

# Transfer Printing and Cellulose Based substrates for modern Textile Printing

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Abstract

Digital printing technology is a technique that has been growing since the 1990s and has a high growth potential when it comes to using different ink types and transfer printing techniques. In comparison to screen printing, digital transfer printing techniques have shown to consume less ink and water and are therefore considered to be a more environmentally friendly alternative when textile printing. Therefore, a digital printing technique called sublimation transfer printing was studied in this thesis. In a sublimation transfer printing process, an image is printed on a paper and then the image is transferred to a textile by using heat and press. Suitable coating of the paper surface has shown to increase the printing properties on the paper and therefore the paper samples used in the thesis were coated with three different coating formulas. The coating formulas used in this thesis were polyvinyl alcohol (PVOH) of a type A, PVOH A with ground calcium carbonate (GCC) and PVOH type B with GCC. PVOH A has a higher degree of hydrolysis than PVOH B. Results showed that there was no significant difference between optical densities between paper samples of different coat weights and coating formulas. The colour bleeding and colour penetration decreased in the printed paper samples for PVOH A + GCC and PVOH B + GCC when the coat weight increased, and the porosity of the coating decreased to some extent. As a conclusion, paper samples coated with PVOH A + GCC with coat weights above 15 g/m<sup>2</sup> showed to give the best properties since the colour bleeding was minimal in those printed coated paper samples.

**Nyckelord**

Keyword

## Table of Content

<b>Abbreviations .....</b>	<b>6</b>
<b>Abstract.....</b>	<b>7</b>
<b>1. Introduction.....</b>	<b>8</b>
1.1. Background of the project.....	8
1.2. Aim of the project .....	9
1.3. Objective of the thesis .....	9
<b>2. Literature review .....</b>	<b>11</b>
2.1. Transfer Printing – an introduction .....	11
2.2. Screen printing .....	12
2.3. Digital transfer printing.....	13
2.4. STP on textiles .....	15
<b>3. Process.....</b>	<b>24</b>
<b>4. Materials and methods .....</b>	<b>25</b>
4.1. Cellulose substrate.....	25
4.2. Preparation of coating formula.....	25
4.3. Coating of cellulose substrate .....	27
4.4. Calculations of coat weight .....	31
4.5. Inkjet printing with water-based ink .....	31
4.6. Measurements of optical density .....	33
4.7. Colour bleeding .....	34
4.8. Sublimation transfer printing .....	35
4.9. Scanning electron microscope measurements .....	35
4.10. Determination of coat thickness .....	36
4.11. Determination of air permeance using Bendsten method.....	36
4.12. Determination of water absorptiveness – Cobb <sub>60</sub> method .....	37
4.13. Statistical analysis.....	38

<b>5. Results .....</b>	<b>39</b>
<b>5.1. Viscosity measurements .....</b>	<b>39</b>
<b>5.2. Coat weights .....</b>	<b>39</b>
<b>5.3. Inkjet printing with water-based ink .....</b>	<b>41</b>
<b>5.4. Optical density measurements.....</b>	<b>43</b>
<b>5.5. Scanning electron microscopy measurements.....</b>	<b>45</b>
<b>5.6. Thickness of coating.....</b>	<b>48</b>
<b>5.7. Air permeance .....</b>	<b>48</b>
<b>5.8. Water absorptiveness as Cobb<sub>60</sub>.....</b>	<b>49</b>
<b>6. Discussion.....</b>	<b>50</b>
<b>6.1. Analysis of the experimental work.....</b>	<b>50</b>
<b>6.2. Analysis of the process.....</b>	<b>54</b>
<b>6.3. Future work .....</b>	<b>55</b>
<b>7. Conclusion .....</b>	<b>56</b>
<b>8. References.....</b>	<b>58</b>
<b>Appendix.....</b>	<b>59</b>

# Abbreviations

Abbreviation	Word
BSE	Back-scattered electron imaging
CaCO <sub>3</sub>	Calcium carbonate
CD	Cross direction
DTP	Digital transfer printing
GCC	Ground calcium carbonate
ISO	International organization of Standardization
MD	Machine direction
MG-paper	Machine glazed paper
PVOH	Polyvinyl alcohol
PVOH A/B	Polyvinyl alcohol A or B
Rpm	Revolutions per minute
SEM	Scanning electron microscope
SiO <sub>2</sub>	Silicon dioxide
STP	Sublimation transfer printing
UV-light	Ultraviolet light
Y, M, C, K	Yellow, magenta, cyan, key-colour (black)

# Abstract

Digital printing technology is a technique that has been growing since the 1990s and has a high growth potential when it comes to using different ink types and transfer printing techniques. In comparison to screen printing, digital transfer printing techniques have shown to consume less ink and water and are therefore considered to be a more environmentally friendly alternative for textile printing. Therefore, a digital printing technique called sublimation transfer printing was studied in this thesis. In a sublimation transfer printing process, an image is printed on a paper and then the image is transferred to a textile by using heat and pressure. Suitable coating of the paper surface has shown to improve the printing properties on the paper and therefore the paper samples used in the thesis were coated with three different coating formulas. The coating formulas used in this thesis were polyvinyl alcohol (PVOH) of a type A, PVOH A with ground calcium carbonate (GCC) and PVOH type B with GCC. PVOH A has a higher degree of hydrolysis than PVOH B. Results showed that there was no significant difference between optical densities between textiles and paper samples of different coat weights and coating formulas. The colour bleeding and colour penetration decreased in the printed paper samples for PVOH A + GCC and PVOH B + GCC when the coat weight increased, and the porosity of the coating decreased to some extent. As a conclusion, paper samples coated with PVOH A + GCC with coat weights above 15 g/m<sup>2</sup> showed to give the best properties since the colour bleeding was minimal in those printed coated paper samples.

# 1. Introduction

## 1.1. Background of the project

Digital printing technology is a printing method that can be used to transfer a pattern from a cellulose based printed substrate to a specific textile. The technology has been growing since the 1990s (Dawber (2008)) and has a high growth potential when it comes to using different ink types and transfer printing techniques (Kight (2011)). Digital transfer printing (DTP) technology offers a high resolution image as the images are transferred to the textile with high accuracy (Dawber (2008)).

Printing techniques used in the textile market may follow the basic principles of the inkjet printing technique. The set up of the inkjet printing technique includes a print head where the ink passes directly to the substrate. The method is a contactless type of printing and has been remarkable for its creative approaches and business opportunities (Kight (2011)). The printing method can be used to transfer an image to a paper substrate. This paper with an image will work as a stamp and transfer the pattern onto a textile. Processes such as heat transfer printing and sublimation transfer printing (STP) are generally clustered under the transfer printing area. In STP, the ink will sublimate under the transfer printing step and transfer onto the fabric creating the desired pattern on the textile (Clarke (2011)). This STP technique allows printing on textile instantly and can be applied to all types of print designs, i.e. the printer does not have to be adapted to a new type of print design which makes the technique more cost-effective. Additionally, the transfer printing methods, such as STP, are ecofriendly due to the low consumption of water and ink (Elsayad and El-sherbiny (2008)).

Digital printing industry is an important player on the paper consumer market, thus different paper qualities are used in different printing processes. Today, the transfer printing technique, which goes under the area of digital printing, is more and more used in textile industry (Tyler (2010)). Also, in the textile industry, one widely used type of textile is cotton. The textile industry has used cotton for many years with tremendous negative effects on the environment. One illustrative example is the environmental changings of the Aral Sea which has drained due to massive

consumption of water for cotton production (Edelstien (2012)). Hence, nowadays the textile printing market has received great attention because of negative environmental effects and environmentally friendly printing and textile and printing alternatives are requested, one of them being STP (Tyler (2010)). Subsequently, due to a request of high quality transfer paper, the textile printing area is important for all paper producers such as BillerudKorsnäs. Even more, BillerudKorsnäs is an international company working towards environmental benefits and focusing on creating sustainable cellulose based products with as low impact on the environment as possible. To manufacture paper that can be used by other industries in order to reduce their negative impact on the environment is the responsibility of BillerudKorsnäs as well.

## **1.2. Aim of the project**

The aim of the project is to identify how a cellulose based substrate surface can be developed by applying different coating formulas for producing paper suitable for transfer printing processes where paper will interact with dyes/ink and textiles. One particular technique, STP, will be used for exemplifying the effect of coatings on the final properties of the printed textile when water-based sublimation inks are used. The type of textiles used in this work will consist of polyester.

The goal is to find the most significant properties of a cellulose substrate when using a specific coating formula and to achieve a successful transfer printing on textiles with high resolution patterns.

## **1.3. Objective of the thesis**

The objective of the project is to identify how a cellulose based substrate (paper) surface can be improved by applying different coating formulas in order to develop surface properties suitable for dyes/ink printing of high printing quality. The objective of the thesis can be further divided into sub objectives as the following:

1. Choose one or more cellulose based substrates.
2. Decide the different coating formulas that will be tested on the cellulose-based substrates.
3. Decide the methods for coating on the substrates.

4. Experimentally determine the coating properties such as dry content, viscosity, suitable coating-weight applied, etc., for a proper application on the substrates.
5. Coating of the substrates and characterization of the coated material.
6. Inkjet sublimation printing (water-based ink) on the substrates and evaluation of the printing result.
7. STP on textile and evaluation of the substrate after STP on the textile.
8. Printing evaluation of the printed textiles.
9. Summarizing and reporting.

## 2. Literature review

In this chapter, a review over the scope of transfer printing area is presented. The chapter includes a history of transfer printing, a description of screen printing and digital printing and will be concluded with information about sublimation transfer printing which is used in this thesis.

### 2.1. Transfer Printing – an introduction

An important and adjustable method for introducing a specific design or pattern to a substrate is called transfer printing. It has been discovered that transfer printing was used as early as in the fourth century, where a block-printed tunic with a Buddhist text was found in China. Further, the earliest known block-printed book dated 868 can be found in the British museum (Sarkodie *et al.* (2018)). Beside its widespread use in the textile industry, transfer printing can also be used in decorating metals and woods (Yin *et al.* (2013)).

Transfer printing implies that a pattern is moved from a transfer substrate, in most cases paper, to a textile such as synthetic or natural fabrics (Yin *et al.* (2013)). The latter one may require pretreatment of the fabric before the transfer process for better adhesion of ink molecules to the fabric (Bemska and Szkudlarek (2013), Chi-Ching Lin *et al.* (2016)). The process where a transfer paper is printed followed by transfer printing on textile is done in several steps. Generally, according to Yin Y *et al.*, a pattern that consists of non-polar disperse dye is printed to a cellulose substrate, in most cases paper (Yin *et al.* (2013)). The transfer paper is then placed on the textile, allowing contact between the dye and the textile. The transfer paper and the textile is placed beneath a heat press and the dye is transferred to a fabric, e.g. by sublimation and interacts to the fabric as a result of hydrophobic forces and van der Waals interactions (Yin *et al.* (2013), Bemska and Szkudlarek (2013), Ujiie (2006)).

#### 2.1.1.1. Environmental and social aspects

Transfer printing is able to have a great impact on the textile industry by offering a faster production at a lower cost for small scale batches in comparison to traditional methods. Additionally, the printing patterns are more enhanced in resolution. The development research of

the transfer printing technique has also led to solutions that aim for a more environmentally friendly method in the textile printing industry which is necessary to compensate for the negative environmental impacts (Yin *et al.* (2013)). One considerable example where the textile industry has had an enormous and destructive impact is the drainage of the Aral Sea located in Central Asia. The drainage was a result from the growing of cotton that was used for the making of textiles in the Soviet Union. The production of cotton was of great economical value to communities and families in Karakalpakstan and although the intention of the production was good, the consequence was a tremendous disaster. The outcome of the drainage of the Aral Sea did not only result in a dried-out sea, it also affected the surrounding society, their social impacts and their way of living (Edelstein, Cerny and Gadaev (2012)). As cotton has shown to have a great negative impact on both the society and environment, this thesis will focus on polyester as a textile.

## 2.2. Screen printing

The textile printing area includes several types of techniques, two of them being the screen printing technique and the digital printing technique. Screen printing is a conventional method where the simplicity of the textile printing technique has been developed during the years. However, there is a high demand of further development of the technology in textile digital printing techniques to reach a technique that has as low environmental impact as possible (Yin *et al.* (2013), Tyler (2010)).

The screen printing and digital printing provide similar products, but the printing process differs between the two techniques. Screen printing is often used in mass production (Tyler (2010)), while digital printing is used for smaller scales of batches. The screen printing process (see Figure 1) of printing on fabrics begins with a high quality work of art where the art is separated in composite colours. Generally, a film positive is created and placed upon a screen with an emulsion where UV-light will expose the screen. After exposure, the screen is washed to eliminate emulsion that has not been exposed by the UV-light. Subsequently, the screen is dried in a cabinet and ready to be used. The screen is placed upon a fabric, where the ink is pressed through holes in the screen to print the fabric. Lastly, the printed fabric is then cured at a high temperature in an oven (Sarkodie *et al.* (2018)). Figure 1 illustrates the process of screen printing on fabric.

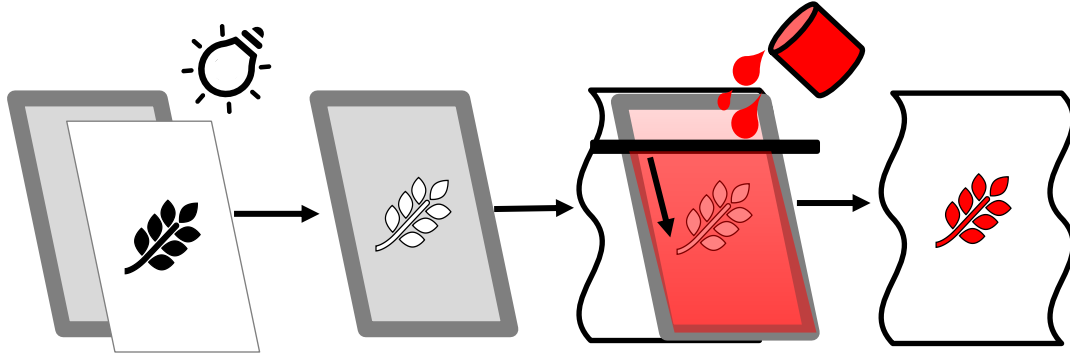


Figure 1. The figure illustrates an overall process of screen printing. Firstly, a film positive (white rectangle with print) is placed upon a screen (grey rectangle) covered with an emulsion and UV-light is exposed to the screen and film positive. After exposure, the screen is being washed and holes are obtained. Secondly, the screen is placed upon a fabric (wavy figure) and ink is pressed through the holes. Finally, a pattern is obtained on the fabric. The process is repeated if more colours will be introduced to the fabric

### 2.3. Digital transfer printing

One example of a digital printing technique is DTP. The usage of DTP techniques has had major breakthroughs since the millennium shift, and both software and hardware systems have been developed since. Figure 2 illustrates an example of a DTP process. Several kinds of commercial machines for transfer printing technique are heading towards the goal of large usage in mass-production. Due to the ability of digitally specifying the colour, the digital printing can provide results with the true colour of the design on textiles (Sarkodie, B. *et al.* (2018), Tyler (2010)).

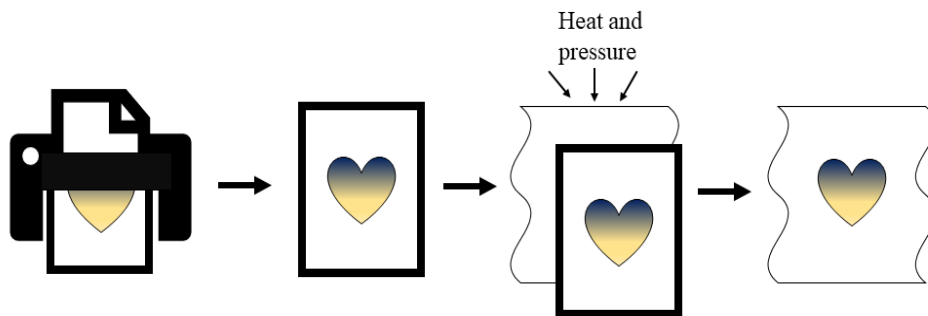


Figure 2. The figure shows an overall process of transfer printing. A transfer paper is being printed by a regular printer. The transfer paper is placed upon a fabric where heat and pressure are applied. A printed fabric is received.

It may give the impression that screen printing and digital printing compete, but one needs to understand that the techniques are used with different intentions. As stated earlier, the screen printing method is used for large scale production, while the digital printing method is used for smaller batch production. Therefore, it is most reasonable to choose the method that is suitable for the objectives of the production. The digital printing technique can therefore have a wide usage and importance in production of customized brands and development of creating innovative designs. The digital printing technique comes with many advantages if one would use the screen printing method for a smaller scale production purpose. The digital printing method will give a reduced cost due to easily available equipment, such as a desktop printer, while each colour in a pattern used for screen printing requires a separate screen. If larger scale batches would be produced by digital printing, a larger number of printers would be needed which means a higher production cost. Also, digital printing entails reduced waste of disperse inks which contains a high amount of water. In screen printing, a large volume of ink is poured over the screen that will cover an area larger than the desired pattern. In digital printing, the amount of ink used is the volume used to print the pattern (Tyler (2010)). As mentioned, digital printing techniques are heading towards the goal of usage in mass-production. Therefore, it is very interesting to investigate the digital printing technique.

One example of a digital printer is typically the desktop printer that you can find almost anywhere nowadays. The technique can be classified into several types of methods, where two of them are the continuous inkjet and dot-on-demand (Sarkodie. *et al.* (2018)). Information about the print head techniques will be further described below.

### 2.3.1. Continuous inkjet

The main principle behind the continuous inkjet, is that the ink will turn into a stream of small droplets when the ink is continuously emerged from a nozzle. The droplets move and are steered to the substrate because of an electric field that is applied upon the droplets. Since the continuous inkjet has a fast printing time, the technique has the potential to reach a deposition rate similar to the screen printing technique (Tyler (2010)).

### 2.3.2. Drop-on-Demand

The Drop-on-Demand technique can be divided into several types of technologies, but the main concept results in a delivery of ink when it is demanded. The demand is regulated by the software and will give information when it is time to drop ink. If there is a demand, the ink will drop onto the substrate. The drops of ink that will appear on the substrate will have a size about 20-30 picolitres but Drop-on-Demand technologies are being developed to decrease the drop-size. The decrease in drop-size will provide better resolution and decrease the rate of ink flow to the substrate (Tyler (2010)).

The majority of the print heads used for Drop-on-Demand are so called piezoelectric print heads (Tyler (2010)). The technology requires a material of a piezo character, that will deform its shape when applying an electrical field. The chamber where the ink is stored is constructed of this material and when applying an electric field, the chamber volume will be reduced. This result in that dye is forced and pressed through the nozzle. When the force is released, the chamber will return to its original shape (Sarkodie, B. *et al.* (2018), Tyler (2010)). This technique has major advantages, such as controlling the size of the drops and increased production rates (Tyler (2010)).

Other types of Drop-on-Demand techniques consists of thermal print heads. In the thermal heads, a volume of ink is heated to the point where a bubble is formed and then explode. The expansion of the bubble creates a wave of pressure in the ink fluid, which will result in a drop of ink that will be emerged from the nozzle and deposited on the fabric. The burst of the bubble also has its disadvantages. The burst may result in reduced quality of the print because of unwished splattering. Also, the ink may get damaged because of the heat which can cause a block in the nozzle (Tyler (2010)).

## 2.4. STP on textiles

In this thesis, the focus will be on sublimation transfer, which is a type of digital printing technique where the ink will sublime which means that the ink will go directly from a solid state to a gaseous state (Yin *et al.* (2013)). Also, only non-polar water-based ink will be used.

In the 90's, the STP technique was introduced to the inkjet printers and became a popular technique due to its easily adaptation to regular standard desktop printers. Therefore, the technique can be used without investing in new and expensive devices (Ujiie (2006)). The STP technique can be used to transfer designs to glasses, mugs, coated metals, but most importantly textiles (Ujiie (2006)). The design is firstly printed onto a carrier substrate, which in most cases is paper (see Figure 2). The ink, which consists of dye dispersed in water and has a sublimation property, will adhere to the surface of carrier substrate. The paper will then be placed upon a fabric, where synthetic based textile is to prefer due to its good interactions with the hydrophobic disperse dyes. The paper will then be placed upon a fabric, where plastic based textiles are to prefer due to its good interaction with disperse dyes. The paper and the textile will be placed on a heat-press, allowing the dispersed dye to sublime and transfer into the textile where the dye will permanently stay (Elsayad and El-sherbiny (2008), Yin *et al.* (2013)).

Factors that affect the properties of the transfer printing and the transfer result on the fabric are the quality of the transfer paper (Yin *et al.* (2013)), temperature, transfer time (Elsayad and El-sherbiny (2008)), and the type of textile used (Yin *et al.* (2013)). Experiments have shown that high temperature with a high transfer time will result in that a higher volume of dispersed dye will transfer from paper to textile. The result also shows that the best quality result is achieved when the paper is properly coated (Elsayad and El-sherbiny (2008), Bemska and Szkudlarek (2013), Ujiie (2006)). Therefore, it is of high interest to investigate these factors to reach the goal to produce a high-quality transfer result.

#### 2.4.1. Cellulose substrates in STP

Some important aspects to consider when choosing the cellulose substrate for STP on textile are mechanical properties of paper (Elsayad and El-sherbiny (2008)) and the structure and the surface smoothness of the paper (Ujiie (2006)). These parameters can be affected by the addition of coating on the paper surface (Elsayad and El-sherbiny (2008)). To understand how to influence the paper structure and mechanical properties, one must know the baseline structure of a cellulose substrate.

#### 2.4.1.1. Short overview of the structure of paper

Paper can be described as a layered structure that consists of a network of cellulose fibers bonded to each other by Van der Waals interactions and hydrogen bonds. The paper also consists of lignin, hemicellulose and other chemicals used to improve the mechanical and structural properties of the paper (Burhenne et al. (2013), Pappersteknik (1996)). In order to improve the stability of the cellulose network, the fibers are mechanically pretreated in a mill until the fibers get soft and flexible. The fibers interact with each other with and/or without any adding of binding agents. Cellulose fibers of wood plants are approximately  $\sim 15 \mu\text{m}$  in diameter and 0.9 mm in length (Ververis et al. (2004)). One paper sheet contains approximately one million fibers per gram (Pappersteknik (1996)).

#### 2.4.1.2. Machine glazed paper

A machine glazed paper, often abbreviated MG-paper, is a paper that is dried on a so called yankee-cylinder under the production process. A yankee-cylinder is a very large cylinder with a smooth surface that is heated up from the inside of the cylinder. The paper web is stretched out over the cylinder's surface, which results in a stronger type of paper (Defonseka (2013)).

The MG-paper is recognized by one rough side, and one glossy side which is the side that has been in contact with the yankee-cylinder (Defonseka (2013)). The MG-paper is one of the possible cellulose substrates that can be used as a transfer paper in textile printing. This kind of paper, MG-paper, will be used as a transfer substrate in this thesis work. To improve the surface structure and mechanical properties of the MG-paper, the paper will be coated with suitable coating formulas.

#### 2.4.1.3. Coating of cellulose substrate

As earlier stated, paper coating has been shown to improve the structural and mechanical properties of a paper surface that is used as a transfer paper in textile printing (Elsayad and El-sherbiny (2008), Bemska and Szkudlarek (2013), Ujiie (2006)). An uncoated paper has an uneven surface with high irregularities, which acquire a higher density of ink. This is a consequence of the irregularities, where ink has to be filled to receive an even surface suitable for giving a high-quality

transfer in textile transfer printing (Elsayad and El-sherbiny (2008)). If the same amount of ink is used when printing on uncoated papers and on coated papers, it might give the impression that the intensity of ink is lower in the uncoated papers due to higher penetration of ink into the paper.

The coating of the transfer paper must be designed to release as much dye as possible in comparison to regular printing paper, where the aim is to permanently retain all the dye or pigments. Subsequently, the porosity of the coating has a crucial influence of the acceptance and releasing of ink and needs to be regulated to achieve a desired transfer paper surface. Also, the drying time for the ink must be low. (Elsayad and El-sherbiny (2008), Ujiie (2006)).

The coating mixture consists of binders, pigments and additives mixed in water. The role of the pigments is to fill the irregularities at the paper surface. Types of pigments that is commonly used is either clay or calcium carbonate ( $\text{CaCO}_3$ ), such as a ground calcium carbonate (GCC). Results shows that a coating with GCC or clay coating decreases the porosity of the paper because of the pigments filling the pores at the surface of the paper. Additionally, plastic pigments have shown to increase the quality of the transfer paper, even better when mixed with the two other pigments mentioned. The results demonstrate an improved outcome of the printed textile, with a high colour density in the textile and low colour density at the transfer paper surface (Elsayad and El-sherbiny (2008)).

Further types of pigments that can be used are silicon oxide ( $\text{SiO}_2$ ), talc and sericite. A study showed that these three pigments and  $\text{CaCO}_3$ , incorporated with a coating of polyvinyl alcohol (PVOH), also increased the quality of the transfer printing on the textile. Silica, which is a pigment that is often used for traditional inkjet paper, is of a hydrophobic character and creates a pore volume that is beneficial for rapidly diffusing the non-polar disperse dye into the paper surface. The talc is also a hydrophobic mineral that can be a substitute for clay, and improve ink absorption, although the drying time might be increased. Sericite is a mineral that increases the smoothness of the paper surface. The mineral can also substitute for clay, which has improved the barrier and opacity properties of the paper (Chi-Ching Lin *et al.* (2016)).

### **Alternative ways to increasing image quality**

Other approaches for increasing the quality of the transferred printing on textiles can be to modify the surface of the fabric. This can either be done by a sol-gel method where the aim is to increase the hydrophobicity of a cellulose fabric, such as cotton, to increase the affinity for disperse dyes (Bemska and Szkudlarek (2013)). It can also be done by modifying the fabric chemically, such as benzylation, acetylation or cyanoethylation (El-Kashouti, El-Halwag and El-Molla (2000)). In order to avoid chemical substance on the textile, the modification can also be done by applying a plastic film on the cotton surface, with the same objective to increase the hydrophobicity (Bemska and Szkudlarek (2013), El-Halwagy, El-Sayad and El-Molla (2001)). In this thesis project, the focus will be on modifying the transfer paper surface and not the textile surface.

#### **2.4.2. Textiles used in STP**

As earlier stated in this study, an optimal textile to be used in STP is polyester due to its hydrophobicity which gives a preferable affinity for the non-polar disperse dyes. Cellulosic fibers, e.g. cotton and wool (El-Halwagy, El-Sayad and El-Molla (2001)), contains hydroxyl groups which has a low affinity for the non-polar disperse dye. Therefore, modifications of the fabric surface are required to achieve a fine patterned product (Bemska and Szkudlarek (2013)).

#### **2.4.3. Chemistry and physics of dyes used in STP**

Depending on which type of cellulose fabric that is used in the STP process, one has to consider the chemical composition and physical appearance of the disperse dye (Yin *et al.* (2013)). Described below are disperse inks used for polyester, and disperse inks used for cellulose substrate such as transfer paper and textile.

##### **2.4.3.1. Disperse dyes for polyester**

As mentioned, polyester consists of a composition of fibers with a hydrophobic nature. The polymer has a degree of crystallinity and the chains are closely packed together. This leads to reduced available interaction sites for the dye molecules. However, the structure of the dyes is planar and small, which advantageously results in that the dyes can maximize its interaction with

the polymer chains. The interaction sites are caused by the -CN and -NO<sub>2</sub> groups on the dyes that creates Van der Waals and dipole interactions to the polyester chains. In order for the dye molecules to diffuse into the polyester, heat needs to be applied where upon the polyester chains swells and the dyes moves until an interaction site is found. When cooling the polyester, the chains will return to its crystalline structure and the dye molecules will be locked in its position (Tyler (2010)). It is also of highest importance that the chosen disperse dye also has sublimation properties for it to work as desired.

#### 2.4.3.2. Disperse dyes for cellulose based substrates

Cellulose, which is a hydrophilic organic compound that can be used as carrier substrate for inkjet based patterns when transferring a print to a textile, requires water soluble or water dispersed dye in comparison to dye used for polyester. One possible dye used for cellulose is molecules with sodium carboxylate groups. Sodium carboxylate groups on the dye molecule provides the molecule with hydrophilic properties, which will interact with acidic sites on paper (Sarkodie *et al.* (2018)).

### 2.4.4. Measurement and techniques

To investigate the surface and structural properties of the transfer paper before and after printing, measurements can be done by using standard measurements. The measurements are described below.

#### 2.4.4.1. Paper before and after transfer

Measurements on the transfer paper needs to be done before and after transfer printing to the textile to see if the properties and structure has changed upon transfer printing. The measurements can be made by using e.g. scanning electron microscopy, tensile tests, water absorptiveness etc. The following methods are described below.

## **Structural and surface properties**

### *Scanning electron microscopy (SEM)*

Scanning electron microscopy is a tool that can be used to characterize the cross-section and the surface properties of a paper. A close-up, three dimensional and detailed images will be produced. The instrument generates an electron beam that is directed at a sample with the help of electromagnetic lenses (Bohelke (2018)). The electrons will interact with the specimen, which will produce signals (e.g. reflected and scattered electrons) that give information about the composition and topography of the specimen (Scanning Electron Microscopy (2019)).

### *Back-scattered electron imaging (BSE)*

Back-scattered electron imaging can be used to analyze a coated paper surface. The detectors are generally integrated within an electron probe micro-analyzer (EPMA) or scanning electron microscope (SEM). The detectors are generally placed above the specimen and collects scattered electrons as a function of the composition of the specimen. Detectors placed on the side of the specimen collects the scattered electrons as a function of the topography of the surface (Goodge (2016)).

### *Air permeance*

To investigate the porosity of the coated paper sample, the air permeance can be measured by using the Bendtsen method ISO standard 5636-3:2013, IDT. (ISO 5636-3:2013, IDT (2013)).

The air permeance of a coated paper sample was measured by using the Bendtsen method (ISO 5636-3:2013, IDT (2013)). The paper samples were placed in a Lorentzen & Wettre tester with a pressure of 1.47 kPa.

### *Coat thickness*

The thickness of the coating can be measured by using a Lorentz & Wettre instrument according to the ISO standard 534:2011 (ISO 534:2011 (2011)). The instrument will measure the thickness of the coating at a selected number of measuring points on one paper sample.

### *Water absorptiveness*

Water absorptiveness can be investigated to examine how much water can be absorbed by the paper sample. This can be done by using the ISO standard ISO 535:2014(E), Cobb<sub>60</sub> as a method. The method implies that a paper sample is firstly weighed and then wettened. The paper sample is then weighed after being wettened to calculate how much water the paper has absorbed. (ISO 535:2014(E) (2014)). Details about the method is further explained in chapter 4.

## **Printing properties on printed paper before and after transfer**

### *Colour bleeding*

To achieve a printed pattern with high resolution, it is important to avoid bleeding. The bleeding phenomena is invasion of one colour into the neighbouring area, which decreases the resolution (Ujiie (2006)). The bleeding can be observed visually, or by using e.g. light microscopy.

### *Color density*

To examine the amount of ink that has moved from the transfer paper to the textile, one can measure the optical density of the transfer paper before and after transfer printing. The optical density can be measured by a densitometer, such as eXact Standard Spectrophotometer, where the instrument is simply placed on the transfer paper and with a click on a button, the density can be received (eXact Standard (2019)). The optical density is defined as by  $\log_{10}\left(\frac{I_0}{I}\right)$  where  $I_0$  is the incident optical intensity and  $I$  is the reflectance in optical intensity.

### *Colour penetration*

An important property of the transfer paper is that the ink does not penetrate through the paper, it should remain at the surface of the coated transfer paper. The penetration occurs due to porosity in the paper and can result in a decreased optical density on the surface. The penetration can be visualized by measuring the optical density on the reversed side of the paper (Ren'ai Li *et al.* (2015)).

#### 2.4.4.2. Textile after printing

After transfer printing, the optical density of the textile can be measured to evaluate how much of the ink that has transferred from the transfer paper to the textile.

### 3. Process

At the beginning of the project, a time plan was established (see Appendix 1). The time plan was divided in three broader sections. The first part includes a literature study on the transfer printing area (see Figure 3). The second part was a laboratory experimental section, which includes preparing coating formulas, coating experiments and inkjet printing. The third part contained the STP experiments, which overlapped with the laboratory experimental part. The characterization part includes SEM- and BSE-experiments, as well as examination of paper and print quality. Lastly, the results were evaluated, and the final report was written.

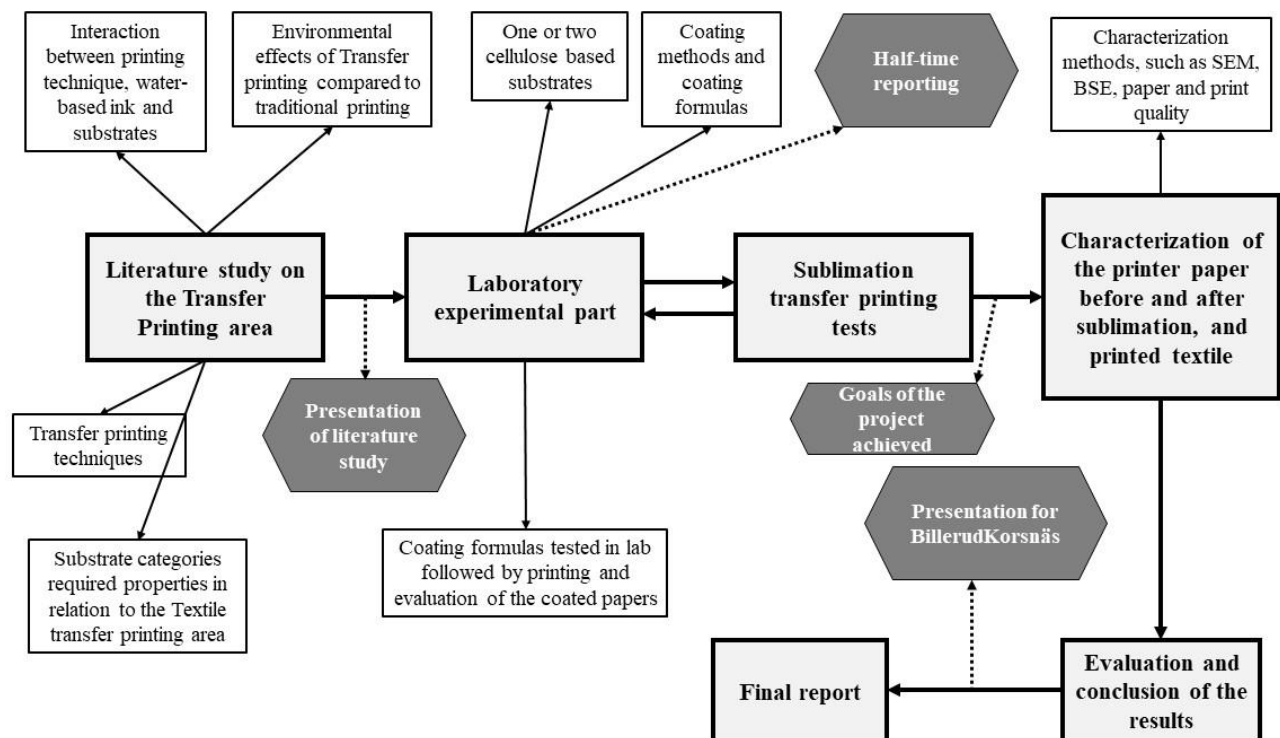


Figure 3. The figure shows a schematic workflow of the thesis project.

## **4. Materials and methods**

In this chapter, the materials and methods used in the laboratory part of the thesis will be presented. The two main parameters that was varied to achieve the goal of the thesis was the type of coating formula and the coat weight on the cellulose substrate. Different coating formulas were used, and different coat weights were applied on the papers.

### **4.1. Cellulose substrate**

The cellulose substrate used was Machine Glazed Paper (MG-paper) obtained by BillerudKorsnäs. Coating is done on only one side of a paper. The coating experiments were done on the glazed side of the MG-paper, and also on the non-glazed side of the MG-paper. Results later showed that coating on the non-glazed side gave a smaller variation in the result in coat weight when using the same coating conditions. Therefore, the experiments continued with only using the non-glazed side of the MG-paper.

### **4.2. Preparation of coating formula**

Chemicals used in the coating formulas were the polymer polyvinyl alcohol (PVOH) (see Figure 5) and ground calcium carbonate (GCC) that can be found in e.g. mineral rocks such as limestone. PVOH is prepared by hydrolyzing polyvinyl acetate using either an acid or base in the process (S.K.Saxena (2004)). Two types of PVOH were used during the experiments. The two PVOH have different degree of hydrolysis. The first PVOH, named PVOH A in this thesis, has a higher degree of hydrolysis than PVOH B.

Three coating formulas were prepared during the experiments. The goal was to achieve a coating that was enough porous for the ink to penetrate into the coating, and to release a high volume of ink during the STP process. The coating properties should also result in a short drying time of the ink and a nonvisible colour bleeding or colour penetration.

Firstly, pre-screening tests were done by using PVOH A as a coating formula to investigate how the chemical will react with the paper samples and to see if the chemical is suitable for STP. During

the inkjet tests, the results with PVOH A as coating formula showed unsatisfied results and the coating formula was modified. The pigment GCC was added to PVOH A with a ratio of 100 parts GCC and 7 parts PVOH A (Lamminmaki *et al.* (2009)). Later on, tests were also done by using PVOH B with GCC with the previous ratio to see if more improved results could be achieved if changing the degree of hydrolysis in the PVOH. The process of varying the coating formula parameter can be seen in Figure 4. The preparation of the coating formulas and viscosity measurements is described below.

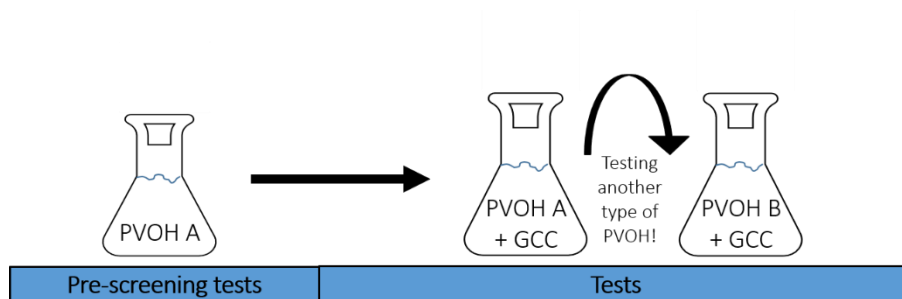


Figure 4. The figure shows the three coating formulas used in the experiments. Firstly, PVOH A was used as a coating formula for pre-screening tests. Secondly, GCC pigments were added to the PVOH A and lastly, another type of PVOH was tested to investigate any differences in the results.

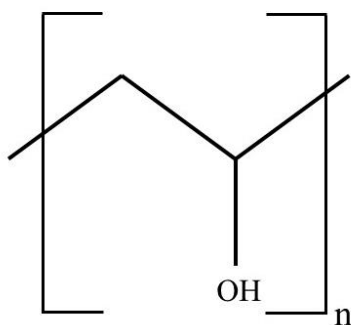


Figure 5. The figure shows the molecular structure of polyvinyl alcohol (PVOH).

#### 4.2.1. Preparation of PVOH A

A solution of PVOH A with a concentration of 9 wt% in dry content was prepared. Firstly, small amounts of PVOH A were added to room temperature (22°C) deionized water while stirring. Secondly, to dissolve the PVOH A, the solution was heated up to 90°C for one hour. The temperature of the solution was being measured during the heating process to avoid reaching a temperature higher than 95°C. A lid was added to the beaker with the heated solution to prevent evaporation of the PVOH-solution. Finally, when the PVOH A was dissolved and the solution gained a transparent appearance, the solution was cooled down to room temperature. The viscosity of the coating formula was measured as described in section 4.2.3.

#### 4.2.2. Preparation of PVOH A/B with GCC

Two solutions of PVOH and GCC were prepared with 60% dry content and a ratio of 7 parts PVOH and 100 parts GCC. The first solution prepared had a PVOH-content of type A and the second of type B. The PVOH-solutions were prepared as described above. The PVOH-solutions were then added to an amount of GCC. Furthermore, solutions of PVOH A + GCC with a dry content of 47% and 54% were prepared. The viscosities of the coating formulas were measured as described in section 4.2.3.

#### 4.2.3. Viscosity measurements of coating formula

The viscosity measurements were done by using a Brookfield viscometer DV-II + Pro. Spindle number two was selected and immersed into a beaker containing the coating formula. The rotation of the spindle was set to 100% rpm and the viscosity of the coating formula was measured in Centipoises (cP).

### 4.3. Coating of cellulose substrate

The MG-papers were prepared for coating by using two different methods which are further described below. The difference in the methods is the technique of stretching out the paper during the drying stage. It is of high importance that the paper is well stretched out during drying, otherwise the fiber in the paper will entangle (Tejado, A. 2010) and the smooth and even surface

will be disrupted. The paper samples were coated by using a rod coater. The goal was to achieve a maximum of  $\pm 1.0 \text{ g/m}^2$  in difference between the paper samples. The process is further described in section 4.3.2.

#### 4.3.1. Preparation of cellulose substrate

##### Method A

Firstly, the papers were weighed to receive the weight of the uncoated paper. To get the papers stretched, they were taped on a pad (see Figure 6). Secondly, the papers were coated by using a rod coater (see Figure 8). A coating formula was applied on the paper and coated by using a suitable rod.

##### Method B

As described in method A, the paper was firstly weighed. The papers were coated by a coating formula by using a suitable rod. This process is further described in chapter 3.4. As can be seen in Figure 7, the papers were stretched by placing the paper on pad and using clamps that surrounds the whole paper.



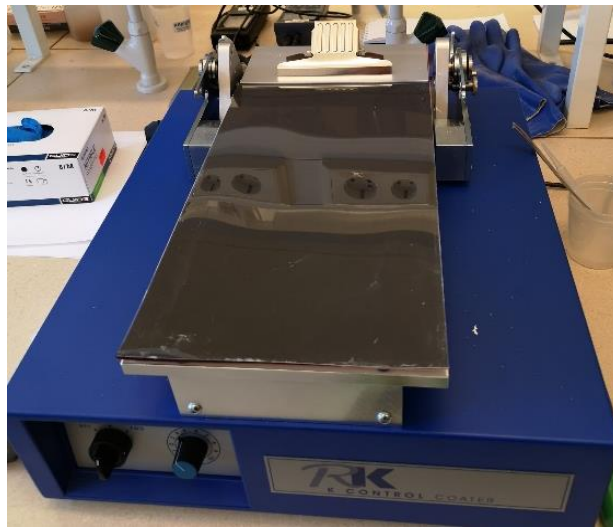
*Figure 6. The figure shows a paper taped on a pad that functions as a drying stabilizer used in method A.*



*Figure 7. The figure shows the clamps and the pad used in the drying process for method B.*

#### 4.3.2. Rod coater

A rod coater of the brand K Control Coater model 101 from RK Print coat Instruments (see Figure 8) was used to coat the paper samples. A chosen rod was applied onto a paper and a coating speed was selected. The rods are of different sizes, numbered 0 to 9, and will provide different coat weights (see Appendix 2) which is the second varying parameter in the experiments. Rod number 0 is of a smooth character, while a wire is added upon rods 1 to 9. The higher the rod number, the higher the diameter of the wire which results in a higher amount of the coating on the surface. For

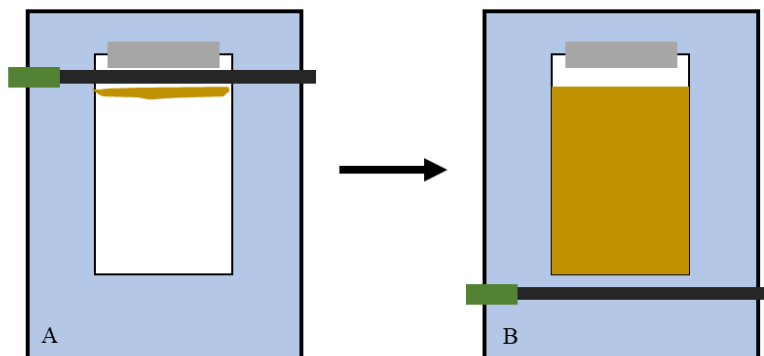


*Figure 8. The figure shows the rod coater used for coating during the experiments.*

more information about the different rods, see Appendix 2.

The selection of rod is also dependent on the viscosity of the coating formula. If a highly viscous coating formula is used, it is preferable to use a low numbered rod, otherwise lines may occur on the coating. The rod coater has 10 different speeds (see Appendix 3). The speeds of the coating process were selected between the highest and the lowest speed installation at the rod coater. Once the rod and rod speed were selected, the paper was applied on the rod coater and attached by a

clamp (see Figure 9). Approximately 2 ml of the coating formula was added on the paper in close contact with the rod.



*Figure 9. The figure shows the rod coater (blue) with a paper sample (white). The paper sample is fixed on the rod coater by a clamp (grey). The black vertical line with a green end at the left shows a rod. A) shows the paper before being coated. The brown horizontal shape shows the coating formula. B) shows the paper after coating.*

#### 4.3.3. Drying process

The paper was dried in an oven at 110°C for 4 minutes and then placed in a room with a climate of 50% humidity and 23°C for 3 hours to ensure that all the paper samples have been in the same climate before the experiments. Finally, the paper was cut out to a dimension of 22x14 cm<sup>2</sup> and then weighed.

#### 4.3.4. Design of coating experiments

The goal of the coating experiments was to achieve paper samples with different coat weights for each coating formula to investigate if the coat weight has an impact on the results of the STP. Therefore, different rods and rod speeds were selected. A table in Appendix 4 demonstrates the set of coating experiments. Generally, one batch contained 5-10 papers. One batch has the same coating formula, dry content, coating method, rod number and rod speed.

Firstly, pre-screening tests were made by using PVOH A as a coating formula with a dry content of 9%. For the experiments including pigments, the coating experiments were made using coating formulas with a dry content of 60%. The goal was to reach three different coat weights for PVOH A + GCC and PVOH B + GCC. Furthermore, the dry content was decreased to 47% to

attempt to obtain high coat weights without receiving any undesired lines in the coating caused by the wire from a rod. Also, experiments were done to receive extreme points in the coat weight by using a dry content of 54%.

#### 4.4. Calculations of coat weight

All paper samples, with a dimension of A4 (29.7x20.6 cm<sup>2</sup>), were weighed before being coated. After coating, the paper samples were cut into dimensions of 22x14 cm<sup>2</sup>. As can be seen in Eq. 1, the weight of the cut paper samples was calculated by multiplying the weight of the A4 with the dimension 22x14 cm<sup>2</sup>, divided by the area of the A4-paper dimensions (611.8 cm<sup>2</sup>). The coat weights were calculated by subtracting the weight of the coated 22x14 cm<sup>2</sup> paper samples with the weight of the 22x14 cm<sup>2</sup> paper samples before coating (see Eq. 2). For more details about how to calculate the coat weight, see Appendix 6.

$$m_{cut\ paper\ sample} = \frac{m_{A4} * A_{cut\ paper\ sample}}{A_{A4}} \quad (1)$$

$$m_{coat\ weight} = m_{coated\ cut\ paper\ sample} - m_{cut\ paper\ sample} \quad (2)$$

#### 4.5. Inkjet printing with water-based ink

To be able to print on the coated cut paper samples, the paper samples were taped on a A4 printing paper and inserted into a Virtuoso SG400-printer. The ink used was water based disperse Sublijet HD ink consisting of cyan, yellow, magenta and key-colour black. Five samples were selected from each batch (see Table 1 and Table 2). Note that this is not the case for the paper samples coated with only PVOH A (see Table 1) due to pre-screening tests where results from one inkjet test directly showed that the coating formula did not meet the desired properties. The paper samples selected from each batch had almost the same coat weights. An image (see Appendix 5) was printed on the paper samples (see Figure 10) and the optical density was measured as described in the following section. The colour used were water-based inkjet print suitable for STP. Also, the drying time of the ink on the printed paper samples was observed.



Figure 10. The figure shows a Virtuoso SG400-printer suitable for sublimation transfer ink with a coated paper sample that is taped on a A4-paper and has being printed with water-based ink.

Table 1. The table shows the paper samples that were used in the Inkjet printing experiment. The dry content of the coating formula was 9% for all the paper samples. The paper samples were chosen from 1-2 samples because of pre-screening tests.

Coating formula	MG-paper side	Paper sample(s) <sup>1</sup>	Rod	Rod speed (m/min)	Coat weight (g/m <sup>2</sup> )
PVOH A	Non-glazed	2	3	6.3	7.8 ± 0.9
PVOH A	Non-glazed	1	3	7.8	8.7
PVOH A	Non-glazed	1	4	6.3	7.2
PVOH A	Glazed	1	3	6.3	7.5
PVOH A	Non-glazed	3	2	6.3	2.2 ± 0.3
PVOH A	Glazed	3	2	6.3	2.1 ± 0.7

Table 2. The table shows paper samples that were used in the Inkjet printing experiment.

Coating formula	Paper samples	Rod	Rod speed (m/min)	Coat weight (g/m <sup>2</sup> )
PVOH A + GCC	5	0	12.0	4.9 ± 0.4
PVOH A + GCC	4	4	6.3	16.0 ± 0.1
PVOH A + GCC	5	1	3.5	6.4 ± 0.2
PVOH A + GCC	5	2	12.0	7.5 ± 0.2
PVOH A + GCC	5	3	12.0	9.5 ± 0.3
PVOH B + GCC	5	1	3.5	6.3 ± 0.4
PVOH B + GCC	5	2	12.0	6.8 ± 0.4
PVOH B + GCC	5	3	12.0	10.2 ± 0.3

<sup>1</sup> Low number of paper samples due to pre-screening tests

## 4.6. Measurements of optical density

The optical density of the printed images was measured by using the optical densitometer eXact from the company X-rite which can be seen in Figure 11. The measurements were done on each paper sample on the colours yellow = A, magenta = B, cyan = C and key-colour = K (see Figure 12)<sup>2</sup>. The measurements were done on the paper samples before and after STP (see Figure 13) to examine how much ink that still remained in the paper sample. Also, the optical density was measured on the polyester sheet (see Figure 16) to investigate the intensity of ink on the polyester, where a high intensity is preferred.



Figure 11. The figure shows the optical densitometer eXact from X-rite. The image shows the setup of one measurement.

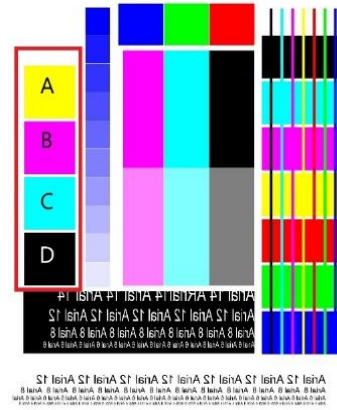


Figure 12. The figure shows the regions where the optical densities were measured for each paper sample. The region of A is yellow, B is magenta, C is cyan and D is key-colour black

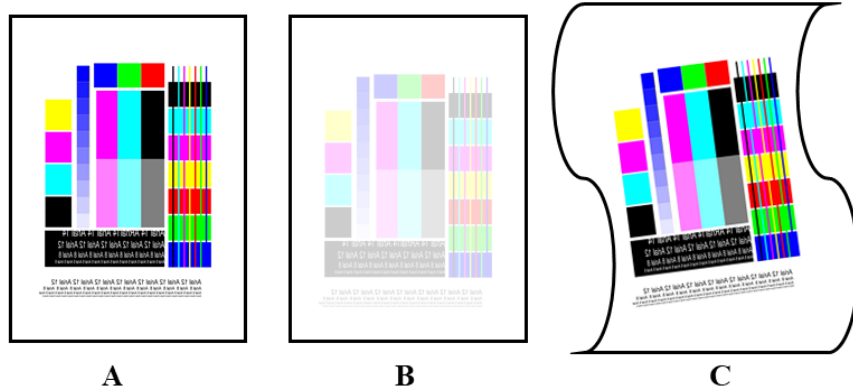


Figure 13. The figure shows the three samples where the optical densities were measured. A) is the printed coated paper sample before STP. B) is the printed coated paper sample after STP and C) is the polyester sheet after STP.

<sup>2</sup> The image does not correspond to the real image-resolution. See Appendix 5 for actual resolution.

#### 4.6.1. Colour penetration

The colour penetration was measured on all printed coated paper samples (see Table 2) after STP. The penetration was obtained by measuring the optical densities on the reversed side of the paper samples. The optical density was measured on three points on the black rectangular area (see Appendix 5).

### 4.7. Colour bleeding

Possible colour bleeding in the printed paper samples was measured in cross direction (CD) and machine direction (MD) (see Figure 15) by using a MATLAB script (see Appendix 7). The script converts an image to a binary image and segments the picture by thresholding. Three images from each batch were imaged. Firstly, printed paper samples were scanned to receive digital images. A region of interest was selected in the images where bleeding was visually present (see Figure 14). The MATLAB script calculated how many black pixels were present in the image. The number of black pixels received was subtracted to a number of pixels from a reference with no visible bleeding. The reference chosen was an uncoated MG-paper printed on the non-glazed side. The remaining pixels symbolized the bleeding in the image. The colour bleeding was measured on the printed paper samples coated with PVOH A/B + GCC before transfer printing. A diagram was made to visualize how the bleeding differs between the samples with different coating formulas and coat weights.

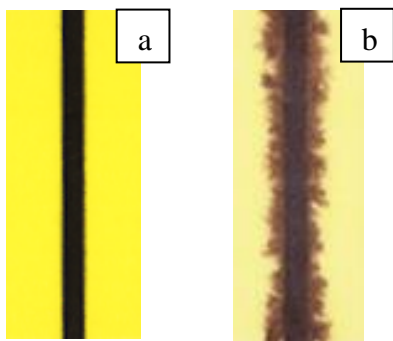


Figure 14. The figure shows the region of interest where bleeding was measured were a) is the reference and b) is an example of a sample with bleeding

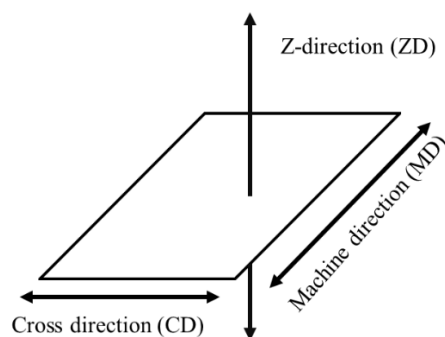


Figure 15. The figure shows the axial directions of a paper sample.

## 4.8. Sublimation transfer printing

The printed coated paper samples were placed on polyester sheets that and then were placed in a heat press (see Figure 16). The temperature of the heat press was 180°C and the samples were pressed for 50 seconds each. Afterwards, the optical density of the paper samples and the polyester was measured as described in chapter 4.6.

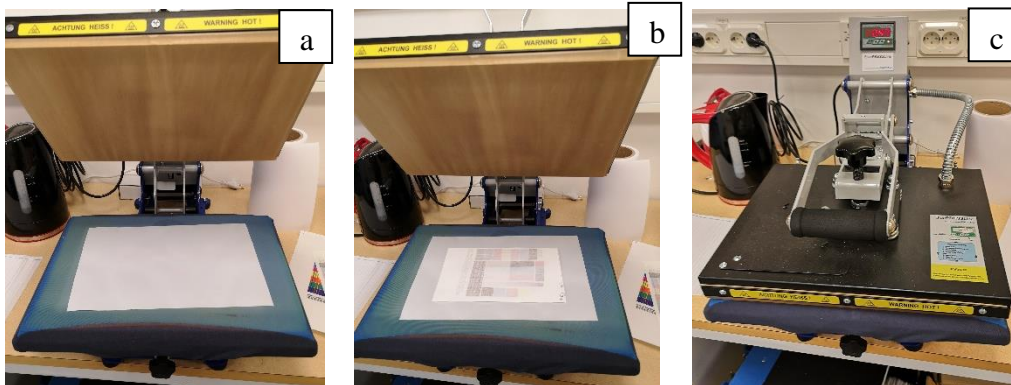


Figure 16. The figure shows the STP-process. a) The polyester sheet is placed on the heat press. b) A printed coated paper sample is placed upon the polyester sheet. c) The polyester and paper sample are heat pressed in 180°C for 50s.

## 4.9. Scanning electron microscope measurements

Coated paper samples were prepared for scanning electron microscope (SEM) analysis by being cut into a dimension of 1x1.5 cm. The samples were placed in a small chamber with springs that kept the samples in place. To be able to receive an even and fine surface of the cross-section samples and to steady the samples during the SEM-tests, epoxy was used. Epoxy enables grinding of the cross-section surfaces. A solution of epoxy was prepared, and the samples were sputtered with a thin layer of gold before contact with the epoxy, to avoid contact between the material and the epoxy. Epoxy was poured into the chamber until the solution had covered the samples. To eliminate air bubbles from the epoxy, the chamber was placed in vacuum for 15 minutes. The chamber with epoxy was then solidified during the night. The stump of epoxy with samples was removed from the chamber and sanded until transparent. A coating of carbon was then added to the stump to enable measurement with SEM. The stump was then placed in the microscope for measurements. Magnified images of the paper surface and cross-section images of the surface were

obtained by SEM and a back-scatter detector. Table 3 shows the samples used in the SEM-experiment.

*Table 3. The table demonstrates the coated paper samples that were used in the SEM-experiment with their different coating weights and dry content.*

Coating formula	Coating weight (g/m <sup>2</sup> )
PVOH A	5.1
PVOH A + GCC	16.4
PVOH A + GCC	5.7
PVOH B + GCC	11.0

#### 4.10. Determination of coat thickness

The thickness of the coating was measured by using the ISO standard 534:2011 (ISO 534:2011 (2011)). To measure the thickness, a Lorentzen & Wettre instrument was used. The thickness was measured on 5 points on one coated paper sample and an average value was obtained. The coat thickness was measured on one reference MG-paper and paper samples coated with PVOH A + GCC. Table 4 demonstrates the paper samples used for thickness measurements and their corresponding coat weights.

*Table 4. The table presents the paper samples used for determination of coat thickness.*

Paper samples	Coat weight (g/m <sup>2</sup> )
5	17.2 ± 0.5
5	8.0 ± 0.1
One reference (MG-paper)	0

#### 4.11. Determination of air permeance using Bendsten method

The air permeance of coated paper samples were measured by using the Bendtsen method (ISO 5636-3:2013, IDT (2013)). The air permeance were tested on paper samples coated with PVOH A and PVOH A + GCC to determine how the porosity differs between samples coated with pigments and without pigments. Also, the air permeance was measure on one reference MG-paper.

One paper sample was placed in a Lorentzen & Wettre tester with a pressure of 1.47 kPa. Each sample was measured on an area of 10 cm<sup>2</sup> and 5 testes were made (see Table 5).

*Table 5. The table demonstrates the paper samples, with corresponding coat weights, used for the air permeance test.*

Coating formula	Paper samples	Coat weight (g/m <sup>2</sup> )
PVOH A	2	6.7 ± 1.8
PVOH A + GCC	2	16.8 ± 0.3
PVOH A + GCC	2	8.0 ± 0.2
PVOH B + GCC	1	7.4
No coating	One reference (MG-paper)	0

#### 4.12. Determination of water absorptiveness – Cobb<sub>60</sub> method

To examine how much water coated paper samples absorbs, water absorptiveness tests were made on coated paper samples by using Cobb<sub>60</sub> as a method (ISO 535:2014(E), (2014)). Water was chosen to be able to compare with the results from the inkjet experiments, since the ink is water-based. All the paper samples were weighed before and after the tests to examine how much water had been absorbed into the paper. An open cylinder was place upon a coated paper sample and deionized water was poured into the cylinder. After 45 s the water was removed, and the sample was untouched for another 15 s. Afterwards, the paper sample was placed between two dry paper sheets, and a rolling pin was rolled over the sample to absorb excess water. The coated paper samples were weighed again to receive the water absorptiveness (g/m<sup>2</sup>). Also, one measurement was done on a reference paper. Table 6 show the paper samples that were used in the experiment with their corresponding coating formula and coat weight.

*Table 6. The table shows the paper sample used in the water absorptiveness tests with their corresponding coat weights.*

Coating formula	Paper samples	Coat weight (g/m <sup>2</sup> )
PVOH A + GCC	2	17.8 ± 0.2
PVOH A + GCC	2	8.0 ± 0
PVOH B + GCC	2	7.3 ± 0
No coating	One reference (MG-paper)	0

### 4.13. Statistical analysis

The results were statistically analyzed by using standard deviation. The standard deviation was calculated for different parameters, such as coat weight and colour bleeding. The standard deviation was calculated when the number of paper samples in one batch were greater than one. The equation for calculating the standard deviation is given in Eq. 3. The standard deviations are represented as black vertical lines in all diagrams and are also given in association with the coat weights for the paper samples.

$$s = \frac{\sqrt{\sum (x_i - \bar{x}_i)^2}}{n - 1} \quad (3)$$

*where  $s$  is the standard deviation,*

*$x_i$  is the value for one sample,*

*$\bar{x}_i$  is the mean value of  $x_i$ ,*

*$n$  is the number of paper samples in one batch.*

## 5. Results

### 5.1. Viscosity measurements

The results from the viscosity measurements are presented in Figure 17. The results show that the viscosity is similar between PVOH A and PVOH A + GCC. When changing the binder PVOH from A to B, the viscosity will increase significantly. Note that the dry content is the same between PVOH A + GCC and PVOH B + GCC.

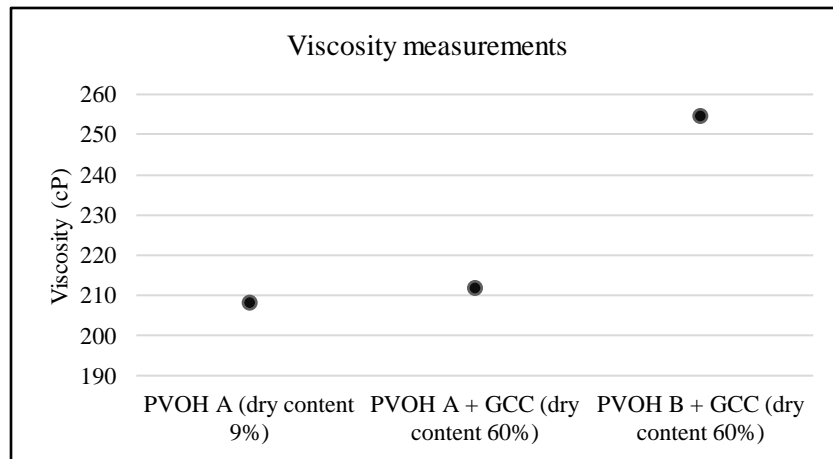


Figure 17. The figure shows the results from the viscosity measurements for PVOH A, PVOH A + GCC and PVOH B + GCC. The viscosity was measured one time for each coat weight.

### 5.2. Coat weights

The results from the coating experiments can be seen in Appendix 8. The results show a high variation between the coat weights in one batch when using method A. Additionally, the coat weight increases when increasing the rod number. These results are further explained in the following sections.

### 5.2.1. Coat weight depending on rod number

During the coating experiments, different rods were chosen to obtain different coat weights. Figure 18 confirms that when increasing the rod number, the coat weight will increase.

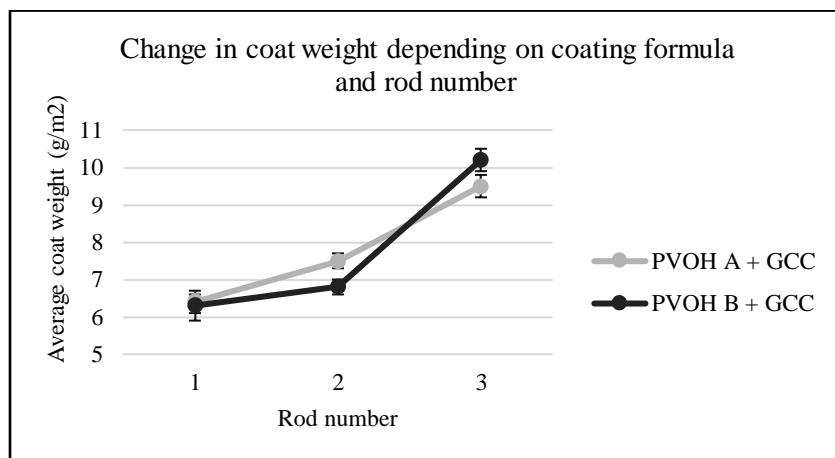


Figure 18. The figure shows the change in coat weight depending on rod number and coating formula that is used. The grey line corresponds to PVOH A + GCC and the grey line corresponds to PVOH B + GCC and the dots corresponds to one batch.  $n = 5$  in one batch. The black vertical lines correspond to the standard deviation of the coat weights.

### 5.2.2. Coat weight depending on method A/B

The difference in coat weight varied significantly in one batch when using method A in comparison when using method B. The goal was to achieve a maximum of  $\pm 1.0 \text{ g/m}^2$  in difference between the paper samples. Figure 19 shows the coat weights and the standard deviation (black vertical line) for 4 batches. The first two stacks are batches coated by using method A and the last two are batches when using method B. The standard deviation in the coat weight is significantly higher for the first two stacks, which marks a higher uncertainty in the results when using method A.

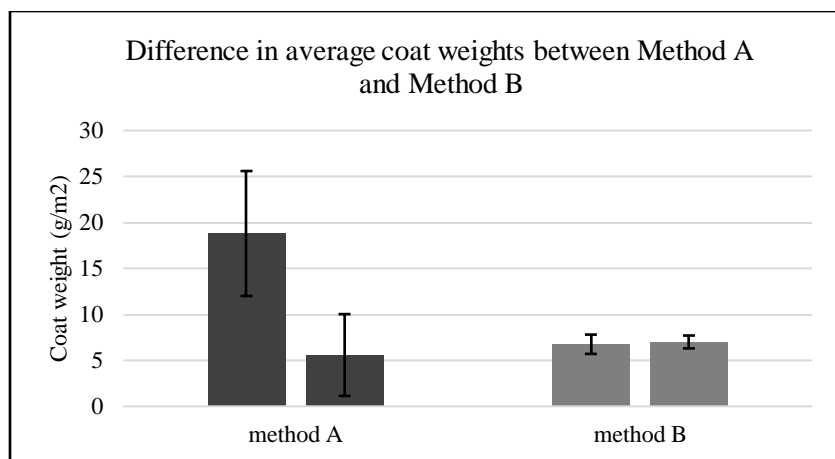


Figure 19. The figure demonstrates the difference in coat weight between different batches when using method A and method B.  $n=8$  in the first staple for paper samples coated with PVOH A + GCC using rod number 3 and speed number 6.3 m/min.  $n=10$  in the second stack for paper samples coated with PVOH A using rod number 2 and rod speed 6.3 m/min.  $n=7$  in the third stack for coated with PVOH A + GCC using rod number 2 and rod speed 6.3 m/min.  $n=8$  in the last stack for samples coated with PVOH A + GCC using rod number 2 and rod speed 12 m/min. The black vertical lines correspond to the standard deviation of the coat weights.

### 5.3. Inkjet printing with water-based ink

In this chapter, the results from the colour bleeding are presented. The optical density measurements are further presented in section 5.4. The drying time for the ink printed on the paper samples coated with PVOH A was estimated to more than 4 hours. The drying time was estimated to under 10 minutes for the samples coated with PVOH A/B + GCC.

#### 5.3.1. Colour bleeding

Figure 20 shows a printed paper sample from a batch that has the smallest colour bleeding. The paper samples were coated with PVOH A + GCC and had a coat weight of  $\sim 16 \text{ g/m}^2$ .

The results from the calculations of the colour bleeding are presented in Figure 21 and Figure 22. A high number of pixels (y-axis) corresponds to high bleeding in the printed coated paper samples. The figures show a trend where an increasing coat weight will firstly increase the bleeding. When using low number of rods, the colour bleeding will increase. When using rod number 3 and higher, the bleeding will start to decrease.

The colour bleeding was measured in paper samples coated with PVOH A/B + GCC. Due to extreme bleeding in printed paper samples coated with PVOH A, these samples were not measured. To see a printed paper sample coated with PVOH A, see Appendix 9. The results show that the colour bleeding is higher in the paper samples coated with PVOH B + GCC, where the bleeding is nearly 800 pixels higher in comparison to PVOH A + GCC with the same coat weight (6.7 ± 0.2 g/m<sup>2</sup>). The bleeding increases significantly between two samples with almost the same coat weight in samples coated with PVOH B + GCC although different rods were used during the coating process.



Figure 20. The figure shows the best results from the colour bleeding test where almost no bleeding is visible. The printed paper sample is coated with PVOH A + GCC and has a coat weight of 16.1 g/m<sup>2</sup>.

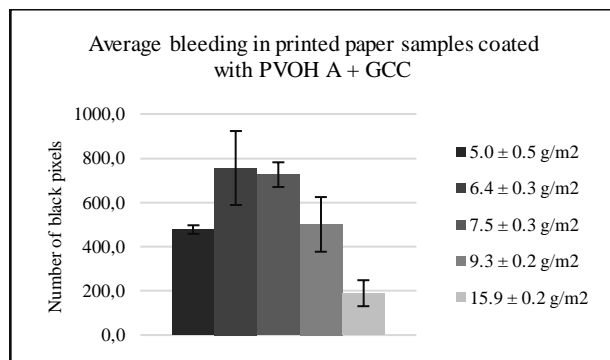


Figure 21. The figure shows the results from the colour bleeding calculations where the paper samples have been coated with PVOH A + GCC. Staple one is coated by using rod number 0, staple two is coated by rod number 1 etc. n = 3 for every batch and the black vertical lines correspond to the standard deviation for the colour bleeding.

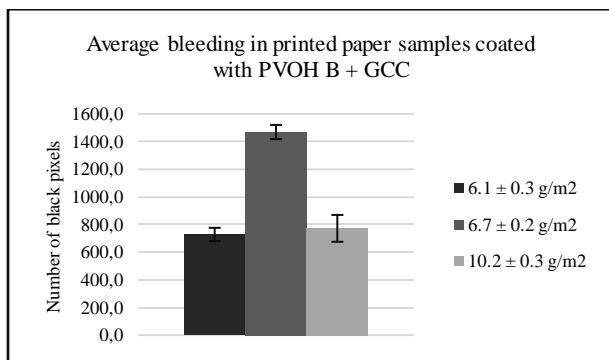


Figure 22. The figure shows the results from the colour bleeding calculations where the paper samples have been coated with PVOH B + GCC. Staple one is coated by using rod number 1, staple two is coated by rod number 2 and staple three is coated by rod number 3. n = 3 for every batch and the black vertical lines correspond to the standard deviation for the colour bleeding.

## 5.4. Optical density measurements

In this chapter, the results from the optical density measurements on printed coated paper samples and polyester are presented. Also, the result from the colour penetration is shown.

### 5.4.1. Paper samples before and after STP

The optical density measurements for printed coated paper samples coated with PVOH A + GCC and PVOH B + GCC are presented in Figure 23-Figure 26. The results show no significant difference in the optical density for the paper samples with different coat weights. However, the optical density is higher in key-colour (K) on printed coated paper samples coated with  $16.0 \pm 0.1$  g/m<sup>2</sup> PVOH A + GCC before STP, though it will not differ in optical density between the batches after STP.

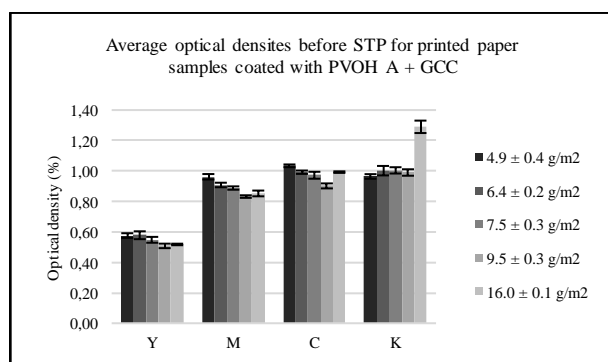


Figure 23. The figure shows the optical densities for 5 batches of printed coated paper samples before STP. The samples are coated with PVOH A + GCC.  $n=5$  for all batches except the last batch where  $n=4$ . The black vertical lines correspond to the standard deviation of the optical densities.

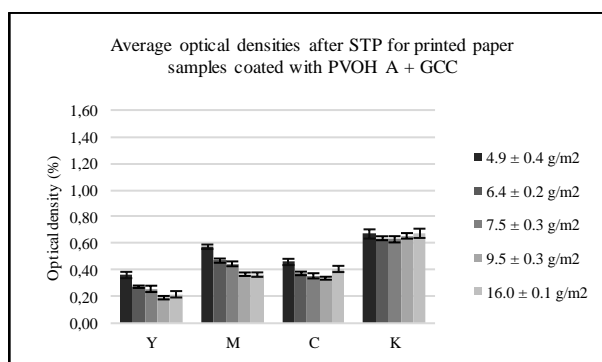


Figure 24. The figure shows the optical densities for 5 batches of printed coated paper samples after STP. The samples are coated with PVOH A + GCC.  $n=5$  for all batches except the last batch where  $n=4$ . The black vertical lines correspond to the standard deviation of the optical densities.

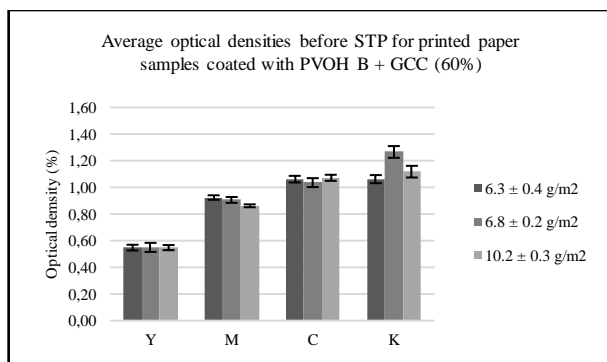


Figure 25. The figure shows the optical densities for 3 batches of printed coated paper samples before STP. The samples are coated with PVOH B + GCC.  $n=5$  for all batches. The black vertical lines correspond to the standard deviation of the optical densities.

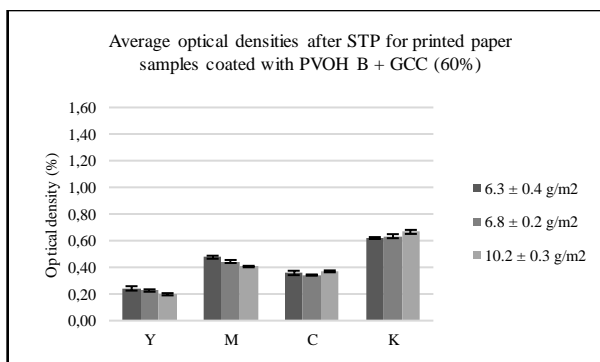


Figure 26. The figure shows the optical densities for 3 batches of printed coated paper samples after STP. The samples are coated with PVOH B + GCC.  $n=5$  for all batches. The black vertical lines correspond to the standard deviation of the optical densities.

#### 5.4.2. Polyester after STP

The optical density measurements for polyester sheets are presented in Figure 27 and Figure 28. The results show no significant difference between the optical densities for different coat weights.

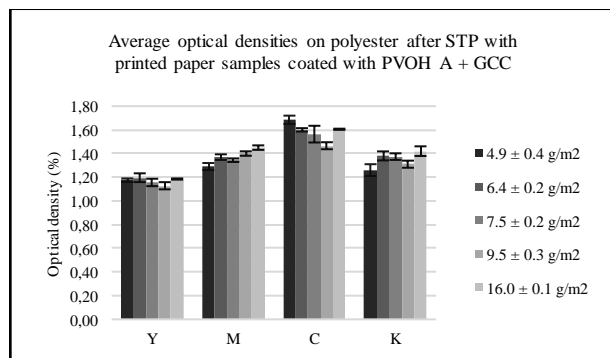


Figure 27. The figure shows the optical densities for polyester after STP. The paper samples used in the STP are coated with PVOH A + GCC.  $n=5$  for all batches except the last batch where  $n=4$ . The black vertical lines correspond to the standard deviation of the optical densities.

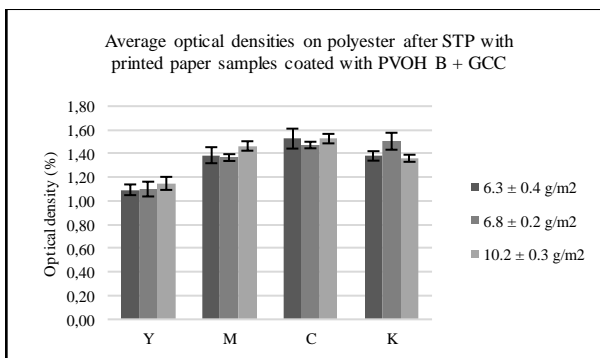


Figure 28. The figure shows the optical densities for polyester after STP. The paper samples used in the STP are coated with PVOH B + GCC.  $n=5$  for all batches. The black vertical lines correspond to the standard deviation of the optical densities.

### 5.4.3. Colour penetration

The results of the colour penetration in printed coated paper samples after transfer printing are shown in Figure 29 and Figure 30. The results for printed paper samples coated with PVOH A + GCC show that the penetration will decrease when increasing the coat weight, while the printed paper samples coated with PVOH B + GCC show no significant change when increasing the coat weight. An image of a visible colour penetration in a coated paper sample is shown in Appendix 10. The appearance of the penetration is of a spotted character, which results in a variation in the three measuring points at one printed coated paper sample. Visually, the results also show that the colour penetration will decrease for paper samples coated with PVOH A + GCC when increasing the coat weight.

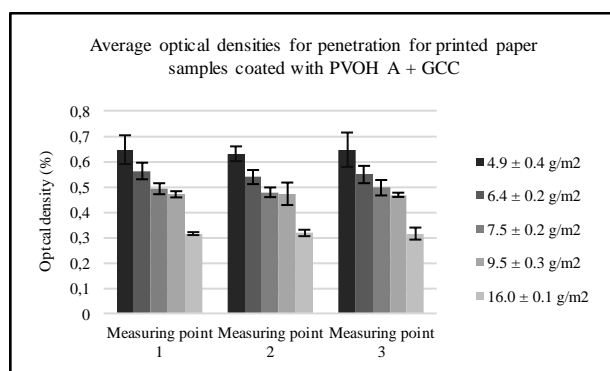


Figure 29. The figure shows the optical densities for penetration in printed paper samples coated with PVOH A + GCC.  $n=5$  for all batches. The black vertical lines correspond to the standard deviation of the optical densities.

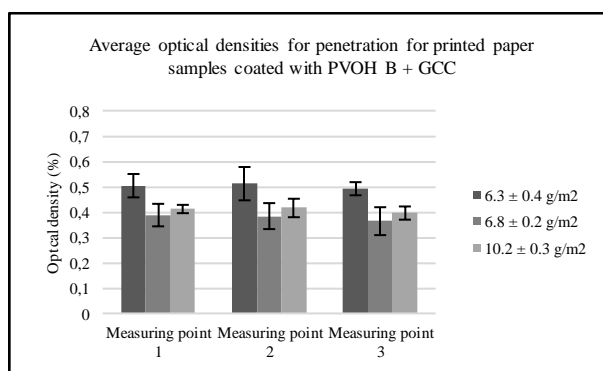


Figure 30. The figure shows the optical densities for penetration in printed paper samples coated with PVOH B + GCC.  $n=5$  for all batches. The black vertical lines correspond to the standard deviation of the optical densities.

## 5.5. Scanning electron microscopy measurements

The results from the SEM-measurements are shown in Figure 31-Figure 40. Section 5.5.1 shows surface images with a magnification of x5000. Surface images with smaller magnification can be seen in Appendix 11. Cross-section images of the coated paper samples are presented in section 5.5.2. Cross-section images of the coated paper sample surface can be seen in Appendix 12.

### 5.5.1. Surface images with a magnification of x5000

The results show that a film has been created when using the PVOH A as coating formula (see Figure 32). In Figure 33-Figure 35, the paper samples have been coated with PVOH A/B + GCC. In comparison to the reference (see Figure 31), the paper samples coated with pigments has smaller stone-like figures on the surface.

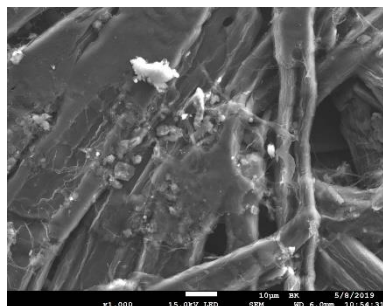


Figure 31. Reference surface (x5000).

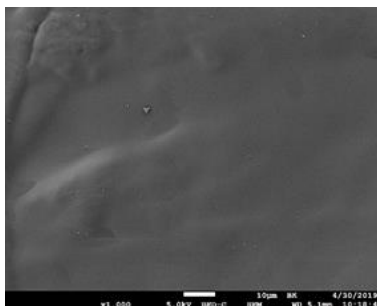


Figure 32. Paper sample surface (x5000) coated with PVOH A. Coat weight 5.1 g/m<sup>2</sup>.

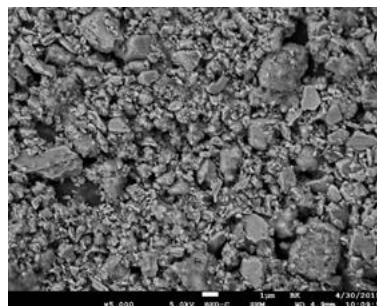


Figure 33. Paper sample surface (x5000) coated with PVOH A + GCC. Coat weight 5.7 g/m<sup>2</sup>.

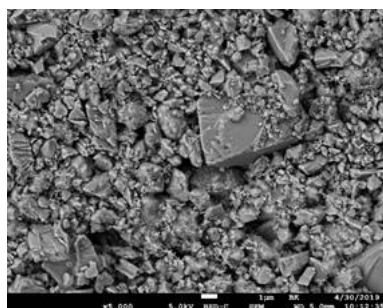


Figure 34. Paper sample surface (x5000) coated with PVOH A + GCC. Coat weight 16.4 g/m<sup>2</sup>.

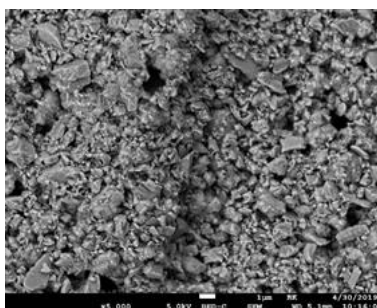


Figure 35. Paper sample surface (x5000) coated with PVOH B + GCC. Coat weight 11.0 g/m<sup>2</sup>.

### 5.5.2. Cross-section images

Figure 36 shows a cross-section image of a reference MG-paper sample with no coating. The uneven surface on the reference sample can easily be distinguished from the other coated paper samples. The cross-section images of the paper samples show that a smaller film has been added on the paper sample surface coated with PVOH A (see Figure 37). Figure 38-Figure 40 shows paper samples coated with PVOH A/B + GCC. Coated paper sample in Figure 38 has a thinner and more uneven coating than coated paper sample in Figure 39 and Figure 40, which is presumably due to a lower coat weight.

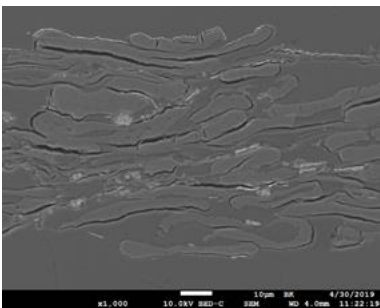


Figure 36. Cross-section image of reference (x1000).

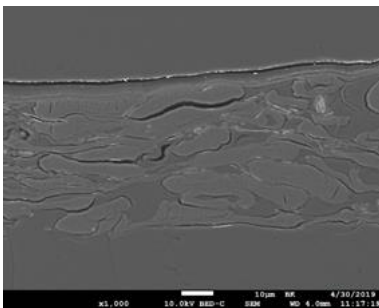


Figure 37. Cross section image of paper sample (x1000) coated with PVOH A. Coat weight 5.1 g/m<sup>2</sup>.

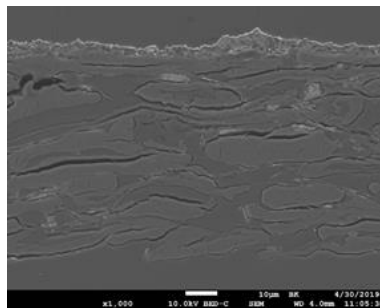


Figure 38. Cross section image of paper sample (x1000) coated with PVOH A + GCC. Coat weight 5.7 g/m<sup>2</sup>.

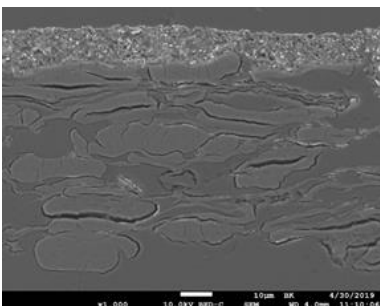


Figure 39. Cross section image of paper sample (x1000) coated with PVOH A + GCC. Coat weight 16.4 g/m<sup>2</sup>.

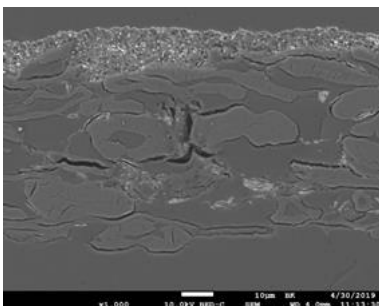


Figure 40. Cross section image of paper sample (x1000) coated with PVOH B + GCC. Coat weight 11.0 g/m<sup>2</sup>.

## 5.6. Thickness of coating

The results from the thickness of coating measurements are presented in Figure 41. results show that when increasing the coat weight, the thickness of the coating on the paper sample also increases. The standard deviation shows the variation within one batch.

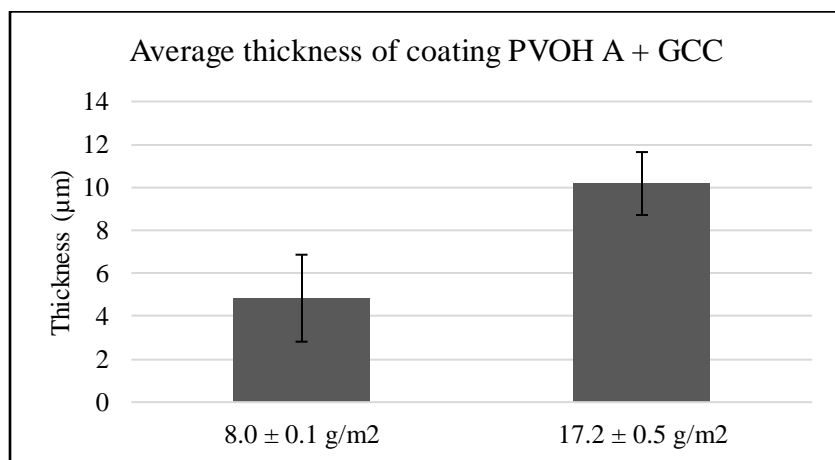


Figure 41. The figure shows difference in thickness of coating depending on the coat weight.

## 5.7. Air permeance

The results from the air permeance tests on paper samples coated with PVOH A + GCC are shown in Figure 42. The first staple is a non-coated MG-paper reference, which shows a high air permeance in comparison to the second and third batch which are coated paper samples. The air permeance of the coated paper samples decreases when increasing the coat weight. The results from the air permeance test on paper samples coated with PVOH A showed no air permeance and can therefore not be seen in the figure. This means that the paper samples coated with PVOH A has no porosity.

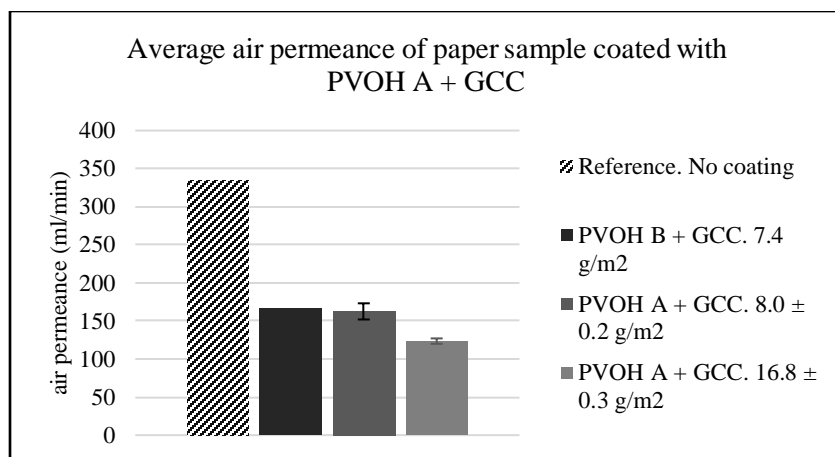


Figure 42. The figure shows the difference in air permeance of a paper sample depending on the coat weight. The reference consists of one paper sample. The second stack consist of  $n=1$  sample and the last two stacks consists of  $n=2$  samples. The black vertical lines correspond to the standard deviation of the air permeance.

## 5.8. Water absorptiveness as Cobb<sub>60</sub>

Figure 43 demonstrates the results obtained from the water absorptiveness experiment. The results show that the paper samples coated with PVOH B + GCC have a lower water absorptiveness than the samples coated with PVOH A + GCC with almost the same coat weight. The results also show that when the coat weight increases, the water absorptiveness will increase.

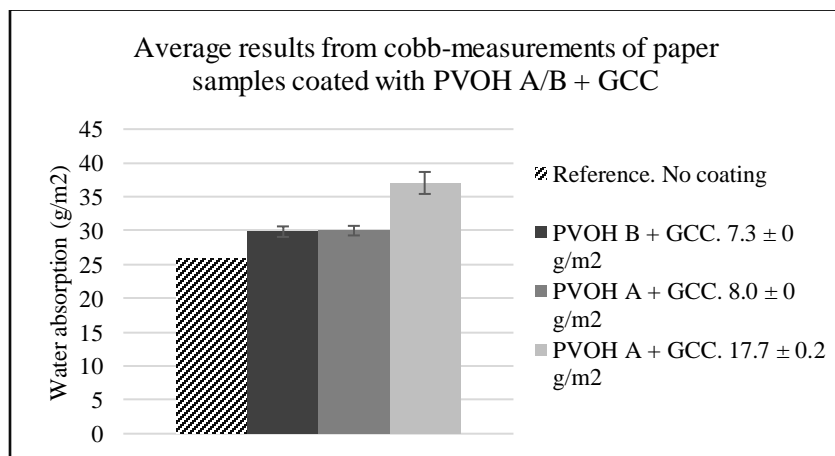


Figure 43. The figure shows the difference in water absorptiveness between paper samples coated with different formulas. The striped staple shows the reference test which consist of one sample. The other staples, or batches, consist of  $n=2$  samples per batch.  $n=5$  for all batches. The black vertical lines correspond to the standard deviation of the optical densities.

## 6. Discussion

In this chapter, the results of the experimental work will be discussed and analyzed. Furthermore, the process of the project will be evaluated.

### 6.1. Analysis of the experimental work

Below is an analysis of the results of experimental work which includes cellulose substrate, preparation of cellulose substrate, optical density measurements on paper samples and textiles, colour bleeding, colour penetration and SEM-images.

#### 6.1.1. Cellulose substrate

The cellulose substrate used for the laboratory experiments was MG-paper. The MG-paper has one glazed side with a smooth surface structure that has been dried on a Yankee-cylinder. The results showed that a smaller variation in the coat weight was obtained when coating on the non-glazed side of the paper, therefore the experiments continued on the non-glazed side. One possible explanation is that the coating formula functions as a gap filler and will fill in the unevenness in the non-glazed structure. The coating will even the surface, resulting in a smooth and printable transfer paper. The glazed side is already of a smooth character, which may result in a more difficult adherence of the coating formula to the surface.

#### 6.1.2. Preparation of cellulose substrate

At the beginning of the project, the paper samples were coated by using method A and then dried. The method, which implies attaching the paper sample to a pad by using tape, was a method resulting in different outstretched paper samples. It was challenging to obtain a paper sample that was plane on the pad. Instead, an oblique paper was received being stretched differently on the long side of the paper. The coating formula would therefore fill in the unevenness, resulting in accumulations of coating on the paper surface. Therefore, another method (method B) was developed where the paper was stretched after being coated. Consequently, the paper samples were plane during the coating process and stretched during drying. In these experiments, the coating became successfully even.

### 6.1.3. Colour bleeding

The paper samples that were coated with PVOH A had a long drying time after being printed with the inkjet printer. The drying time was more than 4 hours which resulted in that the ink was continuously bleeding. This is due to that a non-porous film had been created on the surface, which impedes absorption of molecules into the coating. This also affects the water that the ink-molecules are dispersed in. The water molecules will also be located at the surface of the paper, causing the ink-molecules to move in any direction. As a consequent, the coating formula was modified and pigments (GCC) were added to PVOH A to see if the drying time would shorten and the bleeding would decrease. Results confirmed that the porosity increased with adding pigments into the coating formula. The drying time was decreased for the samples coated with the pigment, where the drying time was less 10 minutes.

Furthermore, the results from the air permeance tests shows that the porosity decreases when increasing the coat weight. This also leads to a decreasing colour bleeding. This can be due to that the ink molecules will have an impeding mobility if the porosity is lowered, but if the porosity is equal to zero the ink will instead behave as the results from the coating experiments using PVOH A. One also has to consider the reference test, where the colour bleeding is very low for the non-coated MG-paper which has high porosity. The results from the colour penetration experiments show that the penetration of ink is high in the reference test. This outcome can be correlated to the high porosity of the uncoated paper, which makes the colour bleed in ZD-direction instead of CD and MD (see Figure 15).

When comparing the printed paper samples coated with PVOH A + GCC and PVOH B + GCC, the results shows that the colour bleeding is smaller for paper samples coated with PVOH A + GCC. As stated in the method, PVOH A is of a higher degree of hydrolysis than PVOH B. This means that there are more polyvinyl acetate molecules in PVOH B than PVOH A. One theory for the difference in bleeding, is that the polyvinyl acetate molecules may be less hydrophilic than PVOH due to the lack of alcohol as a functional group. This may result in more favorable interactions between polyvinyl acetate and the hydrophobic ink molecules, causing the ink molecules to bleed until finding an available interaction site.

As stated, results (see Figure 21 and Figure 22) show that the bleeding increases with an increasing coat weight until reaching a certain point where the bleeding will decrease. For samples with a high coat weight ( $16.3 \pm 0.2 \text{ g/m}^2$ ), the bleeding will decrease significantly. This appearance may be due to that different rods were used when applying the coating formulas on the paper sample surfaces. Note that the rods are of different shape due to the wire that will increase in diameter with increasing rod number (see Appendix 2). Different rods may result in different structures and shape of the coating formula, even though same coating formula is used. One example is the results from SEM (see Figure 54-Figure 57) where the images with PVOH A + GCC as a coating formula show two different structures of the coating. The coating with low coat weight has an uneven structure in comparison to the coating with high coat weight. When using rod number 2, the colour bleeding will be high when coating with PVOH A/B + GCC (see Figure 21 and Figure 22). One theory is that PVOH molecules are dominating at the surface and therefore the majority of interactions will be between PVOH A/B and the ink molecules. This will lead to an increasing colour bleeding which can be correlated with the results obtained from the ink-jet printing when coating with PVOH A (see Appendix 9). One could use infrared absorption spectroscopy (IRAS) to confirm this statement, if the binding between PVOH and ink molecules is known.

In correlation with the results from the colour penetration, the results indicate that when increasing the coat weight, the colour penetration decreases. Therefore, it is more favorable to have paper samples with high coat weight than paper samples with low coat weight due to the colour penetration into the paper samples.

As a conclusion, it is preferable to have high coat weight and low porosity of PVOH A + GCC to avoid bleeding and penetration in printed paper samples. Results from the air permeance tests correlated with colour bleeding for paper samples coated with PVOH A shows that there should be some degree of porosity in the coating to avoid bleeding.

#### 6.1.4. Optical density measurements

The optical density measurements showed no significant difference between the batches and the paper samples in the same batch. At the end of the laboratory experimental part, paper samples

were coated with PVOH A + GCC at a relatively low coat weight ( $5.1 \pm 0.5 \text{ g/m}^2$ ), and a high coat weight ( $15.7 \pm 0.5 \text{ g/m}^2$ ) to investigate if the optical densities would change if changing the coat weight to extremes. The same results were achieved where the optical densities did not show any significant difference between the previous printed batches. Therefore, the choice of coating formulas (PVOH A/B + GCC) and different coat weights will not have an impact on the optical densities of the printed paper samples before transfer printing, after transfer printing and the polyester.

#### 6.1.5. Colour penetration

As earlier stated, the results obtained in the colour penetration measurements indicates that the penetration decreases with an increasing coat weight in paper samples (see Figure 29 and Figure 30). Note that the appearance of the penetration is of a spotted character (see Appendix 10) and not an even penetration which means that there are uncertainties in the results. The spotted character may be due to paper fibers covering ink-molecules. However, the results can be strengthened by the visual appearance of the colour penetration which is decreasing when increasing the coat weight. The decreasing penetration with an increasing coat weight indicates that more ink molecules are absorbed into the coating, and not absorbed into the paper. This can be in compliance with the results from the coat thickness where the coat thickness increases with a higher coat weight and therefore the ink molecules are more likely to be located in the coating rather than in the paper.

The results from the colour penetration of paper sample coated with PVOH B + GCC (see Figure 30) shows that the penetration are higher for paper samples coated with smallest coat weight, but will decrease when increasing the coat weight. Note that the optical density is similar between the last two batches, and therefore it is difficult to draw conclusions of these results. If more coating experiments were done with increasing coat weight even for PVOH B + GCC, the results may would be similar to the printed paper samples coated with PVOH A + GCC.

### 6.1.6. Scanning electron microscopy (SEM)

The images received from the SEM-measurements shows that a film has been created on the paper surface when coating the paper sample with PVOH A (see Figure 32). This may explain the extreme bleeding (see Appendix 9) in the paper samples after inkjet printing due to more ink located at the surface of the coating. As explained in section 6.1.3, the ink will not penetrate the coating. Instead, it will move in CD and MD, causing the ink to bleed.

The SEM-images of the PVOH A/B + GCC coating (see Figure 33-Figure 35) shows that pigments are present as expected. The height of the different coatings can be investigated in the cross-section images in section 5.5.2. The results correlate with the results from the coat thickness measurements. The higher the coat weight, the higher the thickness of the coating. The results also show that the coated paper sample with a lower coat weight has an uneven coating surface in comparison to those with higher coat weights.

## 6.2. Analysis of the process

The workflow of the project was pursued according to Figure 3. The main workload was located during the laboratory experimental part and STP due to pivoting until achieving the goal of the thesis.

A first version of the time plan was established in the beginning of the project. The time plan treated the six main parts of the project separately, where no pivoting was included during the experimental parts. This design of process was alternated early in the project, when it was realized that it will acquire time to achieve the aim of the project. The project was finalized with writing of the final report.

Overall, the design of the process was easy and uncomplicated to follow and is recommended to future thesis writers.

### 6.3. Future work

As successful results were achieved from this thesis, it would be further interesting to investigate the most optimal coat weight to obtain neglectable colour bleeding for printed paper samples coated with PVOH A + GCC. As a suggestion, one should increase the coat weight even further than 16 g/m<sup>2</sup> to see if the colour bleeding will still have a decreasing appearance. Moreover, PVOH with other degrees of hydrolysis may be investigated to see if the results in the colour bleeding and colour penetration manages to improve. In terms of colour bleeding, one may consider to use the same rod during the rod coating experiments to investigate if the colour bleeding appearance received in this thesis would change. Also, different ratios between PVOH and GCC can be tested.

As results have shown in this thesis, ink molecules will still be present in the paper sample after STP. From environmental perspectives, it would be further curious to study if one printed paper sample can be used more than once in the STP process. Also, other types of textiles, such as a combination of cotton and polyester may be tested. The usage of cotton has shown to have great negative impacts on the environment and social communities, but one should not consider polyester (plastic) as a total substitute either. Therefore, a combination (50% polyester, 50% cotton) of the two textiles would be to prefer.

Furthermore, the scope of the study can be expanded to investigate how the results would work in industrial manner. Which coat weight is reasonable to work towards a profitable product? Is the chosen coating formula suitable for machines used in larger production scales or is it necessary to modify the viscosity? Also, one should examine the demand for such product in the textile industry.

## 7. Conclusion

- ❖ In comparison to coating on the glazed side of an MG-paper, coating on the non-glazed side gives a smaller distribution in coat weight between paper samples coated with same rod number and rod speed.
- ❖ In comparison to method A, method B will result in a small standard deviation in the coat weight between paper samples coated with the same conditions. The paper samples used in method B were plane during the whole coating process and the coating was successfully even on the paper.
- ❖ When changing the coating formula, the results showed:
  - No significant difference in colour density between printed paper samples coated with PVOH A + GCC and PVOH B + GCC nor at the textiles used in STP
  - No significant difference in colour penetration between paper samples coated with PVOH A + GCC and PVOH B + GCC
  - Colour bleeding is less in printed paper samples coated with PVOH A + GCC
  - No significant difference in porosity between paper samples coated with PVOH A + GCC and PVOH B + GCC, except when only using PVOH A as a coating formula which showed no porosity.
- ❖ When changing the coat weight, the results showed:
  - No significant difference in colour density between printed coated paper samples with different coat weights nor at the textiles used in STP
  - Colour penetration decreases when increasing the coat weight
  - Colour bleeding decreases when increasing the coat weight
  - Porosity will decrease when increasing the coat weight

The goal of the project was to find the most significant properties of a substrate when a specific coating formula was used and to achieve a successful transfer printing on textiles with high

resolution patterns. The results show that coating paper samples with PVOH A + GCC of a higher coat weight will meet these requirements.

## 8. References

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# Appendix

## Appendix 1 - Time plan

Project week	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
Calendar week	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23
Activity																				
Planning report																				
<b>MS1: Planning report</b>		•																		
Literature research and project planning																				
<b>MS2: Literature study ready to be delivered to supervisor at BK</b>				•																
<b>MS3: Literature study ready to be delivered to examiner and supervisor at LiU</b>					•															
<i>Experiments: Preparing and deciding coating formulas</i>																				
<i>Experiments: Test coating</i>																				
<b>MS4: Substrates and Coating formulas ready</b>								•												
<i>Experiments: coating on cellulose substrate surface</i>																				
<i>Experiments: Water-based inkjet printing on substrate</i>																				
<i>Experiments: STP on textile</i>																				
Preparing half-way presentation																				
<b>MS5: Half-way presentation</b>																				
<i>Characterization: Printed substrate surface</i>																				
Writing final report																				
<i>Characterization: Textile after STP and substrate after STP</i>																				
<i>Characterization: Best coated cellulose surface</i>																				
<b>MS6: Final report draft to ready to be delivered to BillerudKorsnäs</b>																				
<b>MS7: Final report draft ready to be delivered to opponent, examiner and supervisor at LiU</b>																				
Preparing final presentation																				
<b>MS8: Presentation of the Master thesis project</b>																				
<b>MS9: Presentation for the R&amp;D BillerudKorsnäs</b>																				

## Appendix 2 – Rod information.

Table 7. The table shows the different rod numbers and their corresponding wire diameter and wet film deposit. The rods are of the same brand as the rod coater, i.e K Control Coater model 101 from RK Print coat Instruments.

Rod number	Wire diameter (mm)	Wet film deposit ( $\mu\text{m}$ )
0	0.05	4
1	0.08	6
2	0.15	12
3	0.30	24
4	0.51	40
5	0.64	50
6	0.76	60
7	1.02	80
8	1.27	100
9	1.52	120

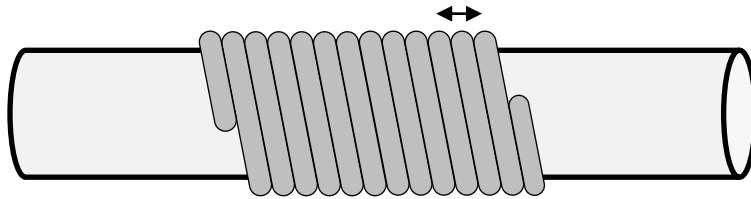


Figure 44. The figure shows a rod (light grey) and a wire (dark grey). The two-way arrow symbolizes the change in the diameter of the wire.

### Appendix 3 - Rod speed.

Table 8. The table shows the 10 speed numbers on the rod coater with their corresponding speed at m/min.

Speed number (display)	Rod speed (m/min)
1	2
2	~ 3.5
3	~ 4.9
4	~ 6.3
5	~ 7.8
6	~ 9.2
7	~ 10.7
8	~ 12.0
9	~ 13.6
10	15

## Appendix 4. Coating experiment setup.

Table 9. The table demonstrates the planning of the different coating experiments.

Coating formula	Dry content (%)	Coating method	Rod	Rod speed (m/min)	Paper samples
PVOH A	9	A	1	6.3	5
PVOH A	9	A	2	6.3	10
PVOH A	9	A	3	6.3	12
PVOH A + GCC	60	B	0	15	8
PVOH A + GCC	60	B	1	2	8
PVOH A + GCC	60	B	1	3.5	9
PVOH A + GCC	60	B	2	12.0	8
PVOH A + GCC	60	A	3	6.3	8
PVOH A + GCC	60	B	3	12.0	11
PVOH A + GCC	54	B	0	12.0	5
PVOH A + GCC	54	B	3	12.0	10
PVOH A + GCC	54	B	4	6.3	19
PVOH A + GCC	47	B	3	6.3	7
PVOH B + GCC	60	B	1	3.5	8
PVOH B + GCC	60	B	2	12.0	8
PVOH B + GCC	60	B	3	6.3	8

**Appendix 5.** Image of pattern used when inkjet printing on paper samples.

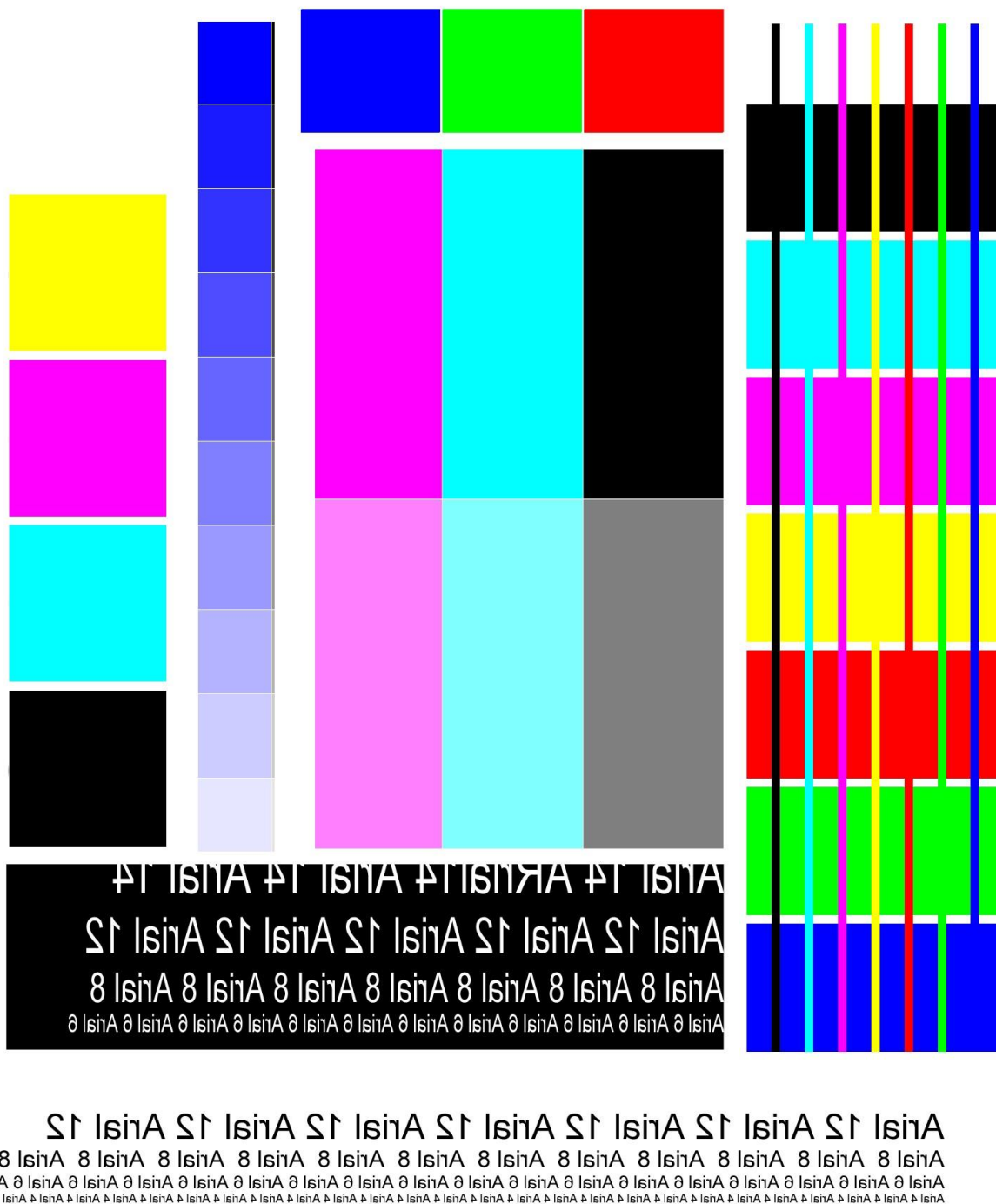


Figure 45. The image shows the pattern used when inkjet printing on paper. The image was used for quality printing evaluation.

## Appendix 6. Example of calculation of coat weight.

*Example: Below is demonstrated an example of how to calculate the coat weight of a paper, assuming the weight of an A4-paper is 3.734 g.*

Table 10. The table shows an example of numbers necessary to calculate the coat weight of a coated paper sample.

Sample	Weight before coating	Weight after coating	Area
A4	3.734 g	-	611.63 cm <sup>2</sup>
Cut sample	X	2.140 g	22x14 cm <sup>2</sup>

- a) Calculating the weight of the cut paper sample before coating:

$$x = \frac{3.734 \text{ g} * 22 \text{ cm} * 14 \text{ cm}}{611.63 \text{ cm}^2} = 1.88 \text{ g}$$

- b) Calculating the weight of the coating on the cut paper:

$$\text{Weight of coating} = 2.140 \text{ g} - 1.88 \text{ g} = 0.26 \text{ g}$$

- c) Convert the answer to g/m<sup>2</sup>:

$$\text{Weight of coating in g/m}^2 = \frac{0.26 \text{ g}}{22 \text{ cm} * 14 \text{ cm} * 10^{-4}} = 8.44 \text{ g/m}^2$$

## **Appendix 7.** MATLAB script for calculating bleeding.

```
i=imread('1.1599.tif'); %%  
imshow(i); %displays image  
  
%% Adjusting the image (intensity values are mapped)  
adjusted_image=imadjust(i,stretchlim(i));  
  
%% Convert the RGB image to gray-values  
gry=rgb2gray(adjusted_image);  
imshow(gry,[]);  
  
%% The image is segmented by using thresholding  
level=0.5;  
threshold_image=im2bw(gry,level);  
  
%% Morphological operation of the image  
%% 1206 is the number of black pixels in the reference image  
bw=bwmorph(threshold_image,'clean',20);  
imshow(bw);  
blackpixels = sum(bw(:) == 0);  
bleedinginimage=blackpixels - 1206
```

## Appendix 8. Results from coating experiments (coat weights).

Coating formula	Dry content (%)	Coating method	Rod	Rod speed (m/min)	Paper samples	Coat weight (g/m <sup>2</sup> )
PVOH A	9	A	1	6.3	5	2.8 ± 1.3
PVOH A	9	A	2	6.3	10	3.7 ± 2.8
PVOH A	9	A	3	6.3	12	4.5 ± 2.1
PVOH A + GCC	60	B	0	15	8	6.1 ± 0.5
PVOH A + GCC	60	B	1	2	8	6.0 ± 0.7
PVOH A + GCC	60	B	1	3.5	9	6.7 ± 0.6
PVOH A + GCC	60	B	2	12.0	8	7.2 ± 0.5
PVOH A + GCC	60	A	3	6.3	8	17.6 ± 5.0
PVOH A + GCC	60	B	3	12.0	11	10.4 ± 1.0
PVOH A + GCC	54	B	0	12.0	5	4.9 ± 0.4
PVOH A + GCC	54	B	3	12.0	10	8.1 ± 0.3
PVOH A + GCC	54	B	4	6.3	19	16.9 ± 1.2
PVOH A + GCC	47	B	3	6.3	7	6.7 ± 0.7
PVOH B + GCC	60	B	1	3.5	8	6.5 ± 0.5
PVOH B + GCC	60	B	2	12.0	8	7.0 ± 0.3
PVOH B + GCC	60	B	3	6.3	8	10.2 ± 0.7

### Appendix 9. Image of printed paper sample coated with PVOH A.



Figure 46. The image shows a printed paper sample coated with PVOH A before sublimation transfer printing.

## Appendix 10. Penetration of coated paper sample.

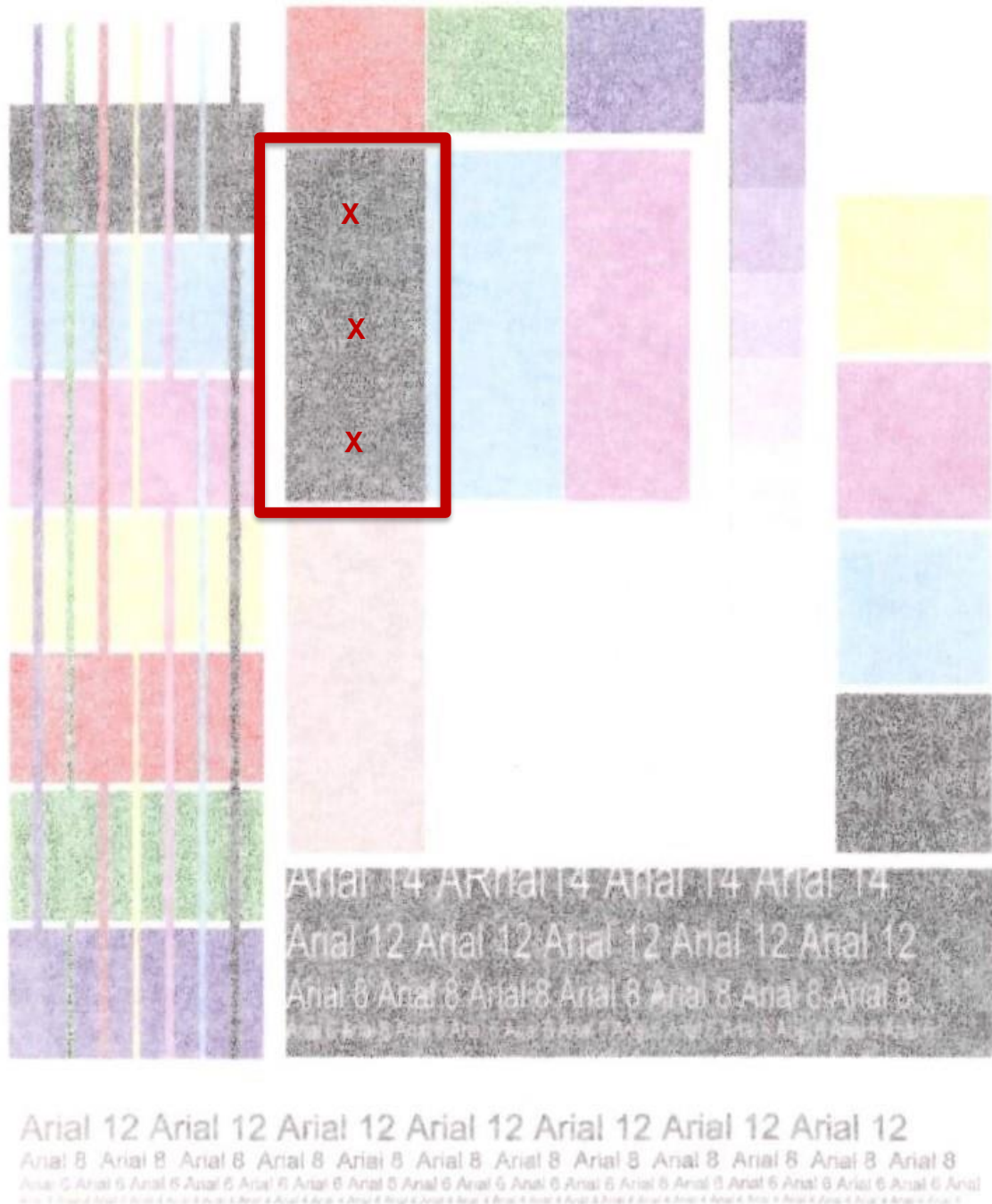
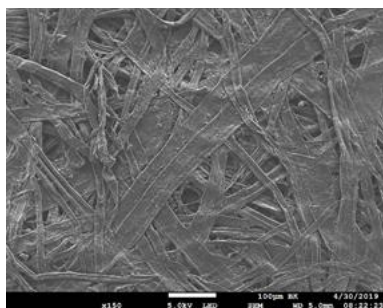
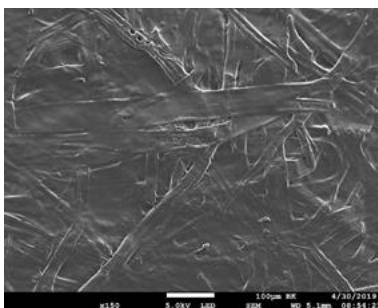


Figure 47. The figure shows an image of the backside of a paper sample coated with PVOH A + GCC that has a coat weight of 6.7 g/m<sup>2</sup>. The red box indicates the measurement area, where the three cross indicates the approximative measurement points.

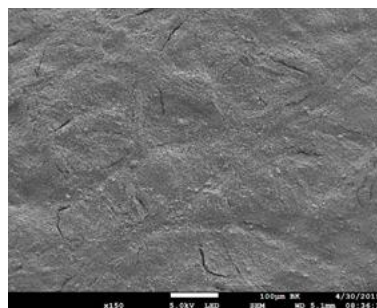
## Appendix 11. Surface images with a magnification of x150.



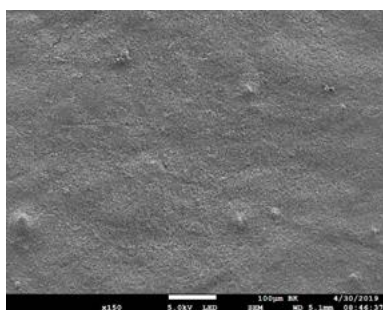
*Figure 48. Reference surface (x150).*



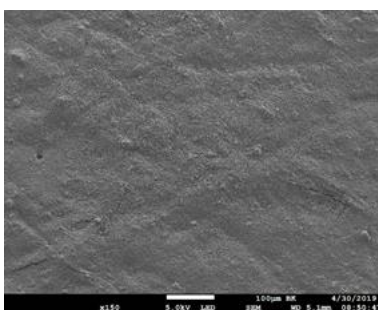
*Figure 49. Paper sample surface (x150) coated with PVOH A. Coat weight 5.1 g/m<sup>2</sup>.*



*Figure 50. Paper sample surface (x150) coated with PVOH A + GCC. Coat weight 5.7 g/m<sup>2</sup>.*



*Figure 51. Paper sample surface (x150) coated with PVOH A + GCC. Coat weight 16.4 g/m<sup>2</sup>.*



*Figure 52. Paper sample surface (x150) coated with PVOH B + GCC. Coat weight 11.0 g/m<sup>2</sup>.*

## Appendix 12. Cross-section images of paper surface.

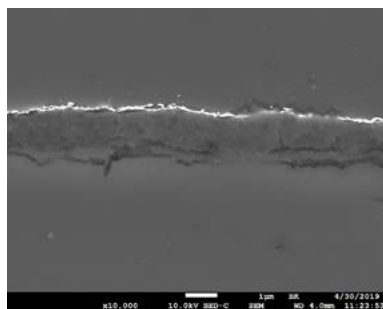


Figure 53. Cross-section image of reference surface (x10.000).

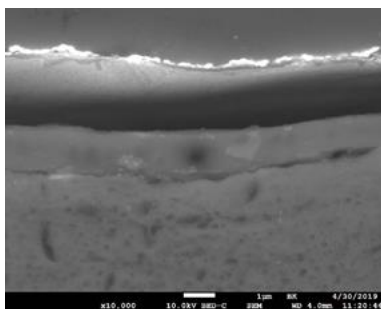


Figure 54. Cross section image of paper sample surface (x10.000) coated with PVOH A. Coat weight 5.1 g/m<sup>2</sup>.

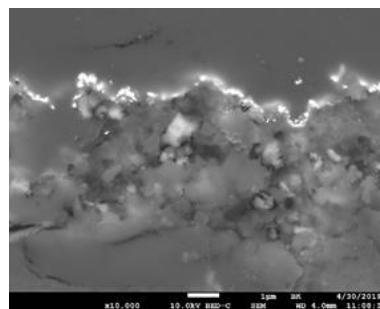


Figure 55. Cross section image of paper sample (x10.000) coated with PVOH A + GCC. Coat weight 5.7 g/m<sup>2</sup>.

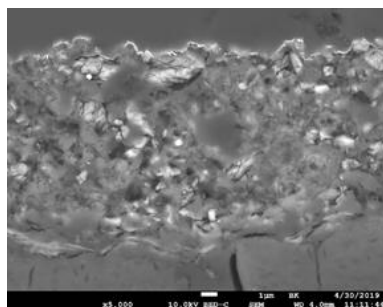


Figure 56. Cross section image of paper sample surface (x10.000) coated with PVOH A + GCC. Coat weight 16.4 g/m<sup>2</sup>.

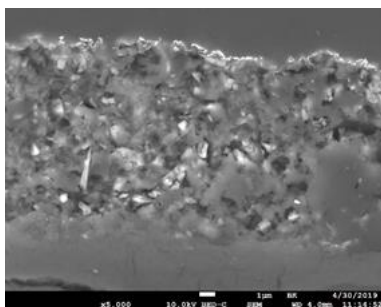


Figure 57. Cross section image of paper sample surface (x5000) coated with PVOH B + GCC. Coat weight 11.0 g/m<sup>2</sup>.

