The Use of Microcellulose in Papermaking

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A Selluloosa on orgaaninen yhdistelmä, jota esiintyy puussa. Sen pääasialla käyttökohteita ovat paperi- ja kartonkituotteet. Mikrosellulosa (tunnetaan myös nimellä mikrokristalliselluloosa) on selluloosalaji, joka koostuu mikrokokoluokan kuiduista. Viime vuosikymmenen aikana mikro- ja nanokuitujen sovellukset ovat olleet suuren kiinnostuksen kohteina.

Tämän työn tarkoituksena on määritellä, mihin paperinominaisuuksiin voitiin vaikuttaa lisäämällä mikrosellua eri ainemäärillä.

Työssä valmistettiin paperiarkkeja, jotka sisälsivät vaihtelevissa määrin mikrosellua sekä vakiomäärän retentioainetta. Arkeista testattiin vetolujuus, neliöpaino, karheus, paksuus, läpinäkyvyys ja opasiteetti.

Testien perusteella nähtiin, että mikrosellu heikentää paperin vetolujuutta, lisää karheutta, eikä aiheuta muutoksia optisiin ominaisuuksiin. Tuloksia tarkastellessa houomattiin, että paperinvalmistuksessa käytetty arkkiformerin raina on liian suuri mikrosellusovelluksia varten. Tämä aiheutti erittäin heikon kiintoaineen retention mikrosellun osalta. Tästä syystä mikrosellun käyttömahdollisuuksia tulee tutkia paremmin sopivammissa olosuhteissa.

ABSTRACT

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Cellulose is an organic compound that is obtained from wood pulp. It is principally used to produce paper and paperboard. Microcellulose (also named as microcrystalline cellulose) is a type of cellulose that has micro-scaled fibers. Over the past decade, the use of micro- and nanofibres of cellulose in various applications has been discussed. Cellulose is an essential part of paper, which affects some properties of a paper sheet depending on paper production methods, size and shape of cellulose.

The aim of this thesis work was to define which properties of paper were affected when using different methods to apply certain amounts of various microcellulose types in papermaking.

Paper sheets were made with different dosages of microcellulose and retention aids. Different properties of paper sheets were tested, such as tensile strength, beta formation, sheet weight, roughness, thickness, transparency and opacity.

These types of microcellulose proved to be inefficient leading to decline in tensile strength of paper, increase of roughness and no change in optical properties. Parameters of paper depend upon raw materials it is made from, application methods, paper machine properties and standards. Microcellulose has to be studied more and applied in papermaking under different conditions. Reliable methods of using microcellulose have to be invented in order to reveal its true potential in paper industry.

Keywords: cellulose, microcellulose, microcrystalline cellulose, nanocellulose, pulp, paper, retention.

CONTENTS

ABBREVIATIONS

MFC	microfibrillated cellulose
MC	microcellulose
NFC	nanofibrillated cellulose
UFC	ultrafine cellulose
SD	standard deviation
AVG	average

1 INTRODUCTION

Cellulose is one of the most abundant organic materials that is used in a wide range of applications. It has been noticed that microfibrillated cellulose is a very potential material that can be used as a reinforcement agent in paper and paperboard industry and many other applications. It has already been used in food, construction, paper and paperboard industries as well as in medicine. It is derived from natural sources (e.g., annual plants, trees, algae). Research in the production and processing methods of micro- and nanofibrillar cellulose leads to significant developments of composite materials nowadays (TAPPI 2013).

Cellulose can be manufactured in many different ways, therefore every cellulose grade has its own properties, such as size and shape, length of fibrils, bulk weight and absorption.

The target of this thesis was to study new ways of applying microcellulose (MC) in papermaking, obtaining results and learning more about papermaking. The idea is to use MC as a reinforcing material in sheets and see how microcellulose affects various properties, like tensile strength and opacity. Methods, that were used to make paper with addition of microcellulose and retention chemical, were developed according to initial test results.

2 THEORY

Presence of cellulose in the wood is the main reason why paper is made mostly from wood. The cellulose is oriented in the direction of the wood fibers that makes the fiber directionally dependent. Among the wood polymers, such as cellulose, hemicellulose and lignin, the most attention has been given to cellulose when considering mechanical properties of the wood elements. Cellulose is stiff hence it is a reinforcing material in composites. Therefore the component that determines properties of the fibre in the most important direction is its length (Ek, Gellerstedt et al. 2009 volume 3, 15).

2.1 Cellulose

Cellulose is the most essential part of plants. It is the main component of plant cell walls. In paper and paperboard industry, trees are used for papermaking because they contain cellulose. Different trees contain certain types of cellulose. It is the most suitable material since it is renewable and has got long enough fibers to make paper products stronger. Trees are basically made microfibril-reinforced hemicellulose, waxes, lignin and other elements.

Cellulose is a polysaccharide that refers to biopolymers. Polysaccharides often share the same chemical formula but at the same time they have different chemical structure. For example, galactose, glucose and fructose have the same formula $C_6H_{12}O_6$ but chemical structure is different. They are soluble in water, however polysaccharides are more hydrophobic if they have large quantities of internal hydrogen bonds (Dufresne 2012, 1).

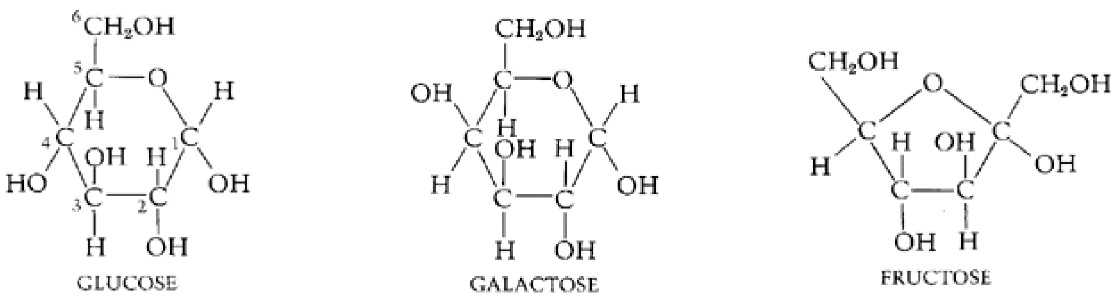

FIGURE 1: Comparison of common polysaccharides chemical formula (Dufresne 2012, 4).

Atoms arrangement, chain conformation and intermolecular bonding influence chemical and physical properties of polysaccharides. Cellulose consists of parallel and linear macromolecules which are compactly arranged because of intermolecular hydrogen bonds. Chemical formula of cellulose is $(C_6H_{10}O_5)_n$. Cellulose is hydrophobic due to intermolecular hydrogen bonding that prevents water from accessing functional groups. Because of high crystallinity, only certain solvents can increase solubility of cellulose without exposing it to chemically assisted degradation (Dufresne 2012, 1).

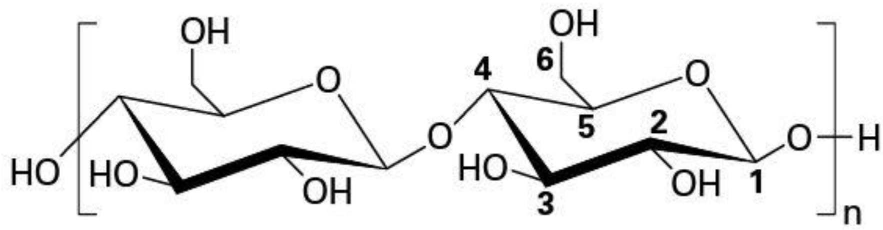

FIGURE 2. Basic chemical structure of cellulose (Dufresne 2012, 4).

2.1.1 Structure and properties of fibres

Mechanical and physical properties of wood depend on length and stiffness of the cellulose fibers and fibrils. Moreover, content and distribution of components in a cell wall of a tree depend on the tree species. Hardwood and softwood have got different S_2-layer properties. When separating fibres of chemical pulp made from softwood they will tend to collapse. Hardwood fibres will tend to improve their shape (Ek, Gellerstedt et al. 2009 volume 3, 15).

2.1.2 Cell wall layers

Cell wall layers of trees are reinforced with cellulose fibrils that are arranged in different positions, angles and directions. Layers S_1 and S_3 are outer and inner layers that are thinner have got the cellulose reinforcing fibrils oriented at a high angle to the fiber direction.

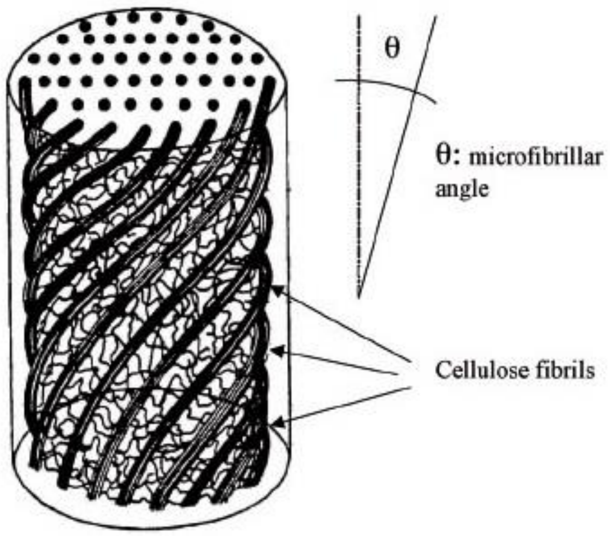

FIGURE 3. Angle of cellulose fibrils (Cristophe Baley, 2007).

These layers provide a very good stiffness and strength to the cell that works as a pipe for water transporting within the stem. Wood fibre material is composed of 70 to 80 %, therefore mechanical properties of the fibre are dominant. The layer $S_{2,}$ is located between two other layers, therefore it is characterized by its low fibril angle. This layer has very low fibril angle thus ensuring stiffness and strength in its fibre direction that ensures bending resistance of the stem against the blowing winds (Ek, Gellerstedt et al. 2009 volume 3, 16; Roger M. Rowell 2012, 48).

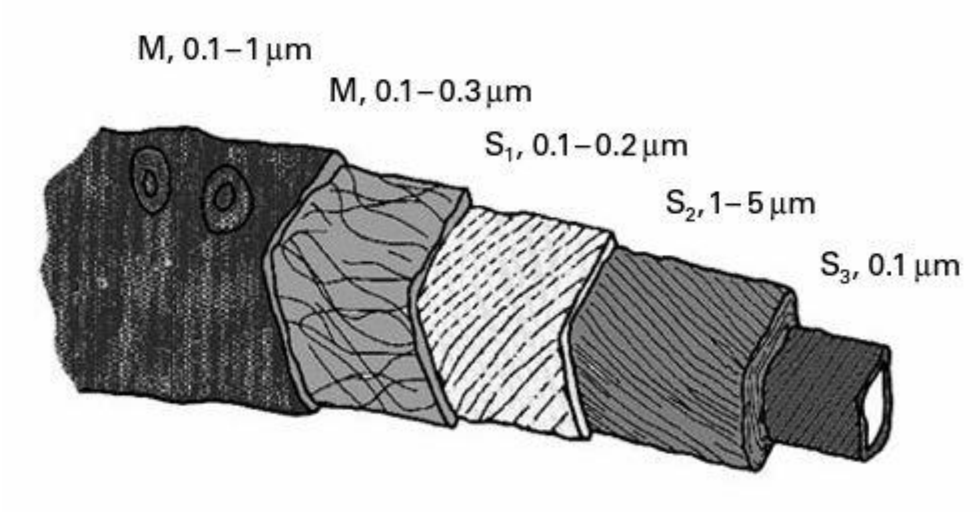

FIGURE 4. Wood fibre with lamella, M, remains on the surface. In the secondary layers the S_1, S_2, S_3, the cellulose fibrils are arranged at a specific angle (Ek, Gellerstedt et al. 2009 volume 3, 16).

There are several types of cellulose in wood: crystalline and non-crystalline. As the density of cellulose increases in the wood, crystalline regions are formed. Most cellulose that was derived from wood contains about 65 % of crystalline regions which means it is highly crystalline. The remaining part of cellulose has a lower packing density which means it is amorphous cellulose. Cellulose can also be accessible and non-accessible which means that the cellulose is accessible to microorganisms, water, and so on. The surfaces of crystalline cellulose are accessible but the other part of the crystalline cellulose is non-accessible. Most of non-crystalline cellulose is accessible but part of the non-crystalline cellulose is covered with lignin and hemicellulose which makes the cellulose non-accessible. These concepts of cellulose accessibility are essential for pulping, chemical modification and moisture sorption. Cellulose has to be studied very well in order to achieve better results in future (Roger M. Rowell 2012, 36–37).

2.2 Pulping

Pulping is the process where recovered paper, hardwood or softwood trees are exposed to fiber separation process in order to extract fibres from wood.

Sulfate Pulping

Sulfate pulping (KRAFT) is a process, where cellulose is separated from lignin and other elements. It is the most common chemical pulping method. This is performed using strong bases, sodium hydroxide, NaOH and sodium sulfide, Na_2S. The active cooking ions are OH and HS. The hydrogen sulphide serves as the main lignin removal agent and the hydroxide keeps lignin fragments in solution. During kraft process, lignin macromolecules are broken up, then fragments of lignin and hemicellulose are solubilized is strong alkaline. In reality, this pulp grade has got utterly high strength. As a result, sulfate pulp has very high cellulose purity due to complete removal of lignin and other elements. (Ek, Gellerstedt et al. 2009 volume 2, 4; Hagiopol & Johnston 2012, 11)

3 MATERIALS AND METHODS

Taking into account choice and properties of raw materials is very important part in the whole work process. The way raw materials are processed and used directly affects final results, therefore different methods were applied in this thesis work.

3.1 Raw materials

3.1.1 Cellulose fibers

The microfibrillated (MFC) cellulose that was used in this study was manufactured by company named J. Rettenmeyer & Schöne. The product name of this microcellulose was Arbocel.

Three types of Arbocel MFC were used:

- Arbocel BWW 40, **200 µm**
- Arbocel BE 600-30, **30 µm**
- Arbocel UFC 100, **8 µm**

According to datasheets of the cellulose, these cellulose grades can be used as reinforcing and filling agents, as well as extender and absorbent. All these different microcellulose types were used in each test series.

TABLE 1. Basic properties of pure cellulose (Kremer Pigmente GmbH, Vento Co., Ltd).

	BWW 40	**BE 600-30**	**UFC 100**
Fiber Length	200 µm	30 µm	
Fiber thickness	20 µm	18 µm	
Bulk weight	110 g/l - 145 g/l	200 g/l - 260 g/l	160 g/l
Whiteness (at 461 nm)	86 % ± 5 %	85 % ± 5 %	

3.1.2 Pulp

Bleached birch and pine sulfate pulp were used in papermaking process. The pulp was received from TAKO board.

3.1.3 Retention agent

The retention agent that was used in this work was Percol PBR 40 (material 55438403).

3.2 Paper making

Process of making pulp and paper sheets is explained together with preparations of MFC and retention agent as well as calculations for trial point.

3.2.1 Trial points for microcellulose

It was decided that in this work there would be multiple trial points where amount of MC was increased gradually. This allowed us to observe the effect of microcellulose amount variation upon behaviour of basis weight, tensile strength, opacity, transparency, roughness and thickness. In the first trial point sheets were made without addition of microcellulose in order to develop a reference for further trials. Microcellulose was added to pulp solution in trial points:

TABLE 2. Trial points for MFC

Trial point	**MFC percentage of pulp mass**
1	0 %
2	5 %
3	10 %
4	15 %
5	20 %
6	25 %
7	30 %
8	50 %

3.2.2 Preparation of microcellulose

The consistency of pulp solution before forming sheets was set to 2g/l in order to achieve approximate basis weight of 60 g/m^2.
Microcellulose was mixed with a birch or pine pulp at its trial points in a form of a liquid solution that had the same concentration as diluted pulp without microcellulose. In order to achieve this specific concentration, 20 g of microcellulose was mixed in 1 liter of water using IKA T-18 Ultra Turrax homogenizer and then this solution was poured into 9 liters of water and mixed constantly during experiments. Continuous mixing was applied to buckets containing pulp, microcellulose and retention aid because pulp and cellulose settle down over time that can decrease accuracy of papermaking process and further testing.

It was calculated how much the consistency was over the standard 2g/l when microcellulose was added into pulp at every trial point fight before making paper sheets. Firstly, 1,2 liters of refined pulp, that had original concentration of 15,7 g/l, was taken and mixed with 8,2 liters of water in a bucket. Additional 5 % of microcellulose weight was mixed with pulp.
Consistency of refined pulp is 15,7 g/l. Since 1,2 liters of pulp is taken for mixing with water, the amount of 1,2 liters of pulp fibers has to be calculated:

$$15{,}7 \text{ g/l} \times 1{,}2 \text{ L} = 18{,}84 \text{ g}$$

The total amount of fibers in the bucket, when the extra 5 % of microcellulose was added, was calculated in the following way:

$$18{,}84\text{g} \times 1{,}05 = 19{,}78\text{g}$$

To calculate the amount of microcellulose in the pulp solution at the 5 % trial point, original 18,84 g was substracted from 19,78 g

$$19{,}78\text{g} - 18{,}84\text{g} = 0{,}942\text{g}$$

Based on this, it was possible to calculate how much of 2g/l microcellulose solution was diluted into the pulp solution that is used in papermaking. The amount of microcellulose for the pulp solution is calculated:

$$mV = C$$
$$V = \frac{m}{C}$$

where

m = Amount of microcellulose in grams

V = Amount of microcellulose solution in litres

C = Consistency of the microcellulose solution g/l

According to the formulas above, amount of microcellulose solution required for 5 % trial point:

$$\frac{0{,}942\ \text{g}}{2\ \text{g/l}} = 0{,}471\ \text{l}$$

Consequently, 471 ml of microcellulose solution is required for 5% set point and mixed with the pulp solution. Amount of microcellulose for the rest of setpoints is calculated in the same way.

TABLE 3. Microcellulose solution and its amount mixed with a pulp solution at each trial point.

Trial point	**MC solution mixed with pulp**
0%	-
5%	471 ml (0,94 g)
10%	927 ml (1,84 g)
15%	1413 ml (2,82 g)
20%	1884 ml (3,77 g)
25%	2375 ml (4,71 g)
30%	2826 ml (5,65 g)
50%	4710 ml (9,42 g)

3.2.3 Pulp preparation and refining

Sheets of pine or birch sulfate were torn into small pieces of about 2×2 cm. According to standard SCAN-C 25:76, approximately 361 g of sulfate sheet was soaked in 5 liters of water for at least 4 hours. After the torn sheet pieces had been in water for 4 hours, they were transferred into refiner (Lorentzen & Wettre, type 3-3) and filled with extra 18 liters of water. At first, deflaking process had to be carried out for 20 minutes without applying weight on a lever arm. After 20 minutes had passed, weight of 5 kg was placed on the lever arm and refining continued depending on wood grade: 40 minutes for birch and 60 minutes for pine. Pine and birch refining time was based on the standard SCAN-C25:96

After refining, the pulp was poured into a bucket. The concentration of the pulp in the bucket was 15,7 g/l. In order to achieve the standard pulp concentration of 2 g/l, 1,2 liters of the pulp was taken and mixed with 8,2 liters of water in a bucket.

In trials, pure pine and birch pulps were used as well as combination of pine and birch pulp that was set to be 80 % of birch and 20% of pine, and by reverse, 80% of pine and 20% of birch pulp. In case of the combination of two different pulps types, the total amount of combined birch and pine pulps was set to be 7 litres as a reference for calculating required amounts of each pulp type to be mixed:

$$7\ \text{L}\times 0{,}2 = 1{,}4\ \text{L for } 20\%$$
$$7\ \text{L}\times 0{,}8 = 5{,}6\ \text{L for } 80\%$$

3.2.4 Preparation of retention aid solution

In first trials, the amount of retention agent used was 0,02 % of standard sheet weight (1,6 g) for each sheet. In the last trial, amount of retention chemical was then raised to 1 % for each sheet, that is 50 times bigger than in previous experiments.

- In the first trial series retention aid was prepared in following way: 0,032grams of retention aid was diluted in 3000 ml of water and continual mixing was added. When retention chemical was added in to sheet making machine, the retention chemical liquid was added 30 ml in to the container.

- In the last trial, 0,1 g of retention aid was diluted in 1 liter of water and for each sheet 16 ml of this solution was taken.

The reason why amount of retention chemical was increased in the last trial was because it was noticed that 0,02 % of a sheet weight might have been too small amount of retention chemical for each sheet. This conclusion was reached when the article named “Potential use of micro-and nanocellulose as a reinforcing element in paper “written by Isko Kajanto and Mika Kosonen was studied deeply. Kajanto et al. researched the use of nanocellulose in a very similar experiment, and the amount of retention chemical they used in their studies was much higher: 1 % of total pulp amount. Therefore it was decided to perform additional test series with larger amount of retention agent.

3.2.5 Preparation of sheets for physical testing ISO 5269-1:2005(E)

The final pulp solution for papermaking was constantly mixed during the whole procedure in order to ensure that pulp and microcelluose were mixed evenly. A sample of 0,8 liter was taken from the pulp solution and poured into container of the sheet former and mixed. Water was removed from the container. The pulp remained on the wire (100-mesh screen). The sheet was removed from the net by laying two blotters upon the wet pulp and then applying weight. This makes the blotters suck in remaining water.

3.2.6 Pressing

Pressing process was carried out using Lorenzen & Wettre (type 964034) device. Blotters with the layer of pulp were put upon each other and then transferred to the pressing machine and pressed for 4 minutes. In accordance with ISO 5269-1:2005, the pressing has to be done two times, but in this case it was done only once. Also, the standard advices that the blotters from the sheet making process should be changed before doing the pressing, but in this case blotters were not changed. They were changed only before drying process.

Pressing process was carried out using Lorenzen & Wettre (type 964034) sheet pressing device. Blotters with the sheet were put upon each other and then transferred to the pressing machine and pressed for 4 minutes under pressure of 0,4 bar. In accordance with ISO 5269-1:2005, the pressing has to be done two times, but in this case it was done only once. Also, the standard advices that the blotters from the sheet making process should be changed before doing the pressing, but in this case blotters were not changed. They were changed only before drying process.

3.2.7 Drying

Drying was done by using Oy E. Sarlin AB (type KOL-K) drum dryer. Used blotters were changed to new ones. The drying time for the sheets was set to 4 hours which was the final step of sheet making.

3.3 Paper testing

Tests of sheets were performed in order to observe changes in paper properties that were caused during papermaking by additional cellulose and retention agent. Moisture content has a significant effect on paper quality, therefore paper has to be measured under standard conditions (temperature and humidity). This affects paper strength and other properties. (Biermann, Christopher J, 1996, 163). Tests were performed under standard conditions (temperature 23 °C, humidity 50 %) in accordance with ISO 187.

3.3.1 Basis weight of paper sheet

Basis weight is the mass of the paper or paperboard per square meter. Basis weight is important because it affects almost all paper and paperboard qualities (knowpap, basis weight).

Determination of sheet basis weight ISO 536:1995(E)

Sheets of paper were cut into 100×100 mm pieces according to the ISO 187 standard and weighted with scales. Obtained mass results are expressed in grams. To calculate the basis weight, which is expressed in grams per square meter (g/m^2), the following equation is used:

$$g = \frac{m}{A} \times 10\,000$$

where

g is basis weight

m is the mass of a test piece, in grams

A is the area of a test piece, in square centimetres

Every sheet of paper was cut into 100×100 mm pieces and was weighted. The final results of the test are based on average that was calculated for each paper grade from weighing 3 to 5 sample pieces.

3.3.2 Tensile strength

The tensile strength is defined as a strongest loading rate that paper or paperboard can withstand without breaking (tensile strength, knowpap).Tensile strength is expressed as kilonewtons per meter (kN/m).

There are various factors that affect tensile strength. The papermaking website knowpap gives us several factors: One of them is refining time of the pulp. The more pulp is refined, the more fibers have ability to bond with each other that leads to improvement of tensile strength. However, tensile strength starts to decrease after certain refining time. Therefore over-refining must be avoided. Second factor affecting paper strength is quality of fibers. Good tensile strength is achieved when using pulp containing long fibers with micro- or nanofibrils. Third factor is basis weight, because the higher the basis weight, the more there are fibers that bond with each other.

Fourth factor is formation. Formation tells the small-scale variation of basis weight. If variation is high, some areas of the paper sheet have weaker tensile strength than others. This lowers overall strength. (knowpap, tensile strength)

Tensile strength ISO 1924-3:2005

The test was performed using Lorentzen & Wettre tester (Lorentzen & Wettre, type 462489). Sheets were cut into sample pieces of 150 mm long and 15 mm wide. Results for each paper grade were based on average value of 6 to 10 tested samples.

3.3.3 Roughness

Roughness tells the unevenness of the paper surface. There are various levels of roughness that appear on paper surface: macro roughness, microroughness and optical roughness. Macro and micro roughness affect printing quality, and optical roughness has effect on gloss. There are different methods to measure roughness, depending on the level of roughness. In this thesis discuss only macro roughness is discussed, which is the most commonly measured by using a method which is based on air leak (knowpap, roughness).

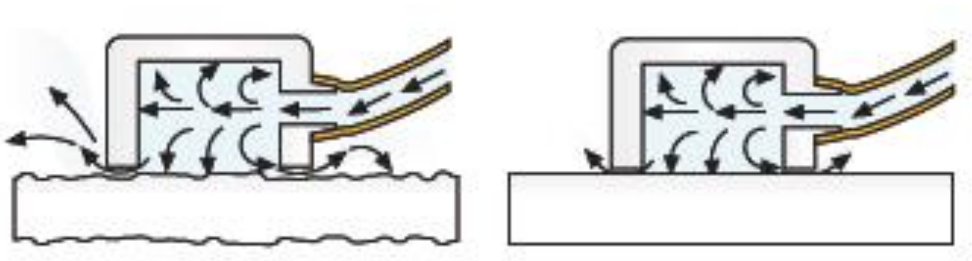

FIGURE 5. Bendsten principle for roughness testing (Europapier International GmbH 2007).

The principle of method is that inverted cup is placed on top of the sample. If the paper sample is rough, the air tends to leak out from the cup. The rate at which air flows outside the cup corresponds to the roughness of the paper. Thus roughness is expressed ml/min (Monica Ek, Göran Gllerstedt & Gunnar Henrikson 2009, 213).

Roughness test ISO 8791-2

Sample sheets were tested with Bendsten method (air leak) where Lorentzen & Wettre (type 967361) equipment was used. Four samples of each setpoint were tested from both sides.

3.3.4 Optical properties

Optical properties and measurements of paper are based on transmittance, reflectance and absorbance. Reflectance is the ratio of reflected light intensity (amount of radiation) by a surface to incident light intensity. Transmittance and absorbance are the ratios of transmitted and absorbed light intensities to incident light intensity (Borch, Richard E. Mark 2002, 98).

Opacity (paper backing) requires only one sheet of paper. It is the ratio between amount of reflected light from a single sheet with black backing to the amount of light reflected by the same sheet backed by opaque pad (Borch, Richard E. Mark 2002, 102).
The reflectance factor of a sheet of paper over black is R_0 and reflectivity is R_∞ that is reflectance factor of a material pad of certain thickness where is no change in reflectance when the pad thickness is decreased or increased. Fiber intensity and orientation can affect reflectivity (Monica Ek 2009, 159). Whiteness is expressed as WI CIE D65/10 + UV in this thesis.

3.3.5 Thickness and density

Thickness of paper is measured as perpendicular distance between the two surfaces of paper under certain conditions because moisture and temperature can affect final result. Basis weight, pressing, beating and refining affect thickness. (Scott, William E., 1995, 55-56)

Measurement of sheet thickness (ISO 534:2005)

Paper sheets were cut into samples of 100 × 100 mm. From three to five samples of very paper trial point specimen were tested once or twice at different areas. Results are expressed in µm.

3.3.6 Zeta-potential

Zeta potential is a key indicator for stability of colloidal dispersions. The magnitude of zeta potential tells how much there are inner repulsion forces within the liquid. Zeta potential can be expressed either a negative of or positive number depending on the electronic charge. (Wikpedia, zeta potential).

TABLE 4. Relation between zeta potential and stability of liquid (Wikipedia, zeta potential)

from 0 to ±5,	Rapid coagulation or flocculation
from ±10 to ±30	Incipient instability
from ±30 to ±40	Moderate stability
from ±40 to ±60	Good stability
more than ±61	Excellent stability

The higher zeta potential goes (higher repulsion forces), the more stable the colloid becomes. Likewise as the zeta potential decreases, the more colloid has tendency to flocculate.

Based on this fact, it was deduced that if zeta potential is high, bonding of fibers with each other is weaker because of the repulsion forces, and thus paper strength will be poor. If this is the case, it is necessary to add retention chemical, either anionic or cationic in order to manipulate internal electric charges of the colloid.

During the tests it was noticed that tensile strength had tendency to become lower as addition of MFC was increased. It was suspected that microcellulsoe fibers were either anionic or cationic, and the increase of microcellulose could cause more repulsive forces within pulp. To analyze this, zeta potential measurement was performed. Zeta potential results showed rather high anionic tendency, which then demanded the use of retention chemicals.

Zeta potential measurement

The measurements were done using BTG Mütek system. The machine had to be disassembled, cleaned, dried and assembled after every measurement. Cleaning and drying was very important because traces of previous samples could affect next measurements.

3.3.7 Pearson correlation

Correlation is one of the most popular statistical methods of data analysis. Pearson correlation coefficient, *r*, is used to measure strength of a linear association between two variables. The value r = 1 means a perfect positive correlation and the value r = -1 is a perfect negative correlation thus the data will lie on the same line (Jeremy Stangroom 2013).

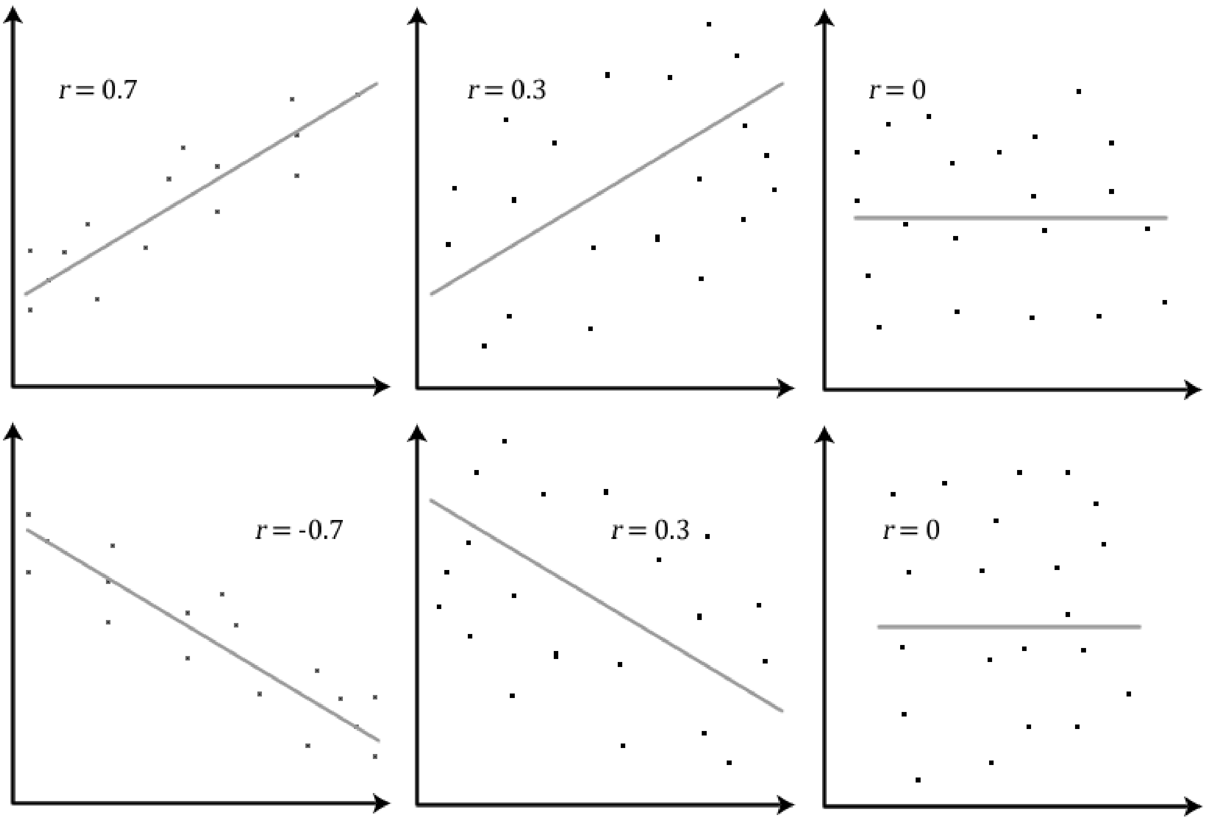

FIGURE 6. Strength of data correlation, positive and negative *r* value. (Lauren Waters 2013)

Correlation test was performed because it was important to know if basis weight of a paper sheet correlates with other properties of the paper sheet. Change of microcellulose amount in trial points may have had an effect on basis weight. Correlation analysis showed which paper properties got affected by microcellulose through basis weight.
The correlation analysis was carried out in MS Excel using Pearson correlation formula. Average values of basis weight for each trial point were compared with average values of tensile strength, roughness, thickness and optical properties.

3.3.8 Test series

In total there was 9 different test series that where done over few months. Each of the series served a different function, for example the first two test series was more about finding and identifying possible problems in sheet making and testing and also finding a propriate range on which to add microcellulose.

TABLE 5. Trials

Date	**Microcellulose grade (µm)**	**Pulp type**	**Retention aid**	**Notes**
24.3	8	Birch	-	Pretest was performed in order to define appropriate range for adding microcellulose (0-25%).
20.3	200	Birch	-	Second pretest. Two additional microcellulose trial points were added: 35% and 50%.
9.4	200	Birch	-	Zeta potential was measured in this test in order to see whether there was need for retention chemical.
16.4	200	Birch	0,05%	Retention chemical was added.
22.4	30	Pine	0,05%	Pine was used in this test to compare its paper sheets with birch sheets from other trials.
24.4	30	Birch	0,05%	Smaller particle size MC was used to compare effects of the retention chemical with previous tests.
29.4	200	20% pine+80% birch	0,05%	Paper sheets made from mixture of pine and birch pulp.

29.4	200	20% birch+80% pine	0,05%	Paper sheets made from mixture of pine and birch pulp.
15.4	200	Birch	1%	Last test containing increased amount of retention aid

4 RESULTS AND CONCLUSIONS

Amount of data in this work is vast, therefore in this conclusions part only the most significant findings and results are shown. Results from each individual tests can be found in appendices part.

4.1 Basis weight

In every trial, basis weight was tested due to its importance in further analyses of paper properties. It was noticed that in trials, where BE 600-30 (30 µm) and UFC 100 (8 µm) cellulose grades were used, basis weight of sheets was getting lower. The size of the wire was bigger than microcellulose particles, consequently 30 µm and 8µm cellulose types caused decrease in grammage of sheets.

If one tries to make sheets by using only microcellulose without pulp with standard concentration of 2g/l, then retention for 8 µm cellulose is close to zero, in other words nothing stays on the wire but goes through. In case of trials with 30 µm, retention was close to zero. Cellulose of 200 µm particle size stayed on the wire, but its retention was still quite poor. The size of the wire that was used in this test was around 120 µm (100-mesh screen). Obviously, particles with length of 8 µm and 30 µm go through the wire easily.

If one tries to make sheets by using only microcellulose without pulp with standard concentration of 2g/l, then retention for 8 µm cellulose is zero, in other words nothing stays on the wire but goes through. In case of trials with 30 µm, retention was close to zero. Cellulose of 200 µm particle size stayed on the wire, but its retention was still quite poor. The size of the wire that was used in this test was around 120 µm (100-mesh screen). Obviously, particles with length of 8 µm and 30 µm go through the wire easily.

4.2 Tensile strength

Based on the methods and results, the tensile strength drops as addition of microcellulose to pulp is increased. This is completely opposite to the findings of Kajanto et al. who saw in their studies that nanocellulose, in fact, improves tensile strength. The reason why tensile strength decreases as the amount of microcellulose is added is that if more microcellulose is added to the pulp solution, and microcellulose does not stay in pulp, it leads to drop in sheet grammage, and thus tensile strength decreases. Even though retention chemicals were used in some experiments, the tensile strength still had tendency to go lower. Therefore, it was decided to perform an experiment with increased amount of retention aid.
Even in this test series it was clearly seen that the more microcellulose was added the more tensile strength got lower, even though retention chemical amount was much higher. To sum up, this specific MFC lowers the tensile strength rather than improves it.

4.3 Roughness

Roughness of sheets appears to increase as 200µm microcellulose is added into the pulp. This increase of roughness is not seen in other test series of sheets containing 30 µm and 8 µm cellulose. Apart from the calendering and coating, the main factor affecting roughness is patterning phenomena that pulp causes on the paper surface. Presumably, 200µm cellulose causes certain type of patterning phenomena that impacts formation of sheet on the wire surface, and thus increasing unevenness of paper surface. The reason why roughness does not increase when 8µm and 30 µm microcellulose are added in to pulp is probably because of the poor retention. The fibers are so small that they slide between bigger fibers and go through the wire.

4.4 Zeta-potential

It was found out that values of zeta potential were high and varied between -49 – (-60) mV. This indicates strong repulsion force between the fibers that means that retention chemical could help. In most cases, use of retention chemical increased tensile strength as expected, but not significantly. Also, despite increased amount of retention chemical, the tensile strength still continued to get lower as MC was added. The reason for this was already speculated in the tensile strength chapter. The main reason for this was already mentioned which was the poor retention.

4.5 Comparison trial of microcellulose grades

Along with the original method of papermaking and microcellulose addition levels, another test was carried out in accordance with standard instructions mentioned in paper making section in order to compare different microcellulose grades and their effect on paper properties. In this case every sheet of paper has the same amount of pulp and microcellulose, where 2 g of each cellulose (200 µm, 30 µm and 8µm) grade was mixed with 200 ml of water separately (100 ml of microcellulose solution was added to the container when making paper hence each paper sheet got 1 g of microcellulose on top of basic pulp concentration) and two sheets containing each microcellulose grade were made and tested.

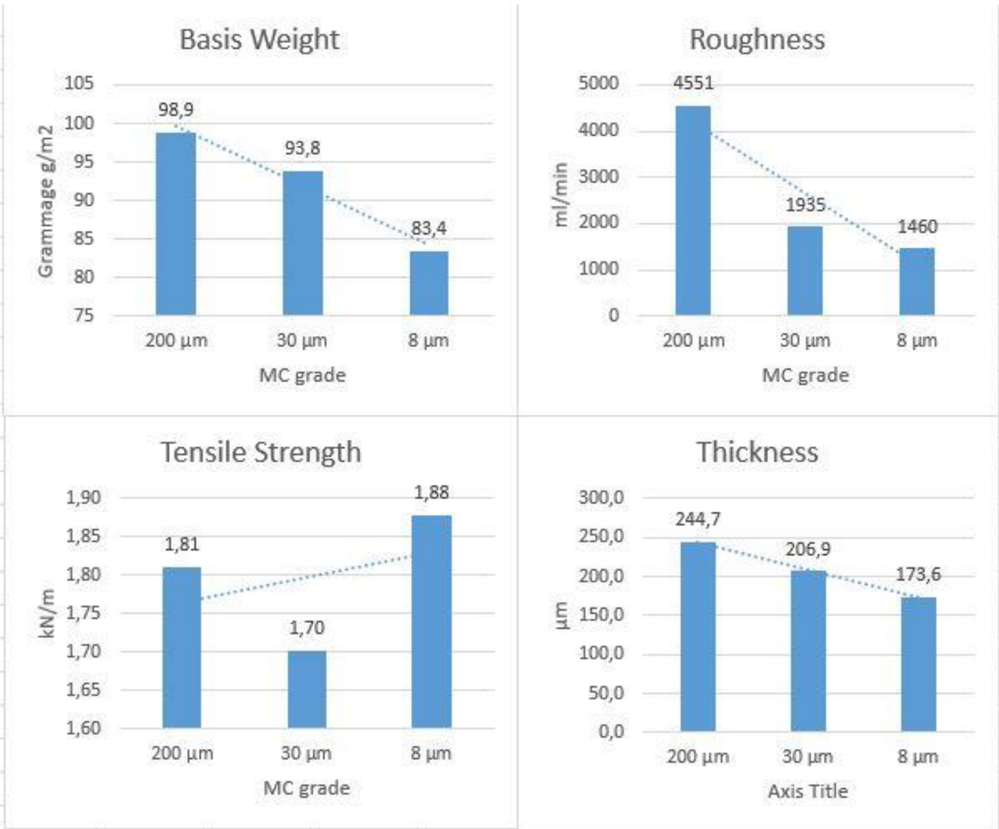

FIGURE 7. Comparison of general properties of paper made with addition of different cellulose types.

This test proves that basis weight and roughness are affected by size of wire screen of the sheet former. However, grammage of sheets with 8µm cellulose is lower than grammage of sheets with 200 µm, tensile strength of sheets with 8 µm is slightly higher, which means that 8 µm cellulose is probably better than 200 µm sized cellulose when it comes to tensile strength.

4.6 Comparison of pine and birch species

Pine has much higher tensile strength than birch, but even when pine and microcellulose were combined, it was clear that tensile strength value decreased as the microcellulose was added. Opacity is slightly higher in case of birch pulp, and in sheets made with birch and pine pulp opacity increased slightly when using retention aid. In case of combination of two pulp types, where pine pulp has stronger tensile strength than birch, it reinforces the weaker birch that leads to moderate stability in tensile strength. The use of retention agent only slightly improves the tensile strength.

In case of combination of two pulp types, where pine pulp has stronger tensile strength than birch, it reinforces the weaker birch that leads to moderate stability in tensile strength. The use of retention agent only slightly improves the tensile strength.

4.7 Overall conclusion

In general, the used methods of applying microcellulose and retention agent in papermaking did not lead to positive results. The main issue in this experiment could have been the wire of the sheet former that seemed to be too big, especially for the MC with 8 µm and 30 µm particle size, because their retention was approximately zero. The 200 µm cellulose partially retained, but still its retention was quite weak. To get more accurate results, different types of sieves with smaller net size should be used in future.

Manufacturing method of the microcellulose should be taken into account because it is important to know whether the MC has lost nanofibrils during processing. They ensure better fiber bonding and paper strength. Certain grades of cellulose are used in medicine and construction chemical which means that the cellulose may have different properties.

In case of microcellulose comparison trial, where the MC was added to pulp in the sheet former, it was proved that these specific microcellulose types degraded tensile strength of paper sheet even when basis weight of sheets containing the MC was significantly higher than original sheets that did not contain the MC. Consequently, these methods and grades of microcellulose would not be efficient in papermaking.

REFERENCES

Michael T. Postek, Robert J. Moon, Alan W. Rudie, Michael A. Bilodeau, 2013. Production and Applications of Cellulose Nanomaterials. TAPPI Press, USA.
Read 10.03.2015
http://www.tappi.org/Bookstore/0101R332Preview.aspx

Roger M. Rowell 2012. Handbook of Wood Chemistry and Wood Composites, Second Edition. Boca Raton: CRC press

Dufresne, A. 2012. Nanocellulose: from nature to high performance tailored materials. Berlin: de Gruyter.

Ek, M., G. Gellerstedt and G. Henriksson 2009. Pulp and paper chemistry and technology. Volume 2, Pulping chemistry and technology. Berlin, Walter de Gruyter

Ek, M., G. Gellerstedt and G. Henriksson 2009. Pulp and paper chemistry and technology. Volume 3, Paper chemistry and technology. Berlin: Walter de Gruyter

Hagiopol, C. and J. W. Johnston 2012. Chemistry of modern papermaking. Boca Raton, CRC Press.

Jens Borch, Richard E. Mark 2002. Handbook of physical testing of paper, Volume 2. New York

William E. Scott and James C. Abbott, 1995. Properties of Paper: An Introduction, Second Edition. Atlanta (Ga): TAPPI Press

Biermann, Christopher J 1996. Handbook of Pulping and Papermaking. San Diego: Academic Press

Wikipedia, Zeta potential. Read: 11.5.2015
http://en.wikipedia.org/wiki/Zeta_potential#cite_note-1.

Isko Kajanto, Mika Kosonen 2012. Micro- and nanocellulose fiber study article: Potential use of micro- and nanofibrillated cellulose as reinforcing element in paper. Journal of science and technology for forest products and process.

Christophe Baley 2007. High-performance natural fibers. JEC magazine #37.

Read: 10.04.2015
http://www.jeccomposites.com/news/composites-news/high-performance-natural-fibres

Europapier International GmbH 2007. Surface characteristics. Vienna. Read: 10.05.2015
http://www.archive.europapier.com/service/knowhow/testingpaper/surface-characteristics

Kremer Pigmente GmbH. Read: 05.04.2015
http://www.kremer-pigmente.com/media/files_public/59770e.pdf

Vento Co., Ltd: Read 05.04.2015
http://www.vento.com.vn/docs/vi/Cellulose%20fiber/BE600-30.pdf

Lauren Waters 2013. Pearson Correlation Coefficient. Westminster College.
Read: 01.05.2015
https://statsmethods.wordpress.com/2013/05/10/pearson-correlation-coefficient-r/

Jeremy Stangroom 2015. Pearson Correlation Coefficient. Read: 01.05.2015
http://www.socscistatistics.com/tests/pearson/

APPENDICES

Appendix 1. Datasheets of cellulose

KREMER
PIGMENTE

59770 Arbocel® BWW 40

Arbocel® are environmentally friendly cellulose fibers which are gained from renewable raw materials.

Technical Information:

Raw Material:	pure cellulose
Description:	medium sized fibers, white
CAS No.:	9004-34-6

Physical and chemical Properties:

Cellulose content:	approx. 95 %
Average fiber length:	200 µm
Average fiber thickness:	20 µm
Bulk weight:	110 g/l - 145 g/l
Whiteness (absolute value at 461 nm):	86 % ± 5 %
Ignition loss (850°C, 4 h):	approx. 0.3 %
pH-Wert	6 ± 1

Sieve Analysis

Sieve residue:	300 µm	100 µm	32 µm
	max. 0.5 %	max 20 %	max. 65 %

Application:
Arbocel® can be used as thickening agent, as reinforcing agent, as absorbent and extender, or as filling agent.

Page 1 of 1

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kremer-pigmente@t-online.de, www.kremer-pigmente.com

FIGURE 8. Datasheet of Arbocel BWW 40 **(**Kremer Pigmente GmbH)

Data sheet

Grade
BE 600-30

ARBOCEL®

natural cellulose fibres

Basic raw material

highly pure cellulose

Characteristic

micro fibre, white

Physical and chemical properties

cellulose content	approx.	99.5 %
average fibre length		30 µm
average fibre thickness		18 µm
bulk density		200 g/l - 260 g/l
whiteness (absolute value at 461 nm)		85 % +/- 5 %
residue on ignition (850 °C, 4 h)	approx.	0.3 %
pH-value		6 +/- 1

Screen analysis

Screen residue (in accordance with DIN 53 734/air jet sieve) with an interior mesh aperture of:

71 µm	32 µm
max. 0.1 %	max. 3 %

As with all natural products slight differences to the above given values may arise.

General references

ARBOCEL® cellulose fibres are environment friendly products, gained from replenishable raw materials.
Among other things, they are used as thickeners, for fibre reinforcement, as an absorbent and diluent or as a carrier and filler in most manifold application fields.

CAS-Nr.: 9004-34-6

J. Rettenmaier & Söhne GmbH + CO
Fibers designed by Nature
Holzmühle 1
D-73494 Rosenberg

FIGURE 9. Datasheet of Arbocel BE 600-30 (Vento Co., Ltd)

Appendix 2. Results of paper testing

1 Pretest of birch sheets with 8 µm cellulose, 24 March.

TABLE 6: Basis weight

	0%	5%	10%	15%	20%	25%	0E %
1	62	64,7	63,8	56,8	57,3	60	66,5
2	62	62	62,5	60	55	59,5	65
3	61,5	63	62,8	58	57	59,49	67
4	62,7	63	61,6	60,6	57	59,18	66
5	62,7	62	63,3	59	56,2		65
AVG	62,18	62,94	62,8	58,88	56,5	59,5425	65,9
SD	0,52	1,10	0,83	1,53	0,93	0,34	0,89

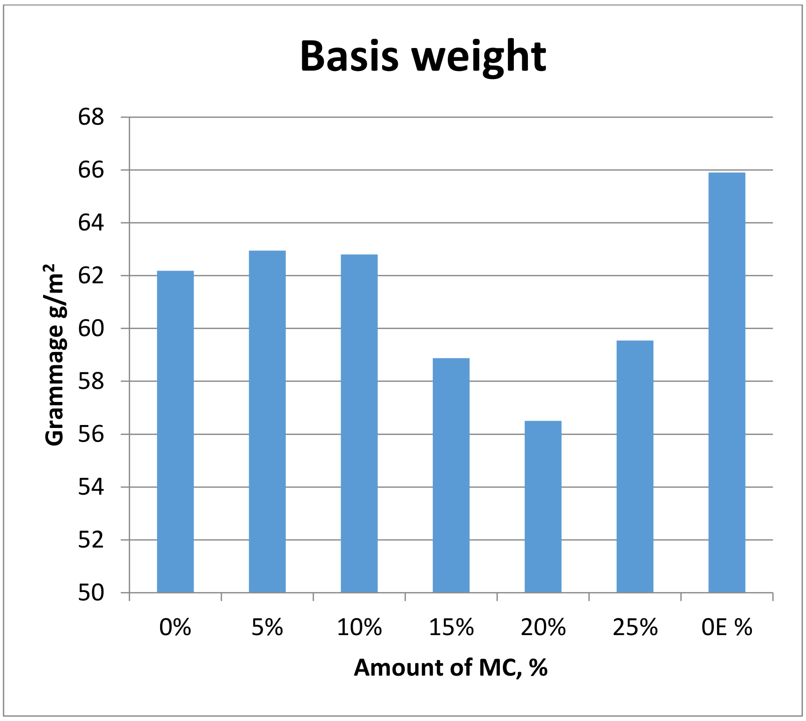

FIGURE 10. Basis weight

TABLE 7. Tensile strength

	0%	5%	10%	15%	20%	25%	0E %
1	3,15	2,92	2,91	2,31	2,33	2,18	3,2
2	2,86	3,16	2,53	2,3	2,28	1,31	3,24
3	3,12	2,93	2,68	2,66	2,31	2,03	3,39
4	3,43	3,02	5,54	2,71	2,38	1,43	3,4
5	3,17	3,11	2,74	2,54	1,85	1,81	2,79
6	3,43	2,97	2,63	2,35	2,42	1,91	3,28
7	3,52	2,68	2,79	2,48	2,23	2,22	3,27
8	3,33	2,93	2,87	2,35	2,13	1,99	3,2
9	3,51	3,25	2,78	2,41	2,03		3,47
10	3,55	3,14	2,79	2,61	1,96		
AVG	3,31	3,01	3,03	2,47	2,19	1,86	3,25
SD	0,22	0,16	0,89	0,15	0,19	0,33	0,20

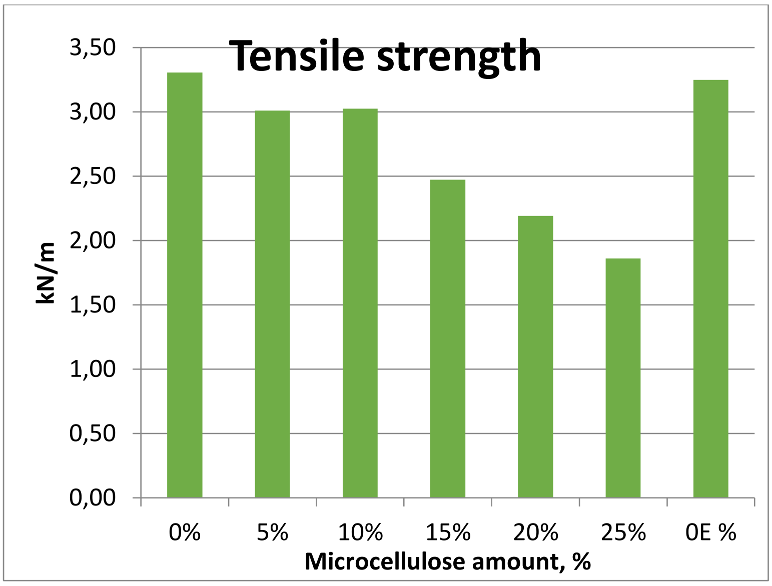

FIGURE 11: Tensile strength

2 Test of birch sheets with 200 µm MC, 9 April.

TABLE 8. Basis weight

	0%	5%	10%	15%	20%	25%	30%	50%
1	62,2	64,5	61,5	61,4	63,1	65	64,4	61,3
2	60,7	60	61,6	61,9	63,9	64,7	62,7	59,9
3	63	62,5	62,2	62,2	64,5	65,3	63,1	59,8
4	63,3	62,4	62,8	61,6	65,5	65,7	63,3	60,3
5	62,7	62,6	60,2	62,5	64,2	64,9	63,6	60,6
AVG	62,38	62,4	61,66	61,92	64,24	65,12	63,42	60,38
SD	1,02	1,60	0,97	0,44	0,88	0,39	0,64	0,61

FIGURE 12. Basis weight

TABLE 9. Roughness

	0 %	5 %	10 %	15 %	20 %	25 %	30 %	50 %
1	1522	1970	3390	3460	3379	3736	3429	3406
2	1307	1642	2733	3117	3133	3484	3569	4904
3	1040	1486	2613	2681	3961	3284	3680	4423
4	1664	2111	2221	2965	2749	3870	4113	3650
5	1220	2143	2112	2827	3218	4692	3782	3958
AVG	1350,6	1870,4	2613,8	3010	3288	3813,2	3714,6	4068,2
SD	246,49	292,42	505,67	299,04	441,77	540,81	258,47	602,08

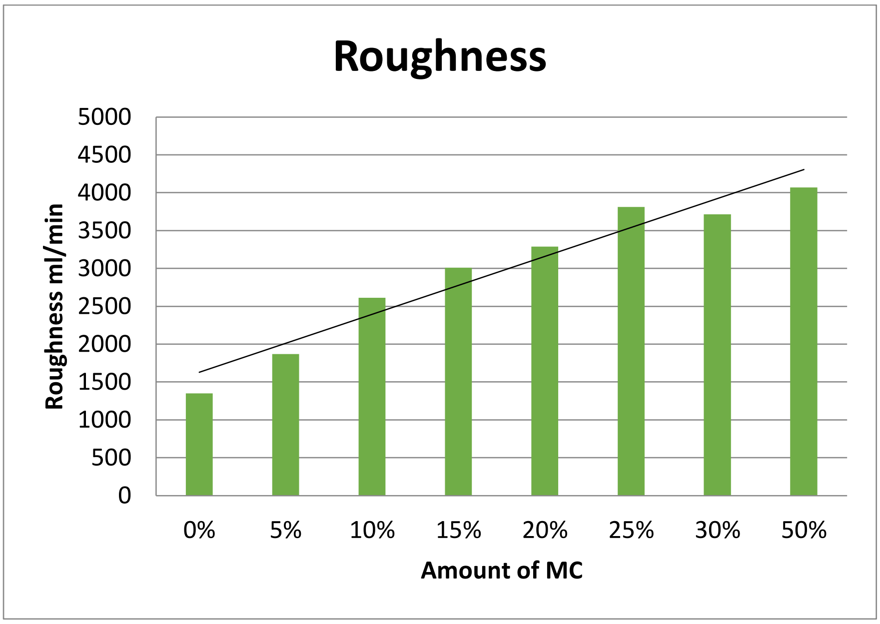

FIGURE 13. Roughness

TABLE 10. Thickness

	0 %	**5 %**	**10 %**	**15 %**	**20 %**	**25 %**	**30 %**	**50 %**
1	111,2	119,4	130,9	139,9	150,1	154,5	154,9	149,7
2	109,1	109,9	132	137,8	149,2	151,3	146	152,6
3	109,2	114,3	133	135,2	149,6	153,1	149	148,2
4	114,5	121,7	130,4	134,8	149,3	146	150,2	151,9
5	107,3	113,9	124,9	138,8	144,6	151,8	150	152,4
AVG	110,26	115,84	130,24	137,3	148,56	151,34	149,6	150,96
SD	2,74	4,70	3,15	2,23	2,24	3,23	3,20	1,93

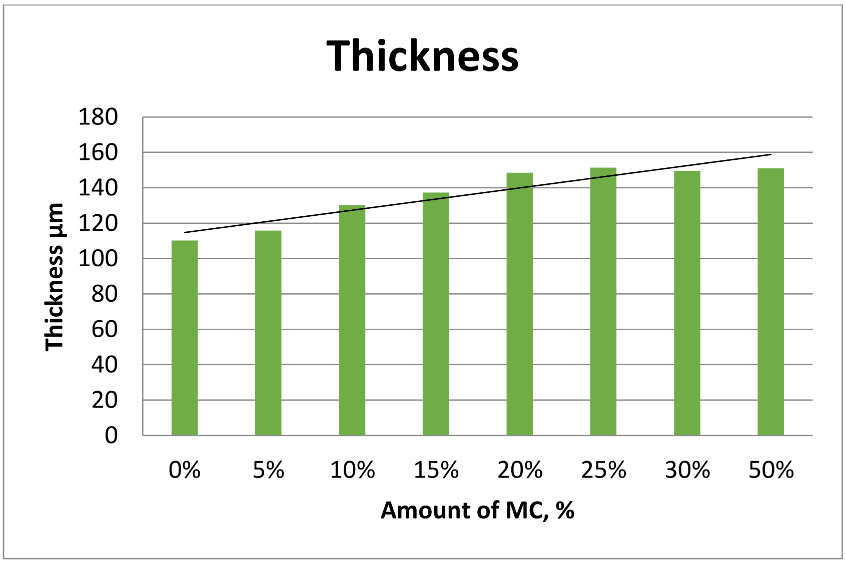

FIGURE 14. Thickness

TABLE 11. Zeta potential

mV	0 %	5 %	10 %	15 %	20 %	25 %	30 %	50 %
1	-49,7	-51,5	-52,1	-58,3	-56,2	-51,9	-51,4	-55,7
2			-54,6	-61,1	-56,1		-47,8	
3					-56			
AVG	-49,7	-51,5	-53,3	-59,7	-56,1	-51,9	-49,6	-55,7

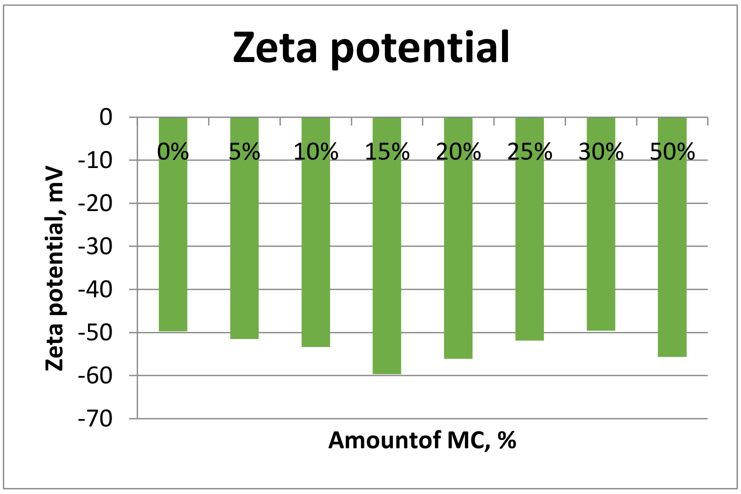

FIGURE 15. Zeta potential

TABLE 12. Optical properties

	0 %	5 %	10 %	15 %	20 %	25 %	30 %	50 %
Opacity	72,75	73,07	72,5	73,65	74,44	74,75	73,86	72,77
Transparency	32,37	32,2	32,58	31,73	30,88	30,87	31,47	32,53
Whiteness	72,59	72,26	72,04	71,67	72,13	72,36	72,54	72,64

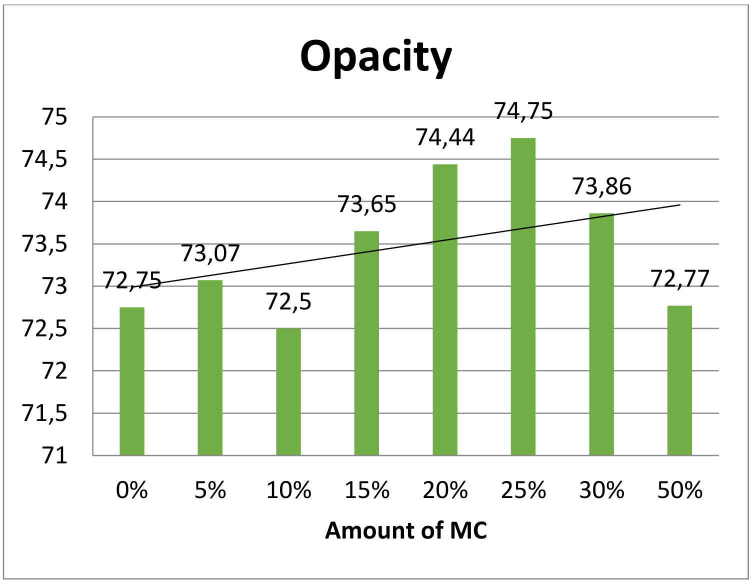

FIGURE 13. Opacity

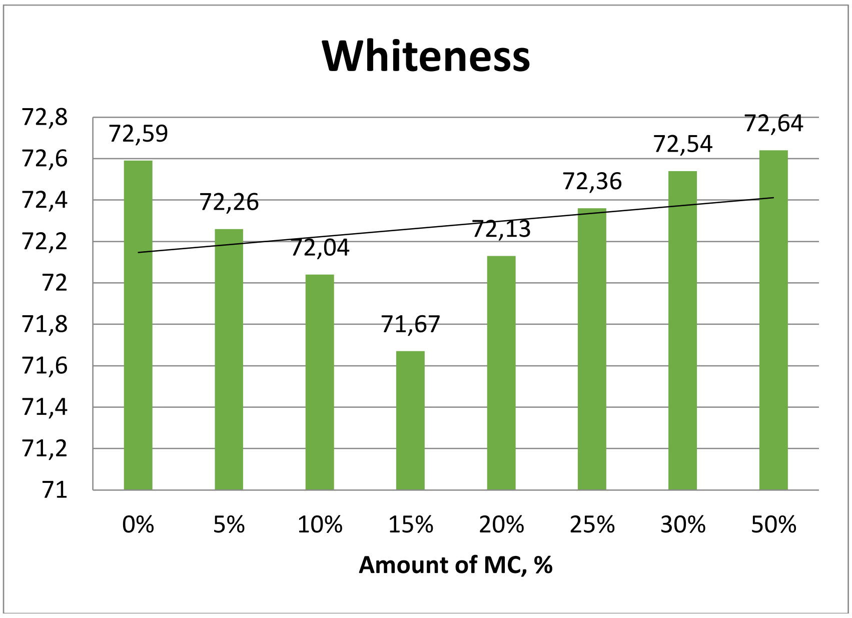

FIGURE 14. Whiteness

TABLE 13. Correlation analysis

	Weight	Tensile Strength	Roughness	Thickness	Opacity
Weight		0,05	0,17	0,29	0,87
Zeta potential	0,287	0,280	-0,322	-0,334	-0,108

3 Test results of birch sheets with 200 µm cellulose, 16 April.

TABLE 14. Weight

	No retention agent					Containing retention agent				
	0 %	**5 %**	**10 %**	**25 %**	**50 %**	**0 %**	**5 %**	**10 %**	**25 %**	**50 %**
1	65,4	61,7	64,5	64	61	67	62,8	61	60	60
2	65,6	61	63,3	64	62,3	65	62,6	63,8	62	61,5
3	66,1	63,1	64,3	63,7	61,5	66,1	63,8	65	64,5	59
4	66,6	62	64,6	63,8	62,7	66,6	61,4	64,5		58
AVG	65,93	61,95	64,18	63,88	61,88	66,18	62,65	63,58	62,17	59,63
SD	0,54	0,87	0,60	0,15	0,77	0,87	0,98	1,79	2,25	1,49

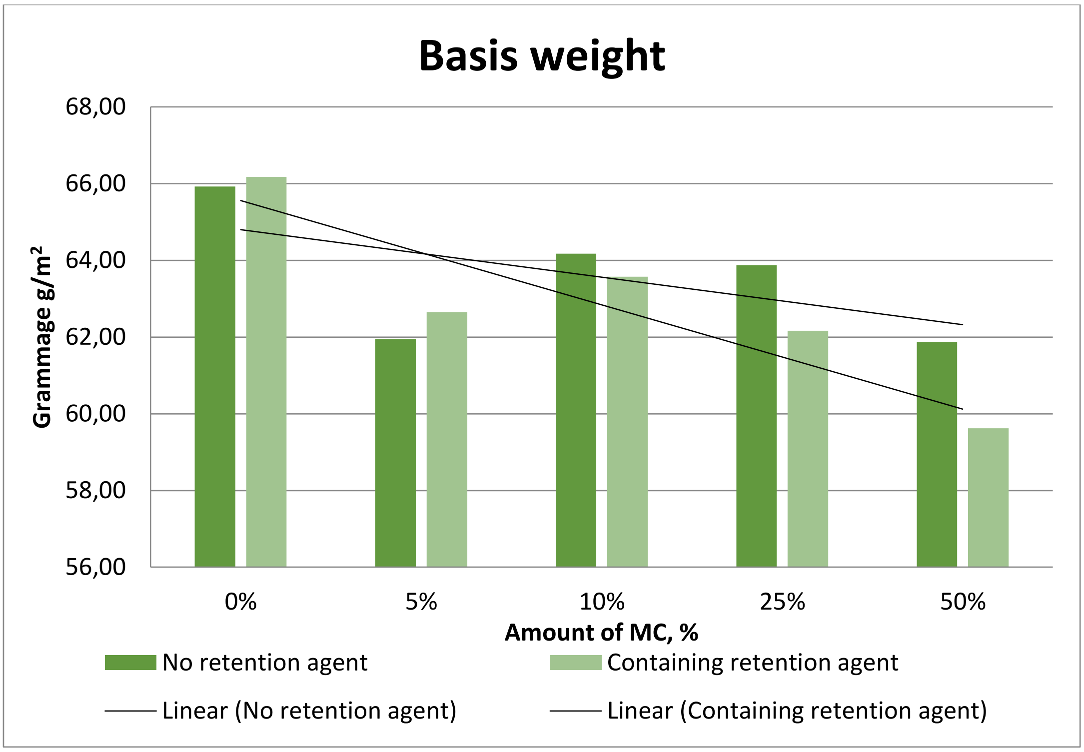

FIGURE 14. Basis weight

TABLE 15. Tensile strength

	No retention agent				
	0 %	5 %	10 %	25 %	50 %
1	2,78	2,18	2,14	1,84	1 1/3
2	2,88	1,84	2,28	1,34	1,44
3	2,21	2,31	2,09	1,84	1,42
4	2,67	2,42	2,08	1,79	1,4
5	2,92	2,22	1,99	1,54	1,16
6	2,52	2,34		1,69	1,43
7	2,78	2,36		1,45	1,42
8	2,65	2		1,74	1,12
AVG	2,68	2,21	2,12	1,65	1,34
SD	0,228	0,198	0,106	0,189	0,129

	Containing retention agent				
	0 %	5 %	10 %	25 %	50 %
1	2,56	2,32	2,21	1,84	1,27
2	2,77	2,22	2,27	1,95	1,53
3	2,84	2,49	2,23	1,9	1,4
4	3	2,04	2,36	1,93	1,52
5	2,67	2,2	2,35	1,86	1,51
6	2,81	2,37	2,43	1,81	1,46
7	2,59	2,47	2,03	1,95	1,36
8	2,58	2,53		1,84	1,48
AVG	2,73	2,33	2,27	1,89	1,44
SD	0,155	0,169	0,131	0,055	0,091

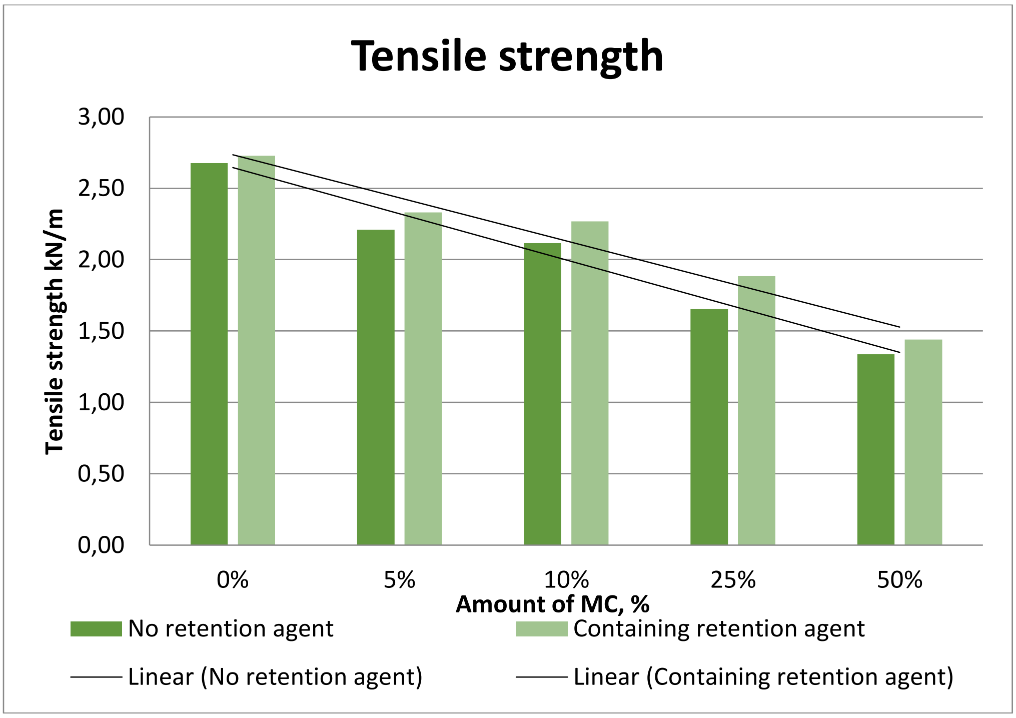

FIGURE 15. Tensile strength

TABLE 16. Roughness

	Without retention agent				
	0 %	5 %	10 %	25 %	50 %
1	1200	1153	2258	3942	4551
2	1266	1578	2617	3220	3813
3	1370	1989	2618	3564	4101
4	1290	1979	2039	3371	3764
5					
AVG	1281,5	1674,8	2383,0	3524,3	4057,3
SD	70,2	397,0	285,2	312,1	361,2

	Containing retention agent				
	0 %	5 %	10 %	25 %	50 %
1	1300	1520	2252	3354	2958
2	1300	1588	1768	2654	3593
3	1507	1753	1937	2597	3383
4	1324	1642			
5		2288			
AVG	1357,8	1758,2	1985,7	2868,3	3311,3
SD	100,1	308,2	245,6	421,6	323,5

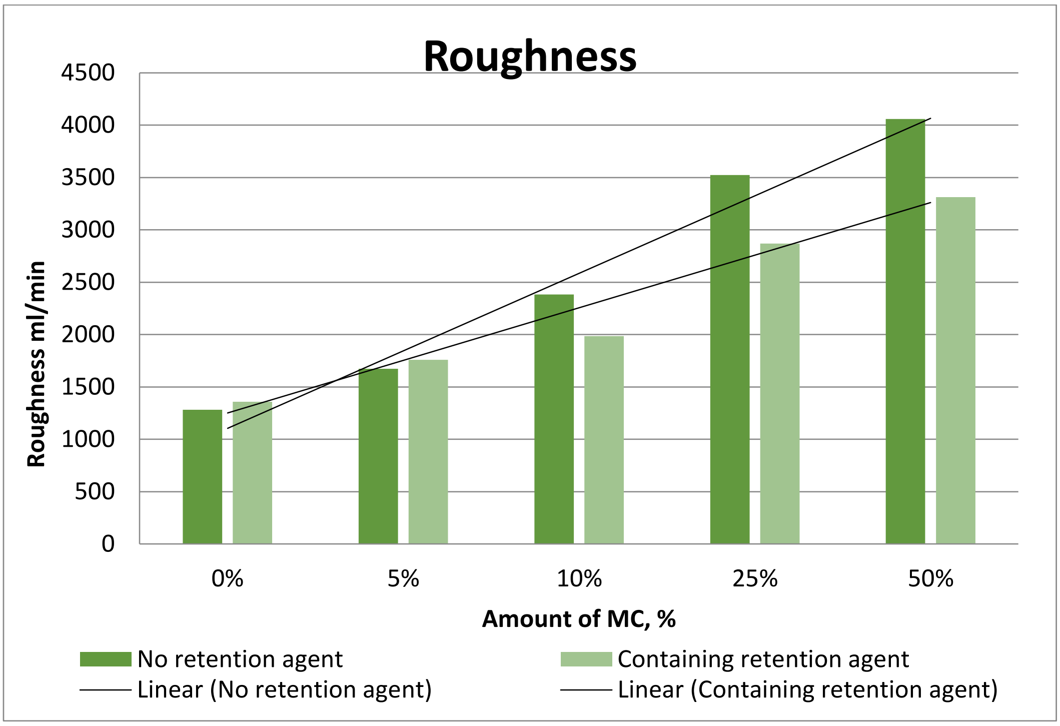

FIGURE 16. Roughness

TABLE 17. Thickness

Without retention agent					
	0 %	**5 %**	**10 %**	**25 %**	**50 %**
1	124,5	130,4	143,6	155,8	164
2	128	133	142,2	155	167
3	127,1	130,7	140,6	153,5	159,6
4	129	128,6	136,7	156,5	161,1
5	124,6				
6					
AVG	126,6	130,7	140,8	155,2	162,9
SD	2,02	1,81	2,98	1,29	3,27

Containing retention agent					
	0 %	**5 %**	**10 %**	**25 %**	**50 %**
1	123,1	139,4	140,4	154	157,2
2	127,1	132,6	140,2	161,1	154,6
3	125,5	124,9	138,6	157,4	152,6
4	122,4	131,8			
5		127,9			
6		127,8			
AVG	124,5	130,7	139,7	157,5	154,8
SD	2,17	5,11	0,99	3,55	2,31

FIGURE 17. Thickness

TABLE 18. Optical Properties

Without retention agent					
	0 %	5 %	10 %	25 %	50 %
Opacity		74	74,8	74,67	73,27
Transparency		31,01	30,36	30,56	31,64
Whiteness		73,09	72,94	72,98	74,96

Containing retention agent				
0 %	5 %	10 %	25 %	50 %
75,42	75,1	74,86	75,02	73,67
29,79	30,35	30,49	30,45	31,96
72,11	71,5	72,52	72,04	71,64

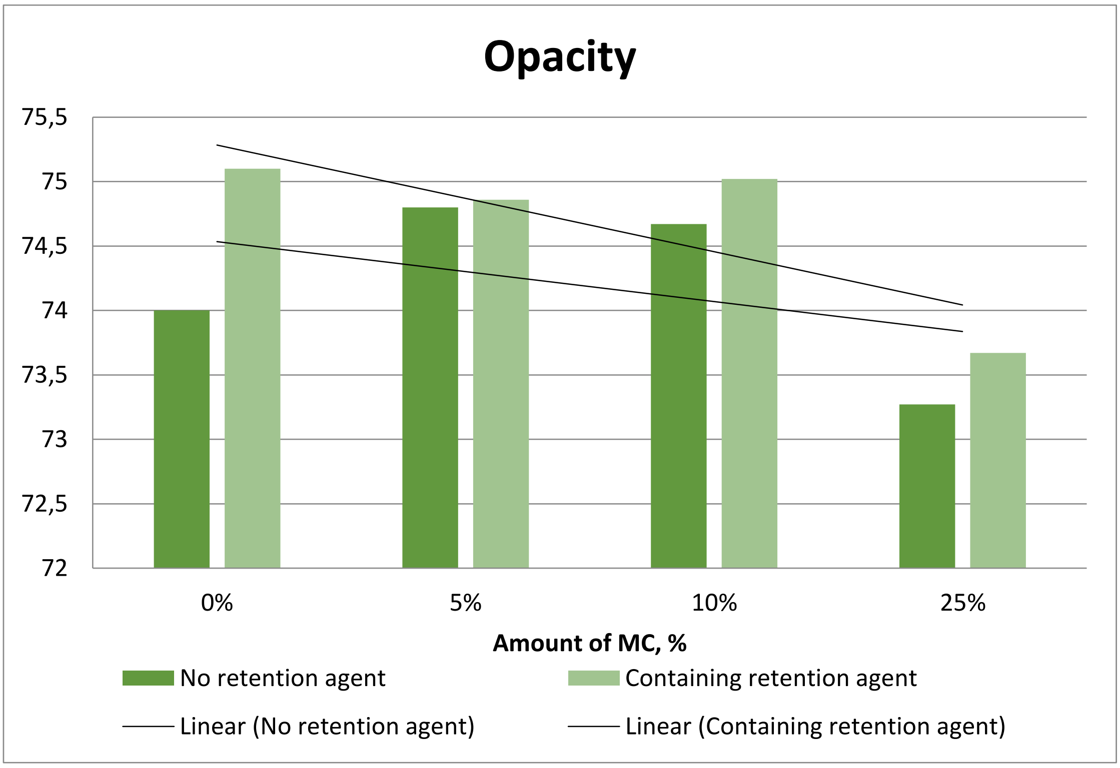

FIGURE 18. Opacity

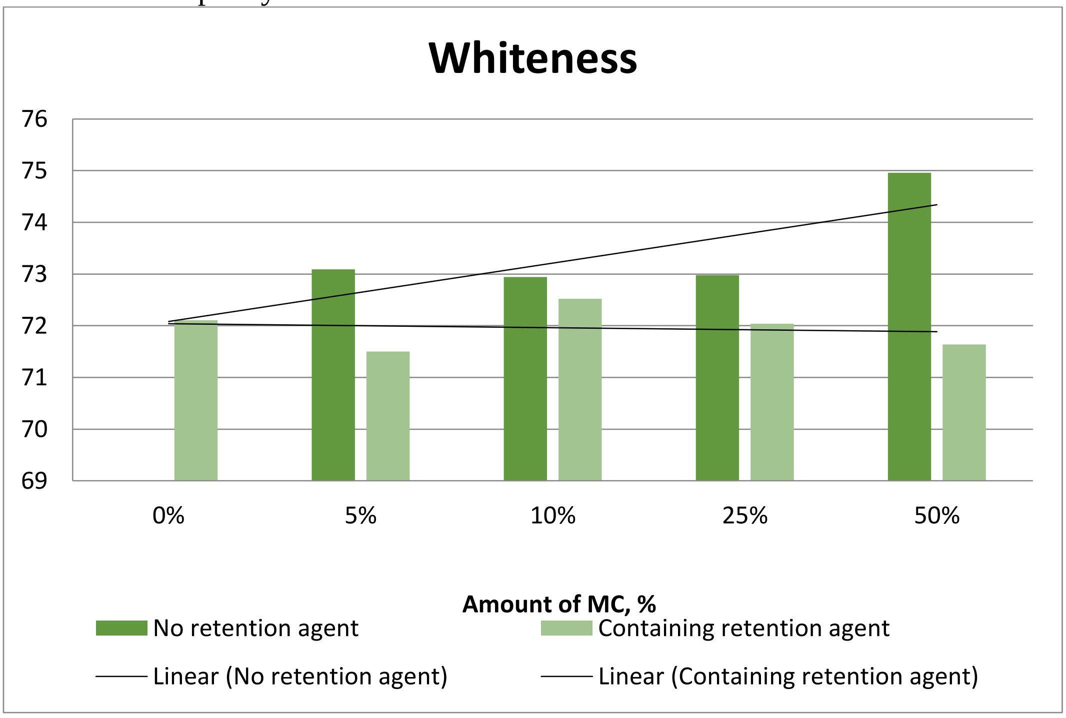

FIGURE 19. Whiteness

TABLE 19. Pearson correlation

	Thickness	Tensile	Roughness	Opacity
Weight	-0,62	0,77	-0,59	0,73

4. Test results of pine sheets with 30 µm cellulose, 22 April

TABLE 20. Basis weight

	Without retention agent					
	0 %	**5 %**	**10 %**	**15 %**	**20 %**	**25 %**
1	64	61	65,2	54,1	59	56,3
2	60,5	66	64,5	58,3	57	61,9
3	62,5	66	64,5	56,3	59,2	63,3
AVG	62,33	64,33	64,73	56,23	58,4	60,5
SD	1,76	2,89	0,40	2,10	1,22	3,70

	Containing retention agent					
	0 %	**5 %**	**10 %**	**15 %**	**20 %**	**25 %**
1	62,7	65,7	64,5	58,8	61	63,7
2	62,6	68,1	63	57,1	59,9	60,7
3	64,3	69,3	66,7	55,8	61,8	54,2
AVG	63,2	67,7	64,73	57,23	60,9	59,53
SD	0,95	1,83	1,86	1,50	0,95	4,86

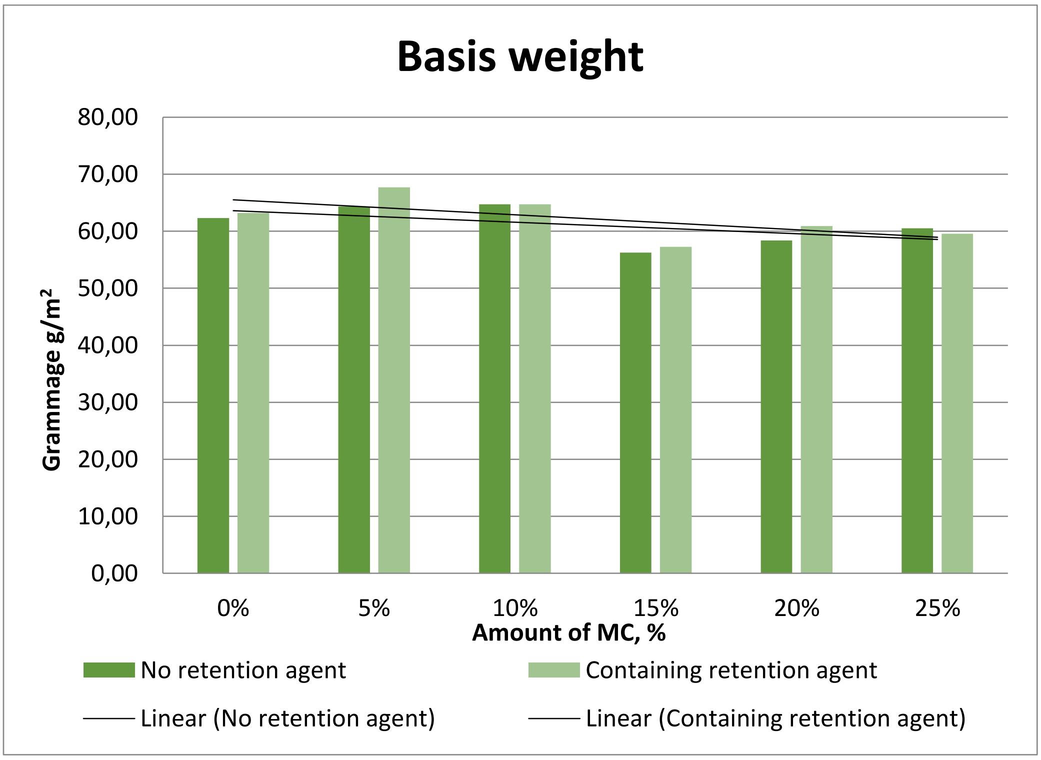

FIGURE 20. Basis weight

TABLE 21. Tensile strength

	Without retention agent					
	0 %	**5 %**	**10 %**	**15 %**	**20 %**	**25 %**
1	4,07	3,38	3,55	2,86	2,75	3,15
2	4,66	4,06	3,56	3,01	2,79	2,72
3	4,19	3,57	3,63	3,26	2,59	3,03
4	3,77	4,13	3,66	2,78	2,99	3,37
5	4,13	4,23	3,36	3,1	3,08	3,09
6	4,54	3,49	3,62	2,75	3,16	2,74
7	3,8	4,07	3,54	2,85	2,8	3,23
8	4,06	4		2,84	2,98	2,98
AVG	4,2	3,9	3,6	2,9	2,9	3,0
SD	0,32	0,33	0,10	0,18	0,19	0,23

	Containing retention aid					
	0 %	**5 %**	**10 %**	**15 %**	**20 %**	**25 %**
1	3,87	3,85	3,85	3,45	2,72	2,51
2	4,04	3,81	4,03	3,5	2,95	3,17
3	4,39	3,92	3,64	3,29	3,13	3,16
4	4,73	3,77	3,53	3,03	3,26	2,93
5	3,74	4,1	3,95	3,19	3,05	2,89
6	3,45	4,36	4,22	3,25	2,49	2,44
7	3,48	4,11	4,34	2,7	3,37	2,67
8					2,67	3,02
AVG	4,0	4,0	3,9	3,2	3,0	2,8
SD	0,47	0,21	0,29	0,27	0,31	0,28

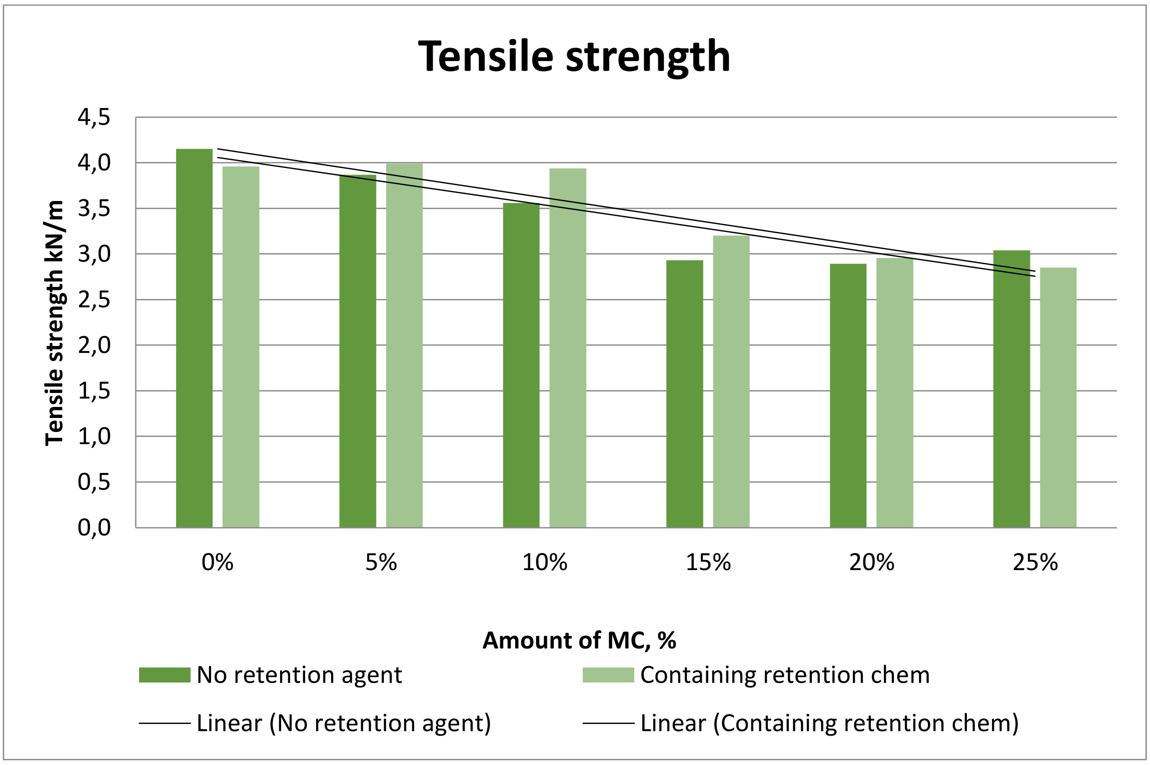

FIGURE 21. Tensile trength

TABLE 22. Roughness

	Without retention chemical					
	0 %	**5 %**	**10 %**	**15 %**	**20 %**	**25 %**
1	1779	2564	2145	1937	1642	2155
2	1539	2231	2012	2252	1838	2281
3	1865	2051	2443	2830	2077	2689
AVG	1728	2282	2200	2340	1852	2375
SD	169,0	260,3	220,7	452,9	217,9	279,1

	Containing retention chemical					
	0 %	**5 %**	**10 %**	**15 %**	**20 %**	**25 %**
1	1926	1951	1453	1566	1898	1665
2	1837	2532	2043	1668	1690	2510
3	1691	1545	2298	1617	1729	3025
AVG	1818	2009	1931	1617	1772	2400
SD	118,6	496,1	433,4	51,0	110,6	686,6

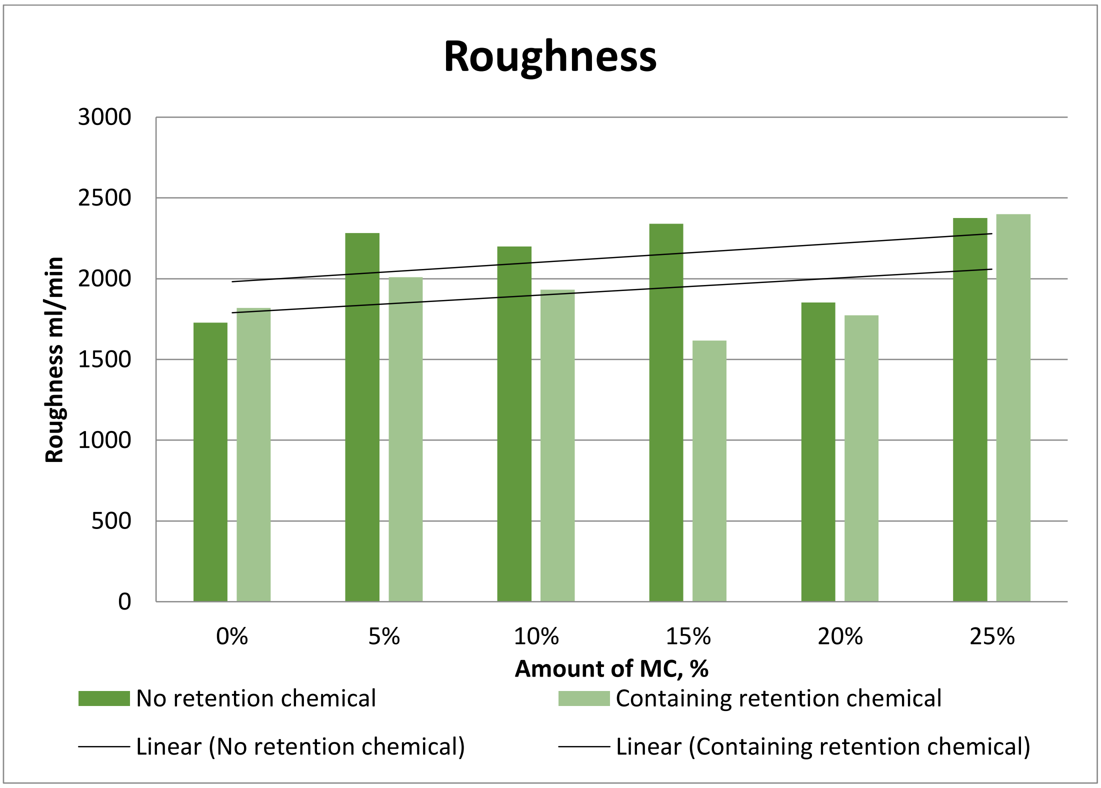

FIGURE 22. Roughness

TABLE 23. Optical properties

	Without retention aid					
	0 %	**5 %**	**10 %**	**15 %**	**20 %**	**25 %**
Opacity	64,52	68,33	68,89	66	66,6	67,9
Transparency	38,04	37,09	35,86	38,52	37,85	36,71
WI CIE C/2+UV	72,96	68,16	72,03	73,11	73,97	74,37

	Containing retention aid					
	0 %	**5 %**	**10 %**	**15 %**	**20 %**	**25 %**
Transparency	66,62	69,95	68,97	66,15	68,08	67,9

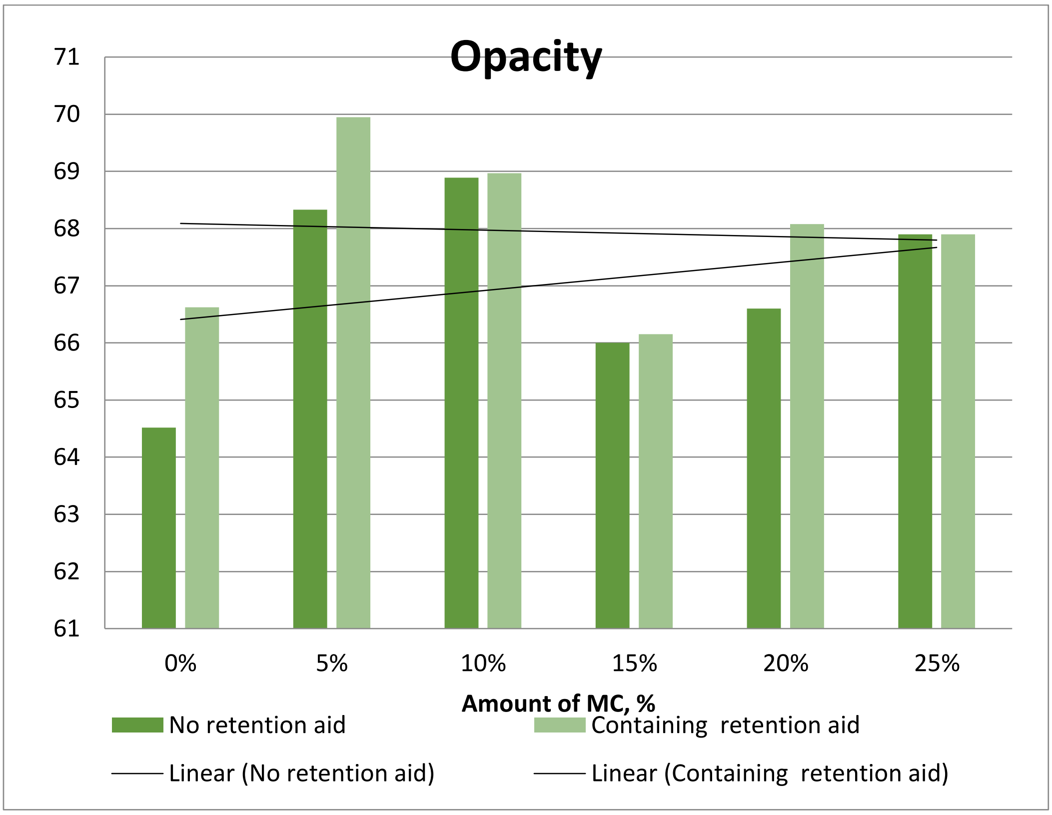

FIGURE 23. Opacity

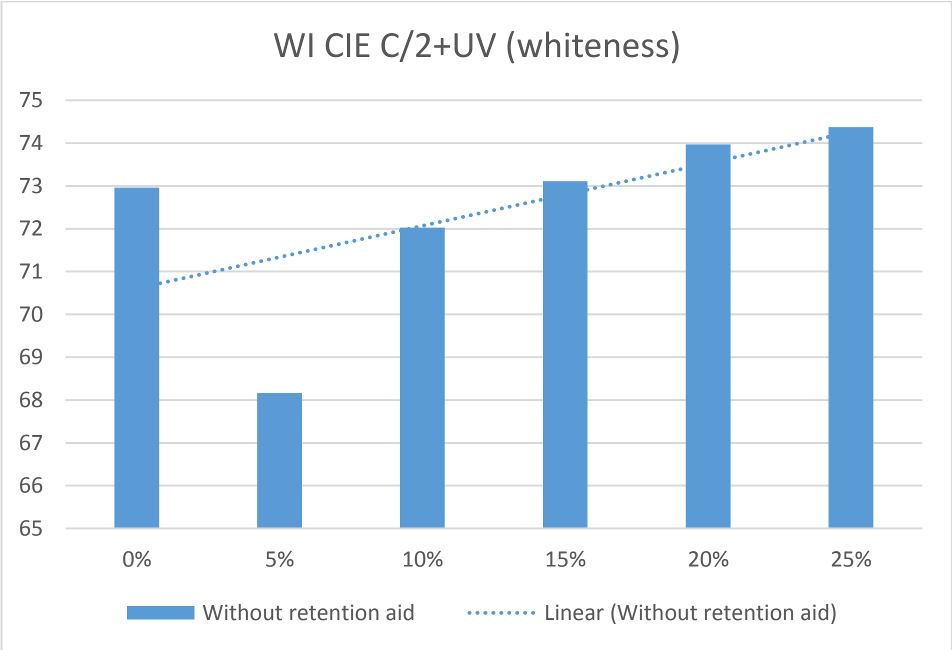

FIGURE 24. Whiteness

TABLE 24. Pearson correlation

	Tensile strength	Roughness	Opacity
Basis weight	0,787	-0,002	0,646

5. Test results of birch sheets with 30 µm cellulose, 24 April

TABLE 25. Basis weight

Without retention agent						
	0 %	**5 %**	**10 %**	**15 %**	**20 %**	**25 %**
1	61,8	62,3	61,6	59	61	60,9
2	61	62,9	62,9	61,6	60,3	59,6
3	64,2	61,6	61,6	59,4	59,9	58,2
4	59,5	60,7	61,8	59,9		60,2
AVG	61,6	61,9	62,0	60,0	60,4	59,7
SD	1,96	0,95	0,62	1,14	0,56	1,15

Containing retention agent						
	0 %	**5 %**	**10 %**	**15 %**	**20 %**	**25 %**
1	63,5	63	61,5	60,9	61,6	59,5
2	63,7	62	61,1	61,5	61,4	59,3
3	63,6	62,1	61,9	61,5	60,4	60,2
4	62	62,3	60,8		62,8	59,2
AVG	63,2	62,4	61,3	61,3	61,6	59,6
SD	0,80	0,45	0,48	0,35	0,98	0,45

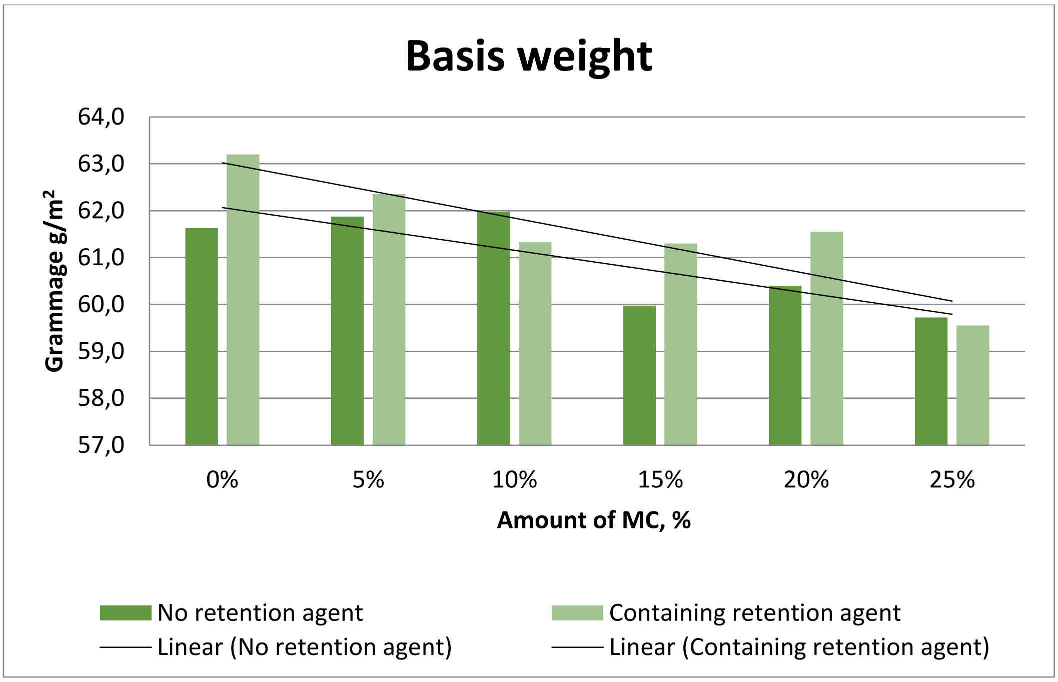

FIGURE 25. Basis weight

TABLE 26. Tensile strength

	No retention agent					
	0 %	**5 %**	**10 %**	**15 %**	**20 %**	**25 %**
1	2,88	2,5	2,22	2,34	2,01	1,81
2	2,81	2,48	2,27	2,13	2,07	1,73
3	2,73	2,89	2,29	2,2	1,94	1,86
4	2,81	2,45	2,41	2,19	1,77	1,77
5	2,85	2,58	2,48	2,19	1,94	1,86
6	2,57	2,36	2,25	2,25	1,24	1,74
7	2,83	2,48	2,28	2,14	1,94	1,77
8	2,54	2,53	2,39	2,18	1,75	1,95
AVG	2,75	2,53	2,32	2,20	1,83	1,81
SD	0,13	0,16	0,09	0,07	0,26	0,07

	Containing retention agent					
	0 %	**5 %**	**10 %**	**15 %**	**20 %**	**25 %**
1	3,17	2,8	2,75	2,22	2,19	1,91
2	2,98	2,8	2,69	2,34	2,25	2
3	3,14	2,73	2,76	2,64	2,22	1,75
4	3,11	2,75	2,55	2,4	2,15	1,91
5	3	2,82	2,49	2,24	2,12	1,97
6	2,66	2,82	2,47	2,39	2,19	2,09
7	2,84	2,92	2,82		1,91	1,9
8	3,08	2,06	2,5		1,98	1,87
AVG	3,00	2,71	2,63	2,37	2,13	1,93
SD	0,17	0,27	0,14	0,15	0,12	0,10

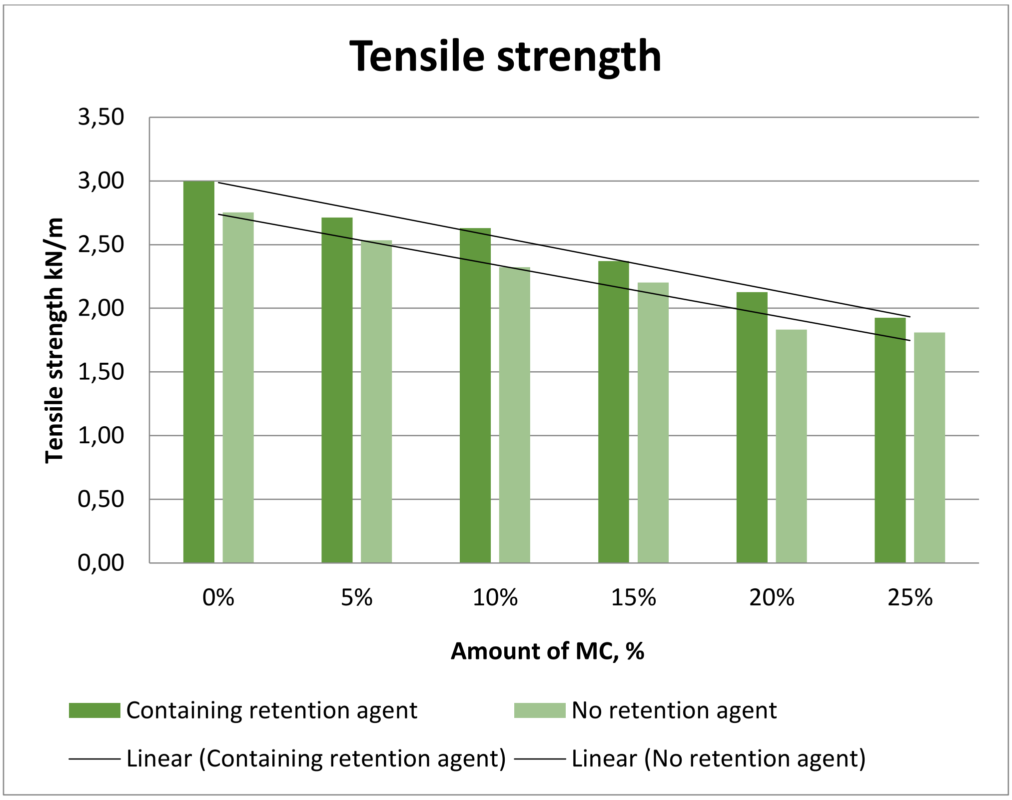

FIGURE 26. Tensile strength

6. Sheets with 80% Pine and 20 % birch pulp mixture, 29 April.

TABLE 27. Basis weight

Without retention agent						
	0 %	5 %	10 %	15 %	20 %	25%
1	66	64,3	50	64,7	64	66
2	67,9	65,2	46	64,9	65	64
3	66,6	63,6	46,7	67,8	63	67
AVG	66,8	64,4	47,6	65,8	64,0	65,7

Containing retention agent						
	0 %	5 %	10 %	15 %	20 %	25 %
1	66,3	66	46,6	64,5	61,5	66
2	65,7	62,5	45,3	65	65	66,9
3		60,8	47,3	66,8	63,7	67
AVG	66	63,1	46,4	65,43	63,4	66,6

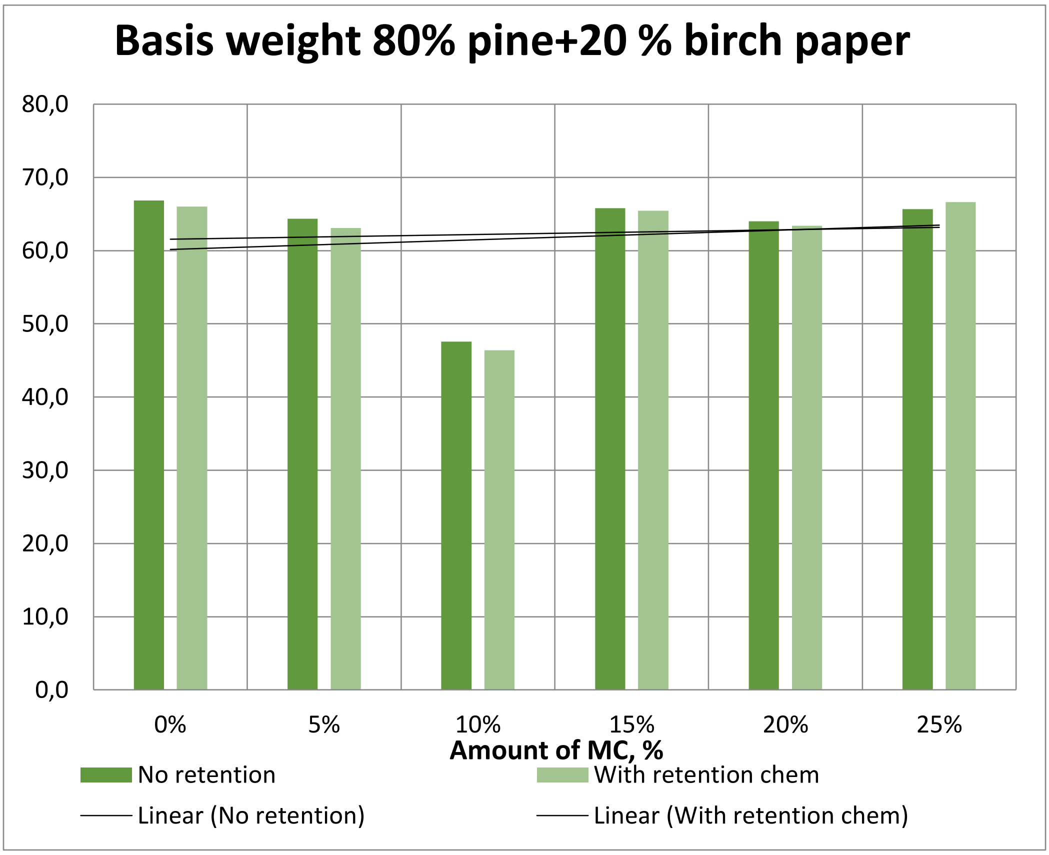

FIGURE 27. Basis weight

TABLE 28. Tensile strength

	No retention					
	0 %	**5 %**	**10 %**	**15 %**	**20 %**	**25 %**
1	3,52	3,11	1,9	2,89	3,39	3,15
2	3,45	2,96	2,15	2,93	3,21	2,93
3	3,36	3,43	2	2,6	3,34	3,12
4	3,52	3,44		3,34	3,13	2,96
5	3,7	3,46		3,22	3,08	2,92
6	3,36	3,3		3,08	3,53	2,97
7		3,34		3,12		
8		3,15				
AVG	3,49	3,27	2,02	3,03	3,28	3,01

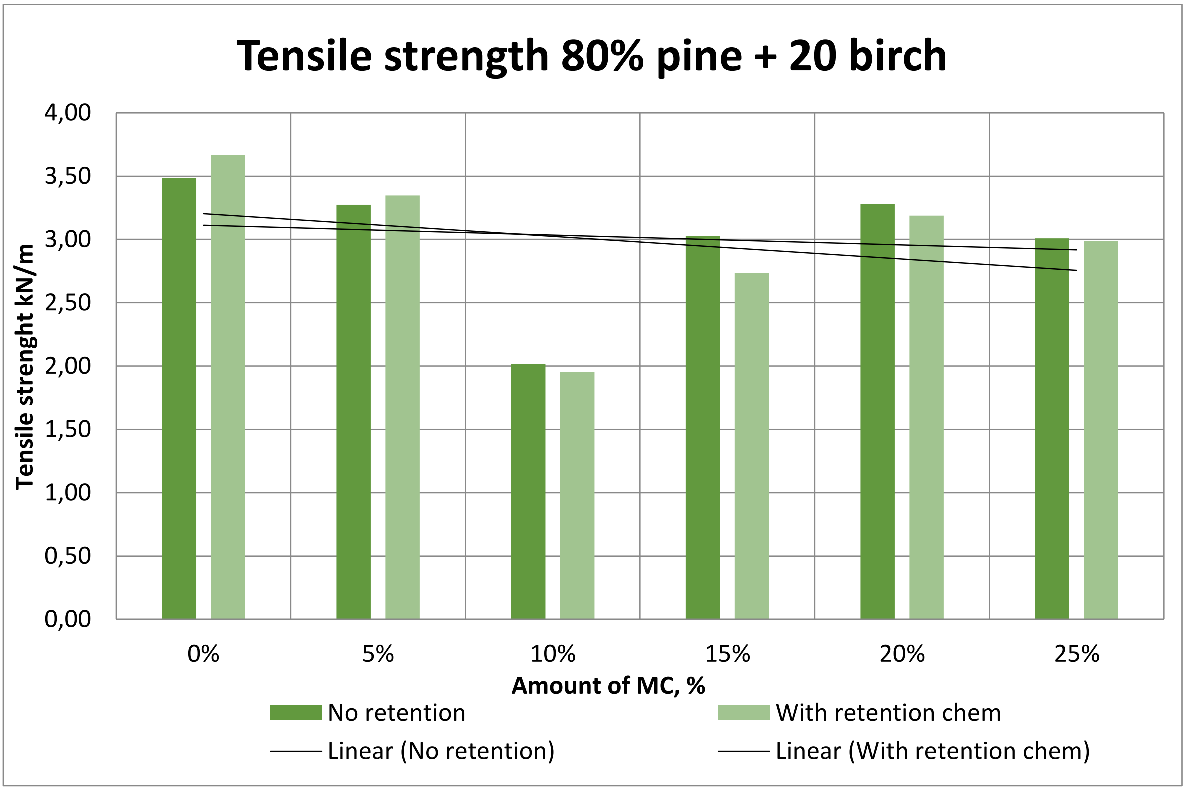

FIGURE 28. Tensile strength

TABLE 29. Optical Properties

No retention						
	0 %	**5 %**	**10 %**	**15 %**	**20 %**	**25 %**
Opacity	71,49	70,82	62,23	71,48	69,82	71,14
Transparency	33,82	34,41	42,32	33,6	35,14	33,94
WI CIE D65/10+UV (Whiteness)	71,11	71,6	73	71,92	72,06	72,17

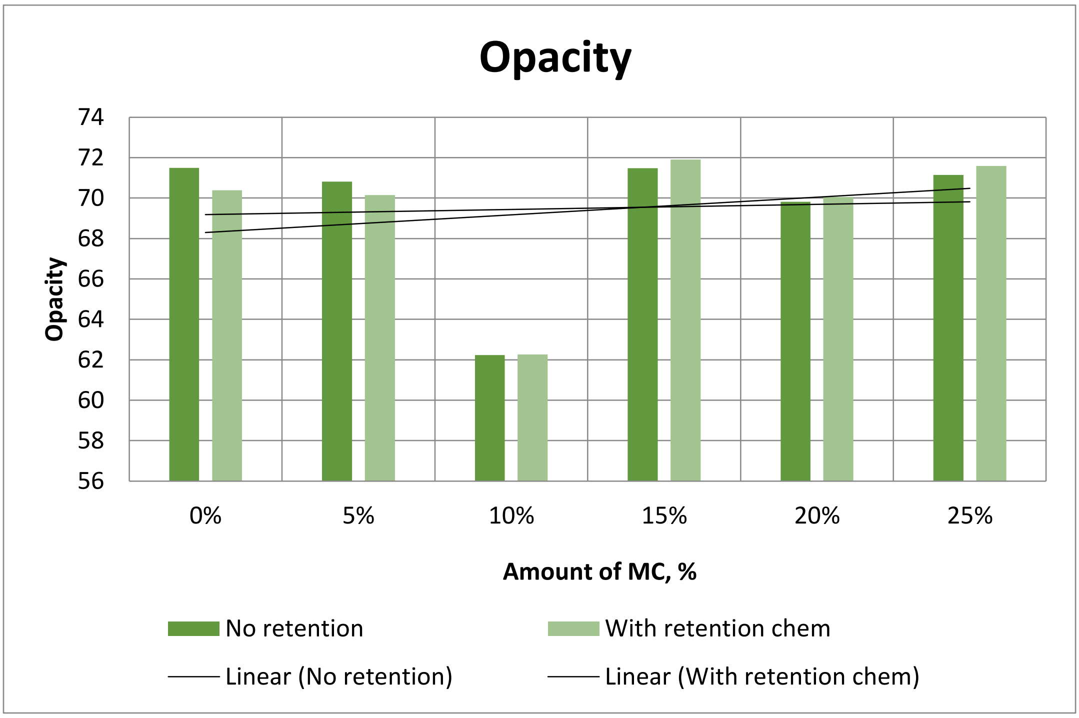

FIGURE 29. Opacity

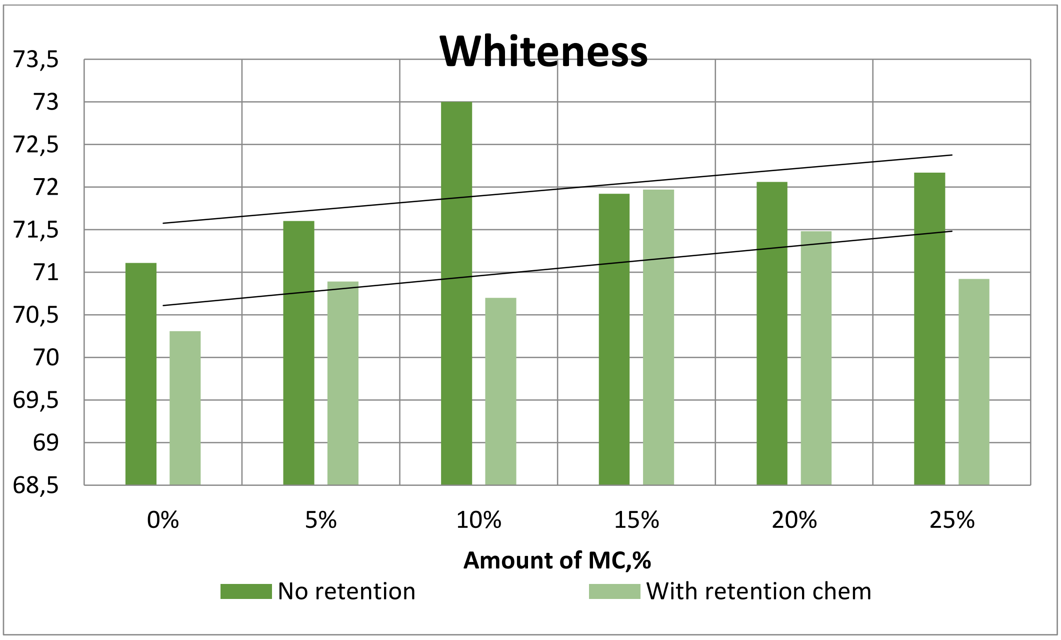

FIGURE 30. Whiteness

TABLE 30. Roughness

Without retention aid						
	0 %	5 %	10 %	15 %	20 %	25
1	2629	2737	2940	3764	2396	3416
2	2484	2333	3551	3316	2898	4008
3	2087	2468	3158	4068	3520	3610
4	2250					3489
5	1797					3852
6	1739					
AVG	2164,3	2512,7	3216,3	3716	2938	3675

Containing retention aid						
	0 %	5 %	10 %	15 %	20 %	25 %
1	1637	2425	3789	4002	3393	3211
2	1860	2821	3517	3875	2873	3572
3	2031	2892	3068	3584	2704	3259
4	1934	3022	3146	4310	3558	3301
5		3246	3188	3071	2715	4069
6		2820	2954	3024	2555	3166
AVG	1866	2871	3277	3644,3	2966,3	3429,7

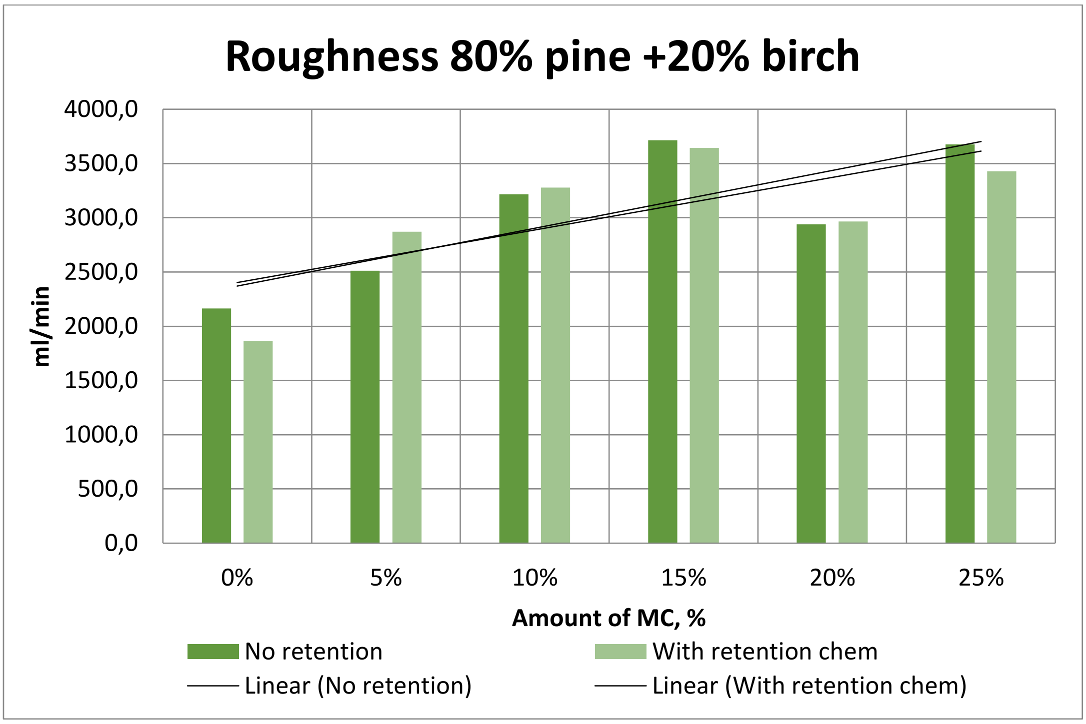

FIGURE 31. Roughness

TABLE 31. Thickness

Without retention agent						
	0%	5%	10%	15%	20%	25%
1	128,6	147,2	142,2	147,8	152,6	165,2
2	135,3	142,8	144,2	152,5	150,4	156,6
3	122,6	146	146,6	148,8	159,4	159,4
4	123,9	140	150	152,3	161,2	160
5	124,1	135,7	143,3	152,6	152,8	159,6
6	122,3	138,8	145,5	149,1		166,6
AVG	126,1333	141,75	145,3	150,5167	155,28	161,2333

Containing retention agent						
	0%	5%	10%	15%	20%	25%
1	126,5	143,9	141,3	153,1	158,7	148,9
2	132,4	141,4	143,2	152,2	157,2	149,8
3	132,5	133,5	142,6	152	145,1	155,9
4	128,6	137,5	145,5	148,5	145,4	146,8
5	122		13,7	147,5	164,5	
6	125,6				154,8	
AVG	127,9333	139,075	117,26	150,66	154,2833	150,35

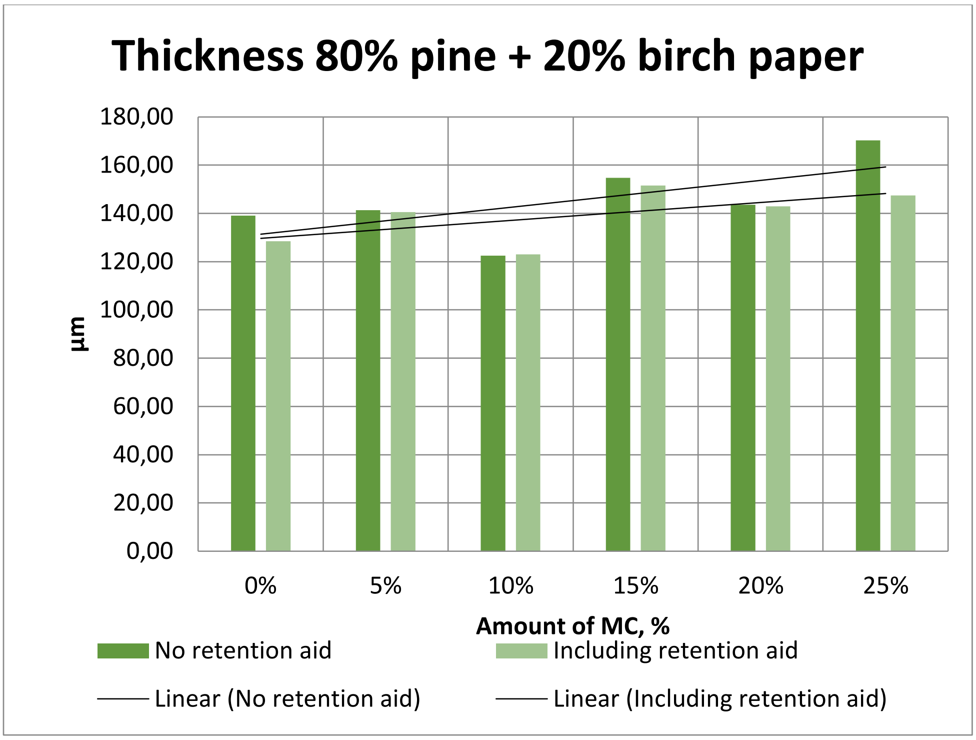

FIGURE 32. Thickness

7. 80% birch and 20 % pine combination, retention chemical, 29 April.

TABLE 32. Basis weight

Without retention aid						
	0%	**5%**	**10%**	**15%**	**20%**	**25%**
1	70	66,4	65,2	63	68,2	73,8
2	69,7	65	64,6	64	65,9	73,7
3	69	67,3	65,8	64	66,9	74,2
AVG	69,6	66,2	65,2	63,7	67,0	73,9

Containing retention agent						
	0%	**5%**	**10%**	**15%**	**20%**	**25%**
1	63,7	66,7	63,5	61,09	72,3	73
2	69,7	61,9	62,6	61,5	73,8	
3	69,3		63,2	60,4	63,4	
AVG	67,6	64,3	63,1	61,0	69,8	73,0

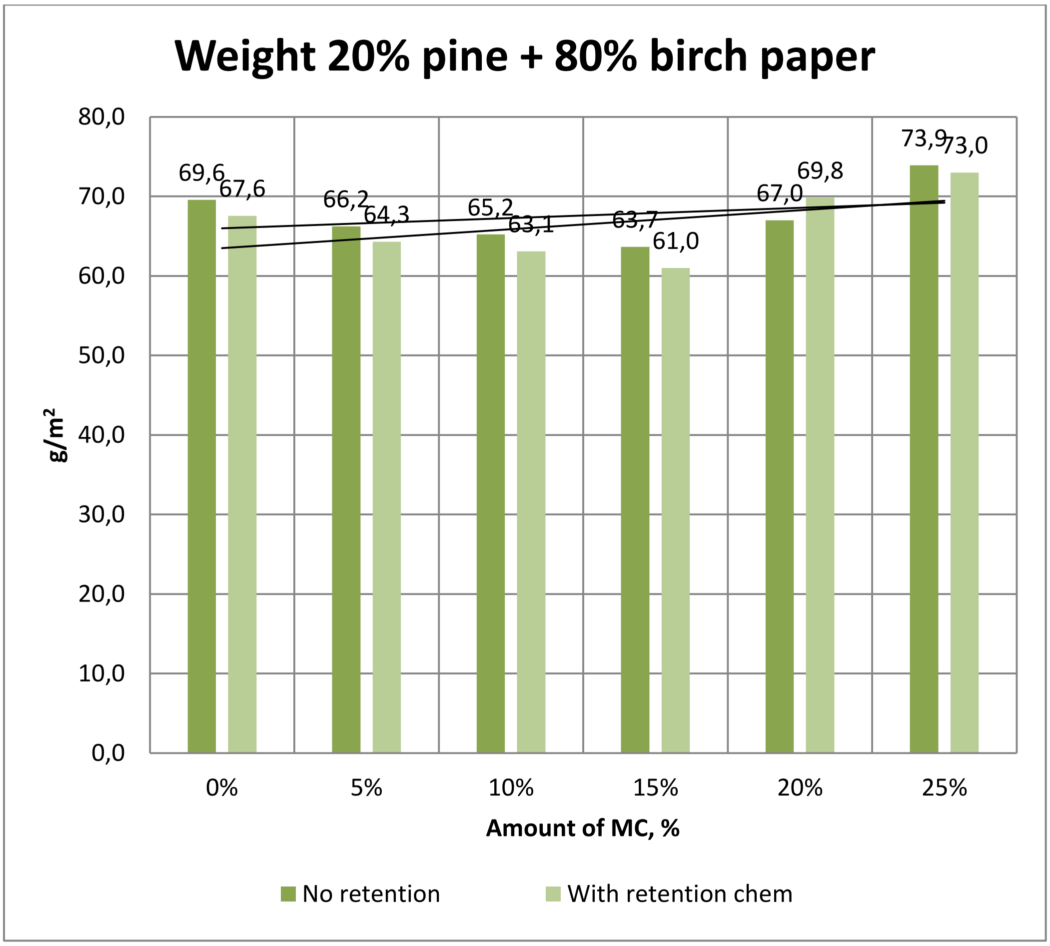

FIGURE 33. Basis weight

TABLE 33. Tensile strength

Without retention agent						
	0%	5%	10%	15%	20%	25%
1	2,48	2,4	2,34	2,07	2,03	2,71
2	2,53	2,54	2,36	2,09	2,63	2,84
3	3,2	2,44	2,3	2,13	2,67	2,73
4	2,16	2,75		2,05	2,08	2,91
5	3,97	2,69		2,04	2,33	2,96
6	3,32	2,67		2,13	2,28	2,76
7		2,78			2,02	
8						
AVG	2,94	2,61	2,33	2,09	2,29	2,82

Containing retention agent						
	0%	5%	10%	15%	20%	25%
1	3,08	2,58	1,72	2,02	2,06	2,68
2	3,07	1,49	2,25	2,3	2,54	2,86
3	2,84	2,52	2,49	2,04	2,38	3,24
4	2,87	2,41	2,45	2,06	2,58	2,83
5	2,96	2,53	1,4	2,29	2,61	2,78
6		2,51	2,79	2,13	2,33	
7		2,65		2,3		
8						
AVG	2,96	2,38	2,18	2,16	2,42	2,88

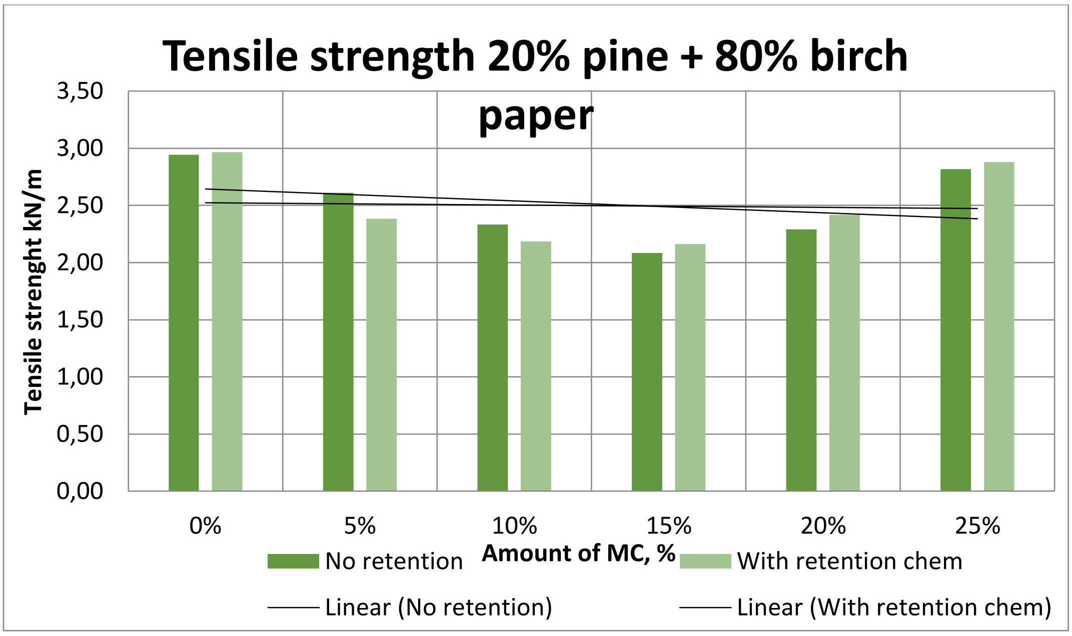

FIGURE 34. Tensile strength

TABLE 34. Roughness

Without retention agent						
	0%	**5%**	**10%**	**15%**	**20%**	**25%**
1	2578	2150	2277	2816	3492	3325
2	2978	2122	2430	3385	3214	3297
3	2877	1860	2934	2616	4132	2965
4	4380	1858	2514	3395	3289	2948
5			2343	3781	4102	2948
6			2279	4329	2977	2802
AVG	3203	1998	2463	3387	3534	3048

Containing retention agent						
	0%	**5%**	**10%**	**15%**	**20%**	**25%**
1	1259	2132	2275	2741	3097	3211
2	1310	2504	2200	2174	2673	3720
3	1487	2331	2374	2606	3543	
4	1524	2176	2294	3283	2797	
5	1140				2591	
6					2439	
AVG	1344	2286	2286	2701	2857	3466

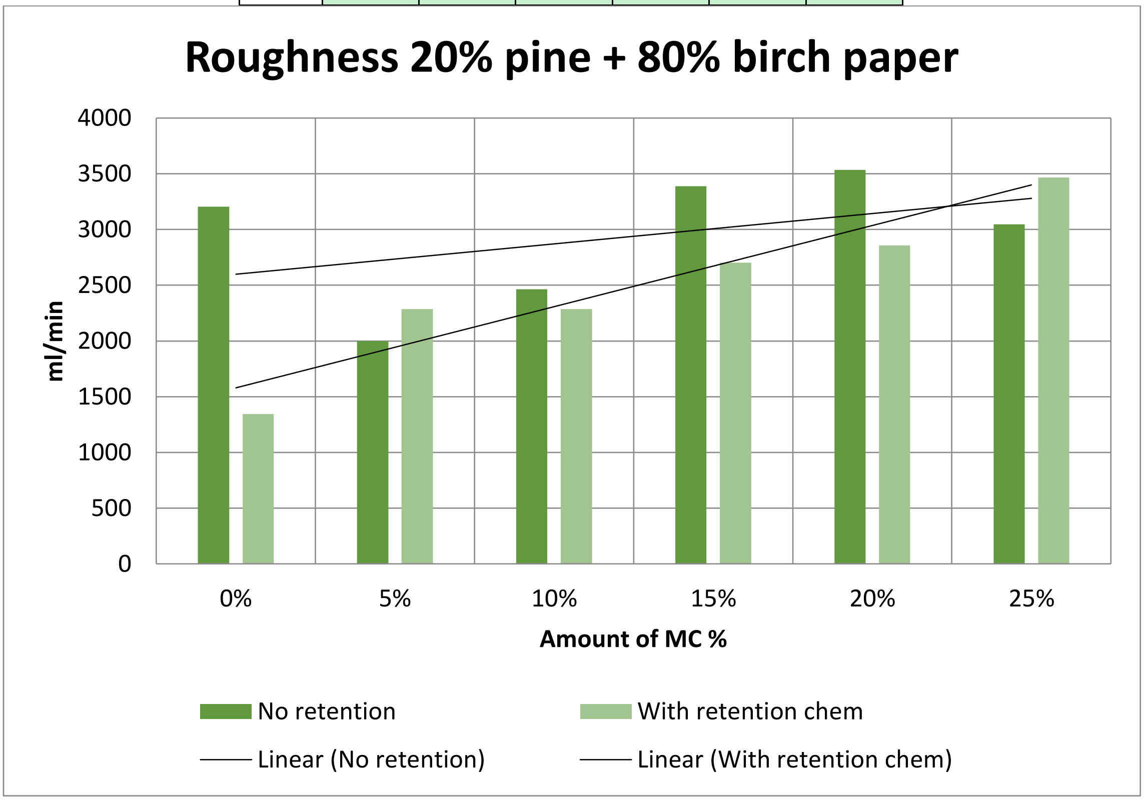

FIGURE 34. Roughness

TABLE 35. Thickness

Without retention aid						
	0%	**5%**	**10%**	**15%**	**20%**	**25%**
1	128,6	147,2	142,2	147,8	152,6	165,2
2	135,3	142,8	144,2	152,5	150,4	156,6
3	122,6	146	146,6	148,8	159,4	159,4
4	123,9	140	150	152,3	161,2	160
5	124,1	135,7	143,3	152,6	152,8	159,6
6	122,3	138,8	145,5	149,1		166,6
AVG	126,1	141,8	145,3	150,5	155,3	161,2

Containing retention aid						
	0%	**5%**	**10%**	**15%**	**20%**	**25%**
1	126,5	143,9	141,3	153,1	158,7	148,9
2	132,4	141,4	143,2	152,2	157,2	149,8
3	132,5	133,5	142,6	152	145,1	155,9
4	128,6	137,5	145,5	148,5	145,4	146,8
5	122		13,7	147,5	164,5	
6	125,6				154,8	
AVG	127,9	139,1	117,3	150,7	154,3	150,4

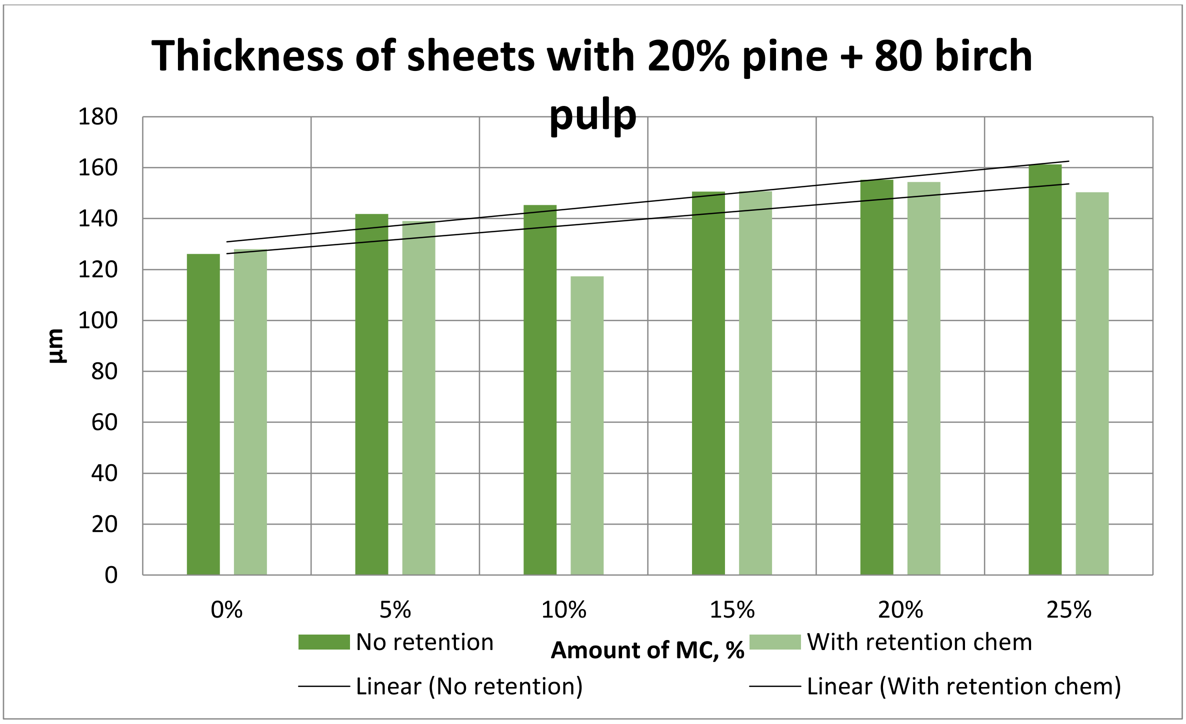

FIGURE 35. Thickness