0	15	80	130	0	210	25.2	80
2.0	20	80	130	0	110	26.1	84

**TRIALS OF ENZYMATIC COOKING PROCESS****Table I** Enzyme dose trial results.

<b>Enzyme dose, kg/t</b>	<b>Converting time, min</b>	<b>Viscosity after the cooking process, mPas</b>	<b>Solids content after the cooking process, %</b>	<b>Viscosity in storage tank, mPas</b>	<b>Solids content storage tank, %</b>	<b>Sample temperature, °C</b>
2.0	20			70	17.7	
1.0	20	410	21.6	90	18.0	
1.8	20	160	22.3	120	18.0	
1.8	20	170				
1.5	20	230				

**Table II** Converting time trial results.

<b>Enzyme dose, kg/t</b>	<b>Converting time, min</b>	<b>Viscosity after the cooking process, mPas</b>	<b>Solids content after the cooking process, %</b>	<b>Viscosity in storage tank, mPas</b>	<b>Solids content storage tank, %</b>	<b>Sample temperature, °C</b>
1.8	20	170	21.4	120	18.5	71
1.8	15	150	21.2			68
1.8	15	200	20.5	120	18.6	72
1.8	13	260	19.5			70
1.8	17	130	19.6			73
1.8	17	160	19.5			72
1.8	17	150		150		72

**PARAMETERS AND RESULTS OF THE SECOND SOLIDS CONTENT TRIAL****Table I** Parameters during the second solids content trial and results.

Sample taken	Aimed solids content, machine tank, %	Solids content, machine tank, %	Viscosity, machine tank, mPas	Solids content, storage tank, %	Viscosity, storage tank, mPas	Starch amount, g/m <sup>2</sup>
8:15	10	10.5	55	18.0	120	0.78
9:30	14 (changed 8:55)	11.6	65			0.97
10:40	14	13.0	75			1.09
11:45	16 (changed 11:00)	14.5	90			1.31
12:30	18 (changed 11:35)	15.5	110			1.45
13:30	18	16.8	130			1.55
15:10	14 (changed 13:40)	16.4	130	18.3	140	Web break

**Table II** Results of the second solids content trial.

<b>Grammage, g/m<sup>2</sup></b>	240	235	239	241	238	238
<b>Thickness, <math>\mu\text{m}</math></b>	471	462	459	463	456	458
<b>Starch amount, g/m<sup>2</sup></b>	0.76	0.80	1.53	1.51	1,56	0.80
<b>Bending stiffness index MD, <math>\mu\text{Nm}^7/\text{kg}^3</math></b>	1.29	1.39	1.31	1.26	1.27	1.42
<b>Bending stiffness index CD, <math>\mu\text{Nm}^7/\text{kg}^3</math></b>	0.58	0.63	0.66	0.63	0.67	0.75
<b>Bending stiffness index geometrical, <math>\mu\text{Nm}^7/\text{kg}^3</math></b>	0.86	0.95	0.93	0.88	0.92	1.03
<b>Estimated geometrical elastic modulus, GPa</b>	4.7	5.7	5.5	4.9	5.4	7.0

**PARAMETERS AND RESULTS OF THE THIRD SOLIDS CONTENT TRIAL****Table I** Parameters during the third solids content trial and results.

Sample taken	Aimed solids content, machine tank, %	Solids content, machine tank, %	Viscosity, machine tank, mPas	Solids content, storage tank, %	Viscosity, storage tank, mPas	Starch amount, g/m <sup>2</sup>
9:34	10	10.2	38			0.80
10:45	18 (changed 10:00)	13.1	70	16.8	120	1.32
11:50	18	15.2	90			1.44
12:50	18	17.0	110			1.50
14:30	18	18.2	140	18.9	120	1.54

**Table II** Results of the third solids content trial.

<b>Grammage, g/m<sup>2</sup></b>	268	268	268	268	268
<b>Thickness, <math>\mu\text{m}</math></b>	521	524	524	521	523
<b>Starch amount, g/m<sup>2</sup></b>	1.53	1.51	1,56	0.80	0.84
<b>Bending stiffness index MD, <math>\mu\text{Nm}^7/\text{kg}^3</math></b>	1.29	1.33	1.38	1.26	1.26
<b>Bending stiffness index CD, <math>\mu\text{Nm}^7/\text{kg}^3</math></b>	0.72	0.65	0.68	0.66	0.64
<b>Bending stiffness index geometrical, <math>\mu\text{Nm}^7/\text{kg}^3</math></b>	0.97	0.93	0.97	0.91	0.90
<b>Estimated geometrical elastic modulus, GPa</b>	5.9	5.5	6.0	5.2	5.1

**PARAMETERS AND RESULTS OF THE VISCOSITY TRIAL****Table I** Parameters during viscosity trial and results.

Sample taken	Aimed solids content, machine tank, %	Solids content, machine tank, %	Viscosity, machine tank, mPas	Solids content, after cooking, %	Viscosity, after cooking, mPas	Solids content, storage tank, %	Viscosity, storage tank, mPas	Starch amount, g/m <sup>2</sup>
7:30	18 (changed 6:00)	16.6	150			18.5	90	1.62
9:15	18	18.1	240	18.4	80			1.67
10:50	10 (changed 10:25)	16.0	150					0.91
12:30	10	12.8	60					0.87

**Table II** Results of the viscosity trial.

<b>Grammage, g/m<sup>2</sup></b>	269	266	269	269	267	268
<b>Thickness, <math>\mu\text{m}</math></b>	528	521	521	521	526	530
<b>Starch amount, g/m<sup>2</sup></b>	0.87	0.87	1.65	1.67	1.68	1.68
<b>Bending stiffness index MD, <math>\mu\text{Nm}^7/\text{kg}^3</math></b>	1.24	1.32	1.27	1.26	1.30	1.34
<b>Bending stiffness index CD, <math>\mu\text{Nm}^7/\text{kg}^3</math></b>	0.64	0.66	0.65	0.69	0.71	0.66
<b>Bending stiffness index geometrical, <math>\mu\text{Nm}^7/\text{kg}^3</math></b>	17.4	17.6	17.7	18.2	18.3	18.1
<b>Estimated geometrical elastic modulus, GPa</b>	5.0	5.5	5.2	5.5	5.9	5.6

**PARAMETERS AND RESULTS OF THE LAST DRYING GROUP TRIAL****Table I** Parameters during ninth drying group trial and results. Cylinder pressures describe pressures of ninth drying group cylinders.

<b>Pressure of higher cylinders, kPa</b>	<b>Pressure of lower cylinders, kPa</b>	<b>Temperature after the drying group °C</b>	<b>Moisture of the back side %</b>	<b>Temperature of the backside °C</b>	<b>Temperature of the topside °C</b>	<b>Curl Driving side</b>	<b>Curl Tending side</b>
80	80	71.1	9.5	72.4	71.1	4	7
60	80	71.3	9.5	72.4	71.1	5	8
60	80	67.6	10.4	71.5	70.9	4	7
60	80	68.8	10.2	71.8	70.9	6	8
60	60	69.4	10.5	70.6	70.0	4	7
60	60		10.6	71.0	70.0	5	8
30	60	69.3	10.9	70.0	69.3	4	7
30	60		11.3	69.4	68.9	6	9
30	60	66.8	10.7	69.5	68.8	5	7
30	30		10.9	69.1	68.5	6	9
30	30	66.3	10.6	68.7	68.1	4	7
30	30		10.3	68.9	68.3	5	8
10	30	67.2	11.6	68.0	67.6	4	7
10	30		11.5	67.8	67.6	6	9
10	30	65.9	11.1	67.5	67.3	6	8
10	10		11.1	67.2	67.0	6	8
10	10	63.0	11.5	66.7	66.7	4	6
10	10		11.0	67.1	66.8	6	7

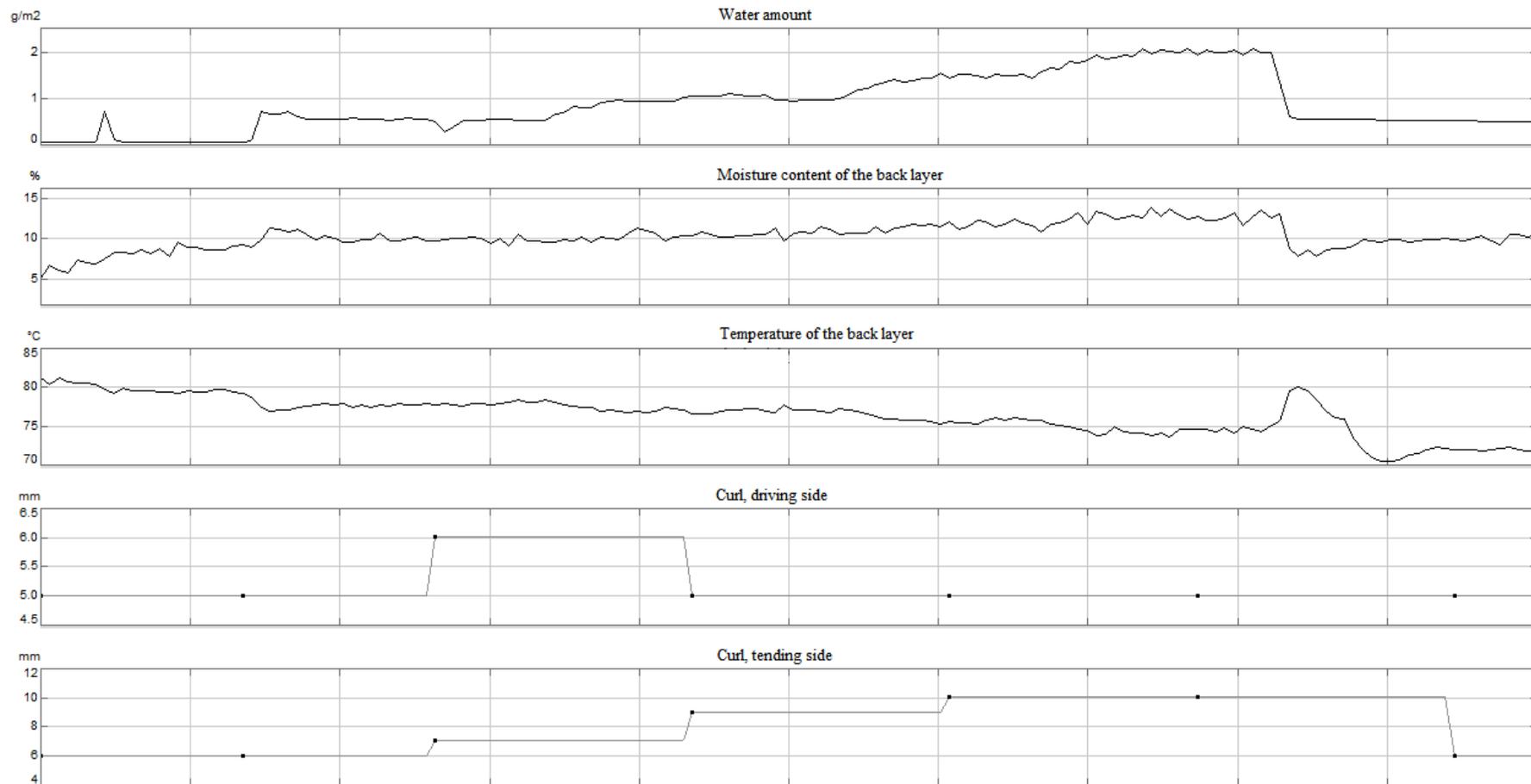
## APPENDIX VI 2(2)

0	0	64.3	11.6	65.9	66.0	4	6
0	0		11.7	65.3	65.3	4	8
0	0	64.7	11.2	65.5	65.5	4	6
0	0		11.4	65.6	65.7	6	8
-10	-10	61.5	12.0	63.9	64.5	6	6
-10	-10		11.6	63.9	64.5	5	6
-10	-10	65.9	12.0	63.8	64.4	5	6
-20	-20		11.8	63.2	63.7	5	6
-20	-20		11.5	62.6	63.4	5	7
-20	-20	60.7	12.0	62.7	63.4	5	7
-20	-20		12.3	62.3	63.2	6	7
20	20		11.2	67.0	66.8	4	6
20	20		10.4	67.6	67.2	6	8
20	20	65.4	10.2	68.9	68.3	4	7
0	0		11.6	67.0	66.6	4	5
0	0		12.1	66.9	66.5	5	6
0	0	64.4	11.7	66.3	66.0	4	7

**PARAMETERS AND RESULTS OF THE SEVENTH DRYING GROUP TRIAL****Table I** Parameters during seventh drying group trial and results.

<b>Time</b>	<b>Pressure difference between lower and upper cylinders, kPa</b>	<b>Time of parameter change</b>	<b>Curl, driving side</b>	<b>Curl, tending side</b>
22:14	36		5	7
22:39	36		4	7
23:04	36		4	6
23:29	66	23:23	4	6
23:53	66		5	7
0:18	66		5	7
0:43	16	0:19	2	5
1:08	16		3	5
1:33	26	1:08	3	6
1:57	26		3	6
2:22	36	2:00	5	8

**RESULTS OF THE MOISTURIZING UNIT TRIAL**



**Figure 1** Moisturizing unit trial results.