New methods for evaluation of tissue creping and the importance of coating, paper and adhesion

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Abstract

Increased competition on the tissue market forces tissue makers to continuously raise their product quality and increase their operating efficiency. The creping process and the conditions on the Yankee cylinder are the key factors in the production process and they therefore need to be kept under good control in order to maintain a high and uniform quality. Due to evaporation, dissolved substances from the wet end and fiber fragments remain on the surface and a natural coating layer always develops on the surface of the Yankee cylinder. However, coating chemicals are also needed and these are continuously sprayed onto the Yankee surface in order to modify the adhesion between the paper and the dryer cylinder. To make it possible to control the process better, on-line measurements of coating thickness as well as of the crepe structure of the tissue paper produced would be very valuable.

The fiber furnish affects the adhesion between the paper web and the cylinder dryer. The strength and uniformity of the adhesion of the paper to the cylinder affects the creping process tremendously and more information about parameters affecting the adhesion is of great interest. To perform trials on a full scale, or even on a pilot machine, is very costly and therefore laboratory equipment is sought in order to be able to measure the adhesion force in a cheaper way.

In the work described in this thesis, the coating layer was analyzed both chemically and morphologically to obtain information about the coating layer before on-line measurements were started. The chemicals added and the constituents of the pulps are known by the paper producers, but exactly what type of material is left on the cylinder and whether there are different layers of coating material still remains to be investigated. The chemical analysis indicated that the adhesive content was higher in the inner layer than in the outer layer of the coating. The relative amount of polyamide-amine epichlorohydrin resin calculated on the basis of the nitrogen content in the resin was low, indicating that the coating layer consisted of a significant amount of carbohydrates or other substances from the wet end. The coating layer could not be considered transparent. It was observed that the coating was not uniform i.e. it was thick, had a patch-wise appearance and contained fiber fragments.

An uneven coating layer affects the adhesion and creping of the paper. Therefore, it was desirable to develop an on-line method to measure the coating thickness. The coating layer contains a lot of fiber fragments and the new method cannot rely on a transparent coating layer. Measurements on a laboratory scale, to be further applied on-line on the tissue machine, have been investigated. The thickness of the
coating layer on a laboratory dryer has been measured, using a method based on fluorescence with an optical brightener added to the coating chemicals sprayed on the Yankee dryer. With a UV-LED (Ultra Violet - Light Emitting Diode), the coating layer was exposed to UV-radiation and the intensity of the light emitted by the optical brightener in the layer was measured. The equipment clearly measures the signal strength of emitted light, but to be able to make good measurements on the coating layer further investigations must be carried out. Trials are for example required to see whether the adhesive itself disturbs the measurement method, and most important to investigate the reason for the disturbances in the measurements when cylinder is rotating.

In this project, new laboratory creping equipment and a new laboratory adhesion method where the equipment can operate with different creping angles was developed. The equipment is connected to a tensile tester to make it possible to measure the force needed to scrape off the adhered paper. It was found that beating of the pulp increased the adhesion. This was expected, since the fiber surface area increases with increasing beating and a larger area for adhesion will then be created. The adhesive used is not particularly reactive and fairly unaffected by pH changes. In the industry, the coating layer is builds up over time to reach a steady state. In this study, the papers with different pH's probably did not have sufficient time to affect the coating layer as much as in a commercial process. This is probably why the pH did not affect the adhesion. The pulp with the highest creping force was a Eucalyptus pulp consisting of 75% Grandis and 25% Globulus, which has a higher hemicellulose content than the other pulp types and also a larger amount of fines which increase the bonding strength in the coating layer.

The coating thickness is important for the adhesion between the paper and the Yankee cylinder and the coating thickness measurements could give a good idea of how the adhesion and therefore the structure of the creped paper vary. A more direct measurement can investigate the structure of the paper produced. From previous studies, it is known that a paper with a finely creped structure has a smoother surface than a coarsely creped paper. A finely creped structure corresponds to a short wavelength and vice versa. Wavelength measurements were made on the tissue paper with an optical fiber sensor which was mounted either perpendicular to or at angles of 10° and 45° to the paper surface. The paper was travelling at a low speed while the measurements were made. The collected signal was mathematically analyzed and the characteristic wavelength was calculated. The values for different paper samples were in close agreement with the wavelengths measured with an off-line method using a commercial crepe analyzer.
Sammanfattning

Vid mjukpapperstillverkning torkas papperet på en stor torkcylinder, Yankeecylinder, med ånga inuti cylindern och med hjälp av torkkåpor som blåser på hetluft på pappersbanan. Papperet klistras fast vid cylindern och efter ungefär ¾ rotation skrapas papperet av cylindern. Papperet rynkas (kräppas) vid avskrapningen och bildar ett papper med en viss kräppstruktur som påverkar papperets bulk, stretch och absorptionsförmåga.


Massan som papperet är tillverkat av påverkar vidhäftningen mellan papper och torkcylinder. Hur stark och jäm denna vidhäftning är inverkar på kräppningsprocessen och mer vetskap om hur olika massaparametrar påverkar är av högt värde. Att genomföra försök i fullskala och även i pilotskala, är mycket kostsamt och därför är en utrustning för att mätta kraften i vidhäftningen i labbskala eftersökt.

I den här avhandlingen har beläggningen på Yankeecylindern analyserats både kemiskt och morfologiskt, för att få nödvändig information om beläggningen innan val av utrustning till mätningar under drift. Papperstillverkare vet vilka kemikalier som sprayas på cylindern och vad som finns i pappersmassan, men vad som blir kvar på Yankeecylindern är mindre känt. Den kemiska analysen indikerade att det finns en gradient av adhesive i beläggningslagret, med högre koncentration närmast cylinderytan. Andelen kolhydrater från fibrer var hög och beläggningen kan inte anses vara genomskinlig, vilket påverkar valet av mätmetod för att mäta tjockleken på beläggningslagret. Lagret var inte jämntjockt utan förekom fläckvis tjockare och innehöll även delar av fibrer.

Ett ojämnt beläggningslager påverkar adhesionen och kräppningen av papper. Det är därför önskvärt att kunna mäta lagrets tjocklek och ojämnhet under drift.


Papers included in this thesis

I Chemical and morphological analyses of the tissue yankee coating
Jonna Boudreau, Holger Hollmark and Luciano Beghello

II A method of measuring the thickness of the coating on a dryer cylinder
Jonna Boudreau and Luciano Beghello

III Laboratory creping equipment
Jonna Boudreau and Christophe Barbier

IV The influence of various pulp properties on the adhesion between tissue paper and Yankee cylinder surface
Jonna Boudreau and Ulf Germgård
*(Submitted for publication)*

V Experiments to find online measurements of the structure of the tissue paper surface
Jonna Boudreau, Magnus Mossberg and Christophe Barbier
*(Manuscript)*
The author's contribution to the papers

Paper I: The author planned the experiments and collected samples. All analyses were made at different company laboratories.

Paper II: The author planned and performed the experiments with equipment put together by Lars Granlög (Innventia).

Paper III: The author planned the testing equipment and adhesion method. The experiments were performed together with Pauliina Kolari. Göran Walan (Karlstad University) planned the creping equipment together with the author and constructed the creping device and adhesion table.

Paper IV: The author planned the experiments and performed the experiments together with Pauliina Kolari.

Paper V: Ingemar Petermann (Acreo) put together the equipment and performed the experiments. Magnus Mossberg (Karlstad University) made mathematical analyses. The Nalco Company performed crepe frequency analyses. The author made pictures using an optical microscope, performed analysis on the pictures and led the project.
Related presentations and reports by the same author


Hedman*, J. (2007): A close-up on Yankee coating - for better control. A PhD project at Karlstad University, Presented at the 8th Tissue World, Nice, France, March 26-29.


*now Boudreau
Symbols and abbreviations

Symbols

\( F_x \) Creping force normal to the doctor blade
\( F_y \) Creping force tangential to the doctor blade
\( L \) Initial length
\( \Delta L \) Elongation
\( R_a \) Surface roughness
\( T_g \) Glass transition temperature
\( \alpha \) Blade angle
\( \beta \) Bevel angle
\( \gamma \) Creping angle
\( \theta \) Angle from paper surface

Abbreviations

AC/DC Alternating current/Direct current
AFM Atomic Force Microscope
ASE Amplified Spontaneous Emission
A.U. Arbitrary Units
CCD Charge-coupled device
Cl Chloride
CD Cross Direction
CTMP Chemithermomechanical pulp
DCT Dry Creping Technology
DSF Dynamic Sheet Former
DTPA Diethylene triamine pentaacetic acid
ECF Elemental Chlorine Free
EDTA Ethylenediaminetetraacetic acid
H Hydrogen
HCl Hydrogen chloride
HPAEC-PAD High Performance Anion-Exchange Chromatography with Pulsed Amperometric Detection
HW Hardwood
IR Infrared radiation
ISO International Organization for Standardization
MD Machine Direction
MUSIC Multiple signal classification
N Nitrogen
<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Full Form</th>
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<tbody>
<tr>
<td>NI</td>
<td>National Instruments</td>
</tr>
<tr>
<td>O</td>
<td>Oxygen</td>
</tr>
<tr>
<td>Paa</td>
<td>Peracetic acid</td>
</tr>
<tr>
<td>PAE/PAAE</td>
<td>Polyamidoamine-epichlorohydrin</td>
</tr>
<tr>
<td>PVA</td>
<td>Polyvinyl alcohol</td>
</tr>
<tr>
<td>R</td>
<td>hydrocarbon groups or chains</td>
</tr>
<tr>
<td>Ra</td>
<td>Roughness average (surface roughness)</td>
</tr>
<tr>
<td>S_p</td>
<td>Pseudospectra</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning Electron Microscope</td>
</tr>
<tr>
<td>SR</td>
<td>Schopper Riegler</td>
</tr>
<tr>
<td>SW</td>
<td>Softwood</td>
</tr>
<tr>
<td>TAD</td>
<td>Through-air-drying</td>
</tr>
<tr>
<td>TCF</td>
<td>Totally Chlorine Free</td>
</tr>
<tr>
<td>T_g</td>
<td>Glass transition temperature</td>
</tr>
<tr>
<td>USB</td>
<td>Universal Serial Bus</td>
</tr>
<tr>
<td>UV</td>
<td>Ultra violet radiation</td>
</tr>
<tr>
<td>UV-LED</td>
<td>Ultra Violet - Light Emitting Diode</td>
</tr>
<tr>
<td>WRV</td>
<td>Water retention value</td>
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1 Introduction

There are basically four ways of making tissue and towel products. Three of them produce paper that is creped, i.e. paper that has wrinkles caused by the action of a doctor blade that removes the paper from a drying cylinder. The dominant technology is the “dry crepe” process, where the thin paper is dried on a large drying cylinder, called a Yankee cylinder. Less common is the “wet crepe” process in which the paper web is creped before it is fully dried. In the wet crepe process, the required adhesion of the web to the drying cylinder is achieved with a thin liquid film. During recent decades, a third process called “Through-air-drying (TAD)” has been widely adopted, primarily in the US. The fourth, and not creped tissue, is called air-laid and is a nonwoven material. It is made from fluff pulp and is more like a textile than the other tissues.

In creped tissue production, the paper is scraped off the cylinder by a doctor blade. A longitudinal section through a creped paper can resemble a sinus curve with a wavelength and amplitude depending on many factors; for example the creping blade, coating chemicals and coating thickness and evenness on the cylinder. The frequency of the paper creping affects the paper quality enormously and it is necessary to measure this on-line to obtain a fast answer to the effects of a change in the paper machine. The coating thickness affects the crepe frequency and it is therefore also of interest to measure the thickness of the coating layer on-line. The creping process and adhesion between paper web and cylinder dryer are very important for the end product and the type of fibres and the different treatments and chemicals used strongly affect the adhesion.

Aim of the study

The aim of this work was to obtain a better knowledge of the coating layer and what the morphology of the coating surface is like. Interesting factors that could affect the adhesion between paper and cylinder dryer are: beating of the pulp, fibre type and pH. To be able to study different parameters, a laboratory creping equipment and adhesion method resembling the commercial tissue manufacturing process were developed. Another aim of this work was to make the tissue process more stable through on-line measurement of the coating thickness on the Yankee cylinder and the crepe wavelength of the paper. The thickness measurements are highly dependent on the morphology and content of the cured coating layer.
2 Theory

2.1 Tissue machine

A tissue paper is a lot lighter than other paper types. The grammage is between 12 and 50 g/m². Regular copy paper has a grammage of 80 g/m² and cardboard up to 300 g/m². The tissue machine is short, about 40 m long compared to other paper machines that can be up to several hundred meters long. The main difference from other machines is that the tissue machine consists of one large drying cylinder (about 5 m in diameter) instead of many smaller drying cylinders. The tissue machines usually run at between 1500 m/min and 2200 m/min, a fine paper machine at about 1000 m/min, a board machine at up to 500 m/min and a newsprint machine at about 2000 m/min. With a short machine and a high speed, the time from head box to reeled paper is only a few seconds.

Tissue production begins with a fibre suspension that is injected between a wire and a felt (or between two wires) and is transported by a felt to a rotating dryer cylinder called a Yankee dryer. The paper web is pressed onto the cylinder with a pressure roll and the web adheres to the surface. The paper is dried on the cylinder which is heated internally with steam and there are also dryer hoods on top of the paper web. The paper is removed by scraping off the web with a blade. The scraping produces a wrinkled, creped, paper. Figure 1 shows an overview of a conventional dry crepe machine.

2.1.1 Wet end

The headbox for tissue paper contains a pulp stock at a consistency of 0.15% – 0.25% solids. The pulp is distributed over the machine width by pipes which are
constructed so that there is a turbulent flow that breaks fibre flocs and gives a good formation. To produce a multilayer tissue paper, the headbox has parallel channels with intermediate separating vanes.

Considering the forming section, there are four types of tissue machine on the market (Kimari, 2000). The crescent former is the most commonly used and the headbox delivers the pulp between the felt and the wire. The sheet is formed between the wire and felt and dewatered through the wire. The felt is wrapped around the forming cylinder and transports the paper to the nip between the pressure roll and the Yankee cylinder. Another configuration is the C-Former; where the headbox delivers the pulp between two wires under the forming cylinder and the paper is thereafter transferred to a felt. Two other machines, less used today, are the Foudrinier machine and the suction breast roll machine.

In a TAD machine, the through-air drying cylinder is placed between the forming section and the Yankee dryer. The paper web is rotating on a perforated cylinder with air blown through the web, creating retaining imprints. The TAD process has a low pressure nip, and water is removed before the TAD cylinder by suction boxes to a dryness of about 25% (Kullander, 2012). The low pressure nip gives the paper a higher bulk than a paper made in a conventional machine. The paper has a high absorbency, bulk softness and surface smoothness. The structure moulded in the paper at the through air dryer gives extra bulk and absorptivity to the paper (Gavelin et al., 1999). A disadvantage of this process is the high energy consumption compared to that of other tissue machines.

### 2.1.2 Pressing and drying

The heart of the tissue machine is the Yankee cylinder, which dries the paper from 40 % dryness to 90 – 98 % dryness. The paper web is transferred onto the drying cylinder by one or two press rolls at a press nip between 2 and 4 MPa (Kullander, 2012), see Figure 2. The paper adheres to the cylinder and is dried at a surface temperature of about 100°C. On top of the Yankee cylinder, drying hoods blow hot air onto the paper web. When the paper is dry, after ¾ of a turn round the cylinder, it is scraped off by a creping blade (creping doctor). The paper is thereafter wound up on a reel with a lower speed than the Yankee cylinder to be able to maintain the crepe structure.
2.1.3 Converting

In most tissue machines, the paper is wound up on a reel before transportation to converting. Depending on the end-product, the converting section can include embossing, printing, perforation, winding and tail sealing, log sawing and packaging (Kimari, 2000). Embossing is very common in the converting line. It consists of pressing plies together and provides softness, bulk and absorbency to the paper (Woodward, 2007). To be able to separate sheets, perforation is very important. For rolled up products, winding and sealing the tail onto the rolls is needed. Finally, the product is cut into the desired width and is than packed.

2.2 Yankee cylinder

Heat is provided by steam inside the cylinder which enters the cylinder through the front journal (to the right in Figure 3). The steam is led through the internal shaft and through the nozzles located on the shaft. The steam pressure is about 1000 kPa (Gavelin et al., 1999). When the steam condenses on the walls of the inner surface of the cylinder, the condensate is picked up by small pipes dipping into grooves. It is collected in about six transversal headers and led through long
bent pipes to the internal shaft and exits the cylinder at the rear journal. The Yankee cylinder is made of cast iron and has a surface roughness ($R_a$) of 0.3 µm – 0.4 µm.

2.2.1 Coating spray application system

Coating chemicals are needed to control the adhesion between the paper web and the cylinder dryer. The coating also protects the surface of the cylinder from corrosion and reduces wear on the doctor blade. The chemicals are sprayed onto the cylinder through nozzles, as shown in Figure 4, attached to a boom and situated beneath the Yankee cylinder in the cross direction (CD) of the paper web. The boom oscillates to reduce streaks on the surface.

Figure 4. Simplified layout of a Yankee cylinder where chemicals are sprayed onto the cylinder dryer with oscillating nozzles.
2.2.2 Creping doctor and cleaning doctor

A Yankee cylinder for tissue production has space for three doctor blades, although many mills only use two of them. The doctor highest up on the cylinder is called the cutting doctor and is used when the creping doctor has to be adjusted i.e. when blades are replaced on the other two doctors. The paper scraped off by the cutting doctor is returned as broke. Under the cutting doctor is the creping doctor which scrapes off the dried paper from the Yankee cylinder. The construction of the creping doctor is extremely important for the crepe structure and the quality of the paper produced. The doctor blade is 1.0-1.25 mm thick and about 100 mm high (Gavelin et al., 1999). The blades are usually made of steel, but ceramic tips are becoming more common. Below the creping doctor, the cleaning doctor can be located. The cleaning doctor should, for example, remove thicker patches of coating to make the surface of the coating as even as possible. The angle of the creping blade is very important for the structure of the scraped off paper and if the service of the blade is delayed, the creping angle decreases and a more coarsely creped paper is produced.

2.3 Creping

2.3.1 Creping mechanism

When the creping doctor scrapes the tissue paper from the Yankee cylinder, the energy from the blade leads to a wrinkling of the paper and partially breaks the physical structure of the sheet. Microfolds are created and piled up on top of each other on the creping blade, see Figure 5. When the pile of microfolds is high enough, the pile falls into a macrofold. A new pile with microfolds is than started and the process continues.

Figure 5. Creping the paper (redrawn from Hollmark, 1972).
When the blade hits the sheet, a stress is created inside the sheet and in the coating layer between the paper sheet and drying cylinder. The part of the paper closest to the creping blade is sheared off the cylinder and this unbounded part is buckled, see Figure 6. The buckled part moves on the surface of the blade tip. When the bonded paper part hits the blade again, new stresses are built up in a continuing process (Ramasubramanian et al., 2011). Delamination and buckling of films have also been studied by Evans and Hutchinson (1984). In their study, it was shown that the thickness of the film affects the stress intensification developed at the perimeter of the delaminated area.

![Figure 6. Buckling of paper at the creping blade tip (redrawn from Ramasubramanian et al., 2011).](image)

The blade is held against the cylinder at a certain angle, Figure 7. The top surface of the blade can be ground to different angles. The angle between the top surface of the blade and the cylinder surface is called the creping angle or impact angle. This angle affects the distance between the crepes in the paper and therefore also the smoothness of the paper surface (Gavelin et al., 1999).

![Figure 7. The angle between the top surface of the blade and the cylinder surface is called the creping angle or impact angle and affects the structure of the tissue paper.](image)
The larger the creping angle is, the fewer micro-crepes are formed in every macro-crepe and the paper is more finely creped, as shown in Figures 8 and 9. The crepe wavelength therefore decreases with increasing creping angle (Ramasubramanian et al., 2011).

Figure 8. The creping process with a large creping angle. Only a few micro crepes pile up in each macro crepe. The large slope of the blade results in an easy release of the crepes from the blade.

Figure 9. The crepe process with a small creping angle. The number of micro-crepes is larger with a small creping angle because the blade can hold the micro-crepes piled up on the blade more easily.

The blade has to be changed about once every four hours when a regular steel blade is used. It is time to change the blade when the crepe structure becomes too coarse and uneven, and when the coating is building up on the cylinder.

After the creping process, the paper is wound up and the reel is transported for further treatment. The reel runs at a slower speed than the Yankee cylinder in order to maintain the wrinkled paper structure. If the winder and Yankee had the same speed, the crepes produced would be stretched too much. The crepe ratio can be calculated as (Ho et al., 2007):

\[
\text{Crepe ratio} = \frac{\text{Yankee speed} - \text{Reel speed}}{\text{Yankee speed}} \quad \text{Eq. 1}
\]
When the crepe ratio is increased the amplitude of the crepes increases and the softness decreases (Kuo and Cheng, 2000). When creping the paper from the Yankee cylinder, energy is needed not only to break the bonds between paper and Yankee coating, but also to buckle the paper and break internal fiber bonds. Figure 10 shows the result of previous adhesion studies with papers of different grammages (Boudreau, 2013).

![Figure 10](image.png)

Figure 10. Creping force at different grammages. The error bars represent the 95% confidence interval (Boudreau, 2013).

When the grammage of the samples is increased while maintaining the dryness at 30% or 50%, the force needed to scrape off the paper increased. The dryness of the paper had a slight impact on the creping, but the influence of the grammage on the creping force was significant. This was expected since creping relies on an initial buckling of the paper at the creping point. The thicker the paper, the higher is the local bending stiffness and the larger should be the contribution to the creping force. Ramasubramanian and Crewes (1998) also observed that higher grammage papers exhibited higher shear strength.

2.3.2 Adhesion between sheet and Yankee dryer

Adhesion is due to the attraction force between the paper sheet and the Yankee cylinder. There are many different theories regarding the adhesion mechanism, but the most common is the adsorption theory (Üner, 2002). To be able to adsorb onto the metal, there has to be close contact between the molecules in the paper and the metal surface. The most important forces are hydrogen bonding, covalent bonding and van der Waals forces (Üner, 2002). Some literature says that, in order
to achieve the greatest uniformity and the strongest adhesion, the sheet must be pressed tightly against the cylinder when the coating is as sticky as possible. The coating is stickiest at its glass transition point ($T_g$) in the case of cross-linking coatings and at the moisture content which gives maximum tack in the case of rewettable coatings. A rewettable coating is a coating for which the adhesive part stays on the drying cylinder and is activated by the moisture in the wet paper web adhering to the cylinder (Neal et al., 2001). The rewettability prevents the coating from building up (Hagiopol and Johnston, 2012) since such a coating is water-soluble and is more easily removed from the Yankee surface. If the sheet is pressed onto the coating before the coating reaches its $T_g$, the adhesion is weak when the pressure is released. The coating is too viscous and most of the coating is pressed through the sheet and into the felt by the pressure roll. If the sheet is pressed onto the coating too late, the adhesion becomes weak because the coating is too hard. If the moisture profile across the machine is not uniform, the coating will set at different times and this will prevent a uniform attachment of the paper web to the cylinder (Stitt, 2002).

*Figure 11* shows different zones of the cylinder where different stages in coating formation take place.

![Figure 11. Diagram showing the different zones of coating formation on the Yankee cylinder (Redrawn from Sloan, 1991 and Hättich, 1999).](image)

The surface energies of the adhesive and of the cast iron cylinder which are brought into contact with each other are of great importance for the adhesion. The roll pressure towards the Yankee cylinder affects the moisture content of the paper web and the adhesion (Kuo and Cheng, 2000). In order to establish molecular contact, an adhesive must, when in the liquid state, wet the surface to which an adhesive bond is to be achieved, and the wetting behaviour is controlled by the
surface free energies of the phases involved. Spontaneous wetting, or spreading of a liquid on a solid substrate, is favoured by a high surface energy of the solid and a low surface energy of the liquid (Kinloch, 1980).

If the web is pressed tightly onto the coating layer with good contact, bonds between fibres in the sheet break when the paper is scraped off the cylinder (Allen and Lock, 1997). Another feature of good contact is that more fibres are pulled up from the surface of the web when it is separated from the Yankee cylinder, and raised fibres give bulk to the sheet. A higher bulk makes the paper softer and more absorbent. A strong and even adhesion also facilitates heat transfer from the cylinder dryer. A higher drying rate of the paper makes it possible to run the machine faster and more energy-efficiently (Archer et al. (2001), Neal et al. (2001)).

If the adhesion is too high at the creping doctor, the blade may pick and drag fibres from the surface of the paper and create holes in the web or even cause the web to break. If the adhesion is even stronger, the paper web may simply pass beneath the blade. If the adhesion is too low, the paper separates from the cylinder before it reaches the creping doctor and this may lead to a long crepe wavelength (Oliver, 1980), or almost no creping at all, and a poor paper quality.
2.3.2.1 Moisture content in paper at the adhering point

The dryness of the paper when it adheres to the Yankee cylinder (before pressing) affects the force needed to scrape off the paper. Boudreau (2013) showed that the dryness of the paper seemed to have an impact on the adhesion and on the force needed to scrape off the paper, see Figure 12.

![Figure 12. Creping force at different moisture contents at the adhesion point of the paper. The error bars show the 95% confidence interval (Boudreau, 2013).](image)

The points can be divided into two groups. One group at a lower dryness level (30% - 36% dryness) with a high creping force and a second group at a higher dryness (39% - 48% dryness) with a lower creping force. The dryer the paper is when it adheres to the metal surface; the more cockling of the paper takes place due to variation in drying tension. The creping force decreases with increasing dryness according to Nordman and Ugglä (1978) and Fuxelius (1967). The 95% confidence intervals shown in Figure 12 is greater with a dryer paper and this could be an effect of poor contact between paper and coating and a larger variation in adhesion. When the paper is moist, the paper and fibers are flat and the contact area between paper and metal is fairly large. With increasing dryness, the paper becomes more uneven and does not adhere evenly to the metal surface. It was observed in this study that the dryer the paper when it is pressed against the metal, the harder it was to ensure that it adhered to the metal. One aspect is less rewetting...
of the coating layer, when it comes into contact with the wet paper, and therefore a less tacky coating to which the paper can adhere.

### 2.3.3 Crepe wavelength of tissue paper

*Figure 13* shows a photograph of an industrial tissue paper with a clear crepe wave structure.

![Figure 13](image)

Figure 13. A close-up of a conventional tissue paper. The paper has a crepe wavelength of about 250 µm.

The adhesion between the paper and the Yankee cylinder affects the force needed to scrape off the dry tissue. Ramasubramanian and Shmagin (2000) showed that an increase in adhesive concentration led to an increase in the creping force. The creping force is the force in the plane of the paper required for a blade to shear off and buckle the paper. These trials were performed on a laboratory scale with polyvinyl alcohol (PVA) as the adhesive.

A stronger adhesion between the paper and cylinder gives rise to a more finely creped paper with a shorter wavelength. Ramasubramanian and Shmagin (2000) also reported the effect of adhesive concentration on crepe wavelength on a laboratory scale. A higher concentration of adhesive, PVA, in the coating gave a shorter crepe wavelength and a more finely creped tissue paper.
2.4 Yankee cylinder coating

2.4.1 Natural coating

The cast-iron surface of the cylinder is most likely covered by a thin oxide layer with a high surface energy (Kinloch, 1980; Nordman and Uggl, 1978). The process water accompanying the fibre web completely wets the cylinder surface. After the machine has been in operation for some time, an evaporation residue is formed on the surface of the cylinder, consisting mainly of hemicellulose, fines and wet-end additives dissolved in the water. The adhesive properties originate from hemicellulose, which consists of low molecular weight sugars with branched or straight chains. The pulp and its pre-treatment affect the adhesion. For example, an increase in beating increases the adhesion. Beating releases hemicelluloses; but the increase in adhesion may be a result of increased fibrillation, which gives more flexible fibres and a greater bonding area (Oliver, 1980). Tissue was made like this in earlier days, when the most frequently used method to control adhesion was to vary the pH of the process water. The adhesion increased with increasing pH, due to the higher fibre charge at higher pH and hemicelluloses were precipitated.

2.4.2 Coating Spray

Today almost all tissue machines are equipped with a chemical spray system with which adhesives and release agents are sprayed onto the surface of the cylinder just before the paper web makes contact. It has been found, by trial and error, that both the amount of substance applied and the position of application are critical for good adhesion and creping, indicating that the tackiness of the sprayed coating layer is an important factor controlling the adhesion at the point of creping. The creping process works best when no wet-end chemicals interfere with the adhesive sprayed onto the dryer (Ampulski and Trokhan, 1993). A common problem in tissue manufacture is the temperature gradient in the paper web on top of the Yankee dryer in the cross direction (CD). The fibres at the edges of the machine, with no web of fibres in contact on the sides, have a higher temperature than the fibres in the middle of the dryer cylinder (Archer and Furman, 2006). If the temperature differs, the adhesion and the creped paper will differ in the cross direction of the paper web. Archer and Furman (2006) have invented a system to divide the cylinder into zones where different coating formulae can be used to overcome the temperature difference.
2.4.2.1 Adhesives

The most commonly used adhesive substance is a polymer in an aqueous solution. The polymers most frequently used are the synthetic polymers: polyaminoamides, polyamines, polyvinyl alcohols, polyvinyl acetates and polyethers (Grigoriev et al., 2005). Polyamide resins cross-linked with epichlorohydrin (PAE) are the most popular. Many adhesives have the same structure as PAE wet-strength resins, but with significantly less cross-linking agent (Archer et al., 2001). The reaction to produce the PAE resin is shown in Figure 14. Polyamidoamine is reacted with epichlorohydrin to form reactive intermediates. An aminochlorohydrin is first formed, and this can continue to react to azetidinium salt (Braga et al., 2009).

![Figure 14. Polyamidoamine activated by epichlorodryn to form reactive intermediate stages (redrawn from Braga et al., 2009).](image)

Adhesives have different hardness levels due to a difference in their glass transition temperatures ($T_g$), which is most commonly between 30°C and 105°C. The properties of the adhesive, like its rewettability, the ease with which the blade cuts into the coating layer, the strength of adhesion etc., are controlled by the cross-linking (Hagiopol and Johnston, 2012). The level of cross-linking affects the $T_g$. Increasing the cross-linking level increases $T_g$, and the coating becomes harder and more brittle (Luu et al., 2004). Figure 15 shows a cross-linking reaction between two aminochlorohydrin chains.
The cross-linking reaction for the PAE adhesives (thermosetting adhesives) is dependent on the pH. No reaction takes place at low pH and the pH is therefore held low until use. The resins cross-link on the Yankee surface and create a plastic coating layer. Üner et al. (2006) studied acid-base interactions between PVA and a metal surface, and they have shown that the adhesion force decreased when the amount of hydroxyl groups was reduced. Figure 16 shows the adhesive crosslinking to fibers at the drying section (Braga et al., 2009).

The build-up of the polymer on the surface of the Yankee cylinder is affected by the solubility, molecular weight and chemistry of the polymer sprayed onto the cylinder. If the polymer is easily dissolved in water, it can be dissolved by the humidity of the sheet and build up unevenly on the cylinder surface. A more uniform coating can be achieved if a hydrophobic polymer is used.

The dryer coating can contain more than one adhesive chemical, and these are used in different ratios depending on the tissue grade being produced. In the TAD process, the paper is drier when it reaches the Yankee dryer and structured from the pre-drying. A smaller contact area on the Yankee cylinder and a drier paper require additional creping adhesives (Pomplun and Grube, 1984). Researchers have tried to invent different levels and types of adhesives to meet the industrial demand.
for adhesives optimizing a specific papermaking line (Cambell, 2002). For example, a mixture of PVA and a water-soluble, thermosetting, cationic PAE resin (Soerens, 1985) or a mixture of a water-soluble cationic starch, optional PVA and water-soluble thermosetting cationic PEA resin (Vinson et al., 1999). Cambell (2002) have solved the problem with a water-dispersable thermally cross-linked PAE resin with one or more multivalent metal ions.

2.4.2.2 Release agent

Release oils are used to reduce the adhesion and facilitate the release of the sheet from the dryer at the creping blade. The adhesive and release oil are mixed together and diluted with water before the mixture is sprayed onto the hot Yankee cylinder. The water evaporates and the mixture of release oil and adhesive forms a tacky coating on the cylinder (Grigoriev et al., 2005).

Release agents are mostly hydrophobic and can be emulsifiable mineral oils, fatty acid esters, polyphosphates, imidazolines or fatty alcohol ethoxylates. The agent blocks cross-linking sites and therefore prevents a network from being formed (Hättich, 1999).

Release oils soften the coating layer and delay the setting of the coating, and this means that the release oil has a great influence on the uniformity of attachment of the paper web to the Yankee cylinder (Stitt, 2002).

A common belief of suppliers of chemicals is that release oil migrates from the Yankee cylinder surface to the surface of the coating facing the air (Hättich, 1999). There is thus a concentration gradient of release oil within the coating layer. The coating layer closest to the cylinder surface is very hard and consists of a completely cured adhesive. The middle layer has emulsified oil that softens the adhesive and the layer on the outside is mainly oil that lubricates the blade.

2.5 Fibre furnish

Both virgin fibres and recycled fibres are used in tissue production. The most common virgin pulp is kraft pulp (Kimari, 2000), but sulphite and chemithermomechanical (CTMP) pulps are also used. The latter is mostly used to give the paper more bulk. The fibre type used depends on the end product. Chemical tissue pulp is based on softwood, mainly pine, and hardwood from birch, eucalyptus or beech (Kimari, 2000). Facial and toilet products contain more
hardwood pulp to gain softness than paper towels which contain more softwood fibres to give greater strength. Spring wood has more slender and thin-walled fibres which mean that less beating is required to get flexible fibres.

Beating of the pulp fibrillates the fiber surface and the outer layer of the fibers may be damaged (Norman, 1992). The surface area increases and more sites for bonds between fibers are created. Figure 17 shows SEM images of a spruce pulp where the fibers are (a) unbeaten and (b) beaten to 2000 revolutions in a PFI mill. Beating fibers increases the bond strength not only through a larger surface area from fibrillated fibers but also through more flexible fibers. The amount of fines increases with increasing beating, but levels out to a constant level (Laivins and Scallan, 1996).

Figure 17. SEM micrographs of (a) Unbeaten fibers and (b) beaten fibers (reprint from Kullander et al., 2012).

2.6 Tissue paper properties

Tissue paper is a light weight-paper consisting of one or more plies. Most tissue products such as bathroom tissue, kitchen towels, facials, napkins, wipes and fluff pulp in baby diapers are made from creped paper. Depending on the end-product different parameters are important to different degrees. Examples of such properties are wet and dry strength, softness, absorbency etc. Increasing softness for example, often means lowering the paper strength (Ampulski and Spendel, 1991). Nowadays, many auxiliary chemicals make it possible to combine properties to various degrees. Many tissue grades consist of two or more plies. If a very soft and strong tissue is desired, one side can be made of short fibre pulp to give softness and the other side with long fibre pulp to give strength. There is a natural two-sidedness of the paper. The side on the Yankee cylinder is softer with small craters and the felt side has peaks and feels rougher. When the paper webs are
converted and two webs are pot together to a paper, it is best to put the rougher stronger side in the middle and keep the smoother sides on the outside.

### 2.6.1 Paper strength

When the paper web is scraped from the drying cylinder, the paper is sheared off and compressed, forcing the bonds to be weakened and the fibres inside the paper to buckle, become distorted or even break. The fibres and fibre bonds are important for the tensile strength and for the creping process, as they affect the structure of the creped paper. Stiff and long fibres and many fibre bonds in the fibre network lead to a stiffer paper and the paper will be more coarsely creped.

#### 2.6.1.1 Tensile strength

The tensile strength is the maximum force needed to break a paper under tension. Beating the pulp increases the paper strength (Gigac and Fišerová, 2008) by increasing bonding sites and making the fibers more flexible. Fines have a larger surface area than fibres and they can swell twice as much (Laivins and Scallan, 1996). They also increase the fibre-fibre interaction in the pulp slurry. Having a large surface area and being rich in hemicellulose (Hrún and De Ruvo, 1978), fines increase the fibre-fibre bond strength. Seth (2003) has shown that both tensile strength and Scott bond strength increase with increasing content of fines.

The strength of the final paper is reduced by the creping process as inter-fibre bonds are broken (Stitt, 2002, Grossmann, 1977). Kuo and Cheng (2000) showed that when the pressure of the press roll was increased, the tensile strength of the paper decreased. In their study, the dryness was high (higher than 55% before the pressure nip) and fibre bonds were probably destroyed in the nip.

#### 2.6.1.2 Wet strength

The main task for most tissue products is to soak up a liquid and retain it inside the paper. Depending on the product, the paper needs to have a sufficient wet strength to avoid falling apart within a certain time. A bathroom tissue cannot have a high wet strength (needs to be able to be flushed through drains) whereas kitchen towels need to have a much higher wet strength. When the paper is in contact with water, the hydrogen bonds between fibres easily break and the paper falls apart. To protect these hydrogen bonds, a wet strength resin can be used. Polyamidoamine
epichlorohydrin (Braga et al., 2009) is commonly used as a wet strength resin and it forms a network around the fibres (Hägkvist et al., 1998).

2.6.2 Softness of tissue

The softness of tissue paper is a combination of bulk softness from crumbling of the paper and surface smoothness. Both are important for the feeling of softness for the end-users. The creping of paper softens the paper (Grossmann, 1977) both through making the paper bulksoft when interfiber bonds are broken and because the surface smoothness increases. Liu and Hsieh (2004) show in their study, that softness increases with decreasing tensile index. The balance between softness and paper strength is delicate and can be partly solved by adding chemicals to the pulp or on top of the paper. Kuo and Cheng (2000) have investigated different parameters affecting the surface smoothness of the creped paper. In their study, the surface smoothness was measured with a sled method using the frictional force to resemble a fingertip running on top of the paper. When creped paper is produced, the angle between the Yankee cylinder and the blade becomes larger, the paper is more finely creped and the surface smoothness increases. Increasing the pressure between the press roll and the drying cylinder to a moderate level, increases the paper adhesion and surface smoothness.

Bulk softness, i.e. how easily a paper can be crumbled, is affected by beating of the pulp. Whether the softness is affected positively or negatively by beating depends on the fibre furnish (Gigac and Fišerová, 2008).

2.6.3 Absorption

The main task of tissue paper is to soak up liquid and to retain the liquid in the paper. Water is absorbed in the fiber walls (Bristow, 1971 and Lyne, 2002) by capillary uptake and also by capillary forces in the hollow spaces between the fibers (Schuchardt and Berg, 1991). The amount of water absorbed depends on the paper structure created during the creping operation and on the types of fibers and chemicals used in the process. The capacity is measured in g water/g paper, and the rate of absorption in seconds per centimeter (s/cm), are the key factors (Kimari, 2000). Keeping the structure porous and avoiding collapse enhance the absorption. Therefore a high wet strength and stiffer fibers like thermo mechanical pulp can help to maintain the paper structure. Bleached hydrophilic chemical pulp in an interior or outer layer and stiff fibers from mechanical pulp in the other layers would be beneficial.
The water retention value (WRV) increases with increasing beating, but the total absorption in the final tissue decreases, due to the lower bulk of the final tissue. Beating fibrillates fibers and allows swelling by hydration (Carrasco et al. 1996). Beating also decreases porosity (González et al., 2012) and slows down the rate of absorption. Gigac and Fisěrová (2008) showed how beating decreases the paper absorption depending on the pulp type. Fibre swelling, measured as WRV, is affected by the pH, and a high pH in the furnish also increases the adhesion (Gavelin et al., 1999). The degree of swelling of the pulps when the pH is changing depends on the amount of acidic group and the cell wall flexibility (Lindström and Carlsson, 1982). Increasing the amount of acidic groups in the fibers also increases the WRV.

2.7 Measurement methods

Tissue making is a complex process with many parameters that affect the end product. To be able to measure parameters on-line and to have a good control over the process is desirable. Nowadays, it is already common to measure for example grammage and moisture content on the paper web.

Habeger and Baum (1987) have developed an off-line method to measure fibre orientation, to be applied on-line. The method uses a microwave signal which passes through a paper sample. The dielectric constant of the paper depends on the orientation of the electric field and it is largest for fibres parallel to MD (Habeger and Baum, 1987). A disadvantage of the method is its sensitivity to the moisture content of the paper web.

On-line measurements of surface smoothness have been made with optical methods (Brewster, 1993). In the first method, a light beam is directed perpendicular to the paper surface and two detectors collect the light reflected at a low angle from the paper surface. The detectors are placed at equal distances from the light source and at specific angles for the reflecting light. A smooth sheet scatters the light equally to the two sensors whereas a rough paper leads to a difference in light intensity between the two detectors. A slightly different method mentioned by the same author uses an array of detectors and a low angle light beam. A smooth paper activates a small number of sensors in the array and a coarse paper activates a larger number of detectors. Chase and Goss (1997) developed a method to characterise the surface of a moving paper web. They used a laser light with focusing lenses to illuminate a small region of the paper. The light scattered from this region was collected by a photosensitive detector and the image of the region gave a signal representing variations in the height of the illuminated
region. Raunio et al. (2012) have made laboratory measurements on crepe frequency based on image analysis. Through which the crepe frequency was calculated. Images were made off-line with a digital camera. The crepe frequency was computed from the 2D Welsch Spectrum showing the frequency distribution of the image. Nalco Company has developed an off-line method to measure the crepe wavelength. Their NCAT equipment is also built on image analysis to calculate the wavelength of the crepes (Bonday, 2010).

Nuyan et al. (2007) have developed a method to measure the paper thickness on-line. The method combines magnetic reluctance as a distance measurement to metal and a non-contact optical laser reading the distance to the paper surface. The difference between the two distances gives the paper thickness. A similar study has been made by D’Emilia (1999) for the measurements of coating thickness for paper packages used in the food industry. A non-contact method was used with eddy-current to measure the distance to the steel surface on which the coating was sprayed. The surface of the coating was at the same time measured by an optical fibre.

The wavelength methods described above are used off-line or on a laboratory scale. No known methods to measure the paper surface properties reliably on-line in the tissue machine have been reported. A method that is fast enough to measure the wavelength at the speed of tissue production is required. Nor is there any known method for measuring the coating thickness on-line on the Yankee cylinder. The following ideas have been tested in the present work in a laboratory environment to provide the basis for the development of on-line measurement methods in the future.

### 2.7.1 Fluorescence

Studies have been made by Archer and Furman (2006) using fluorescence in coating chemicals to measure coating thickness. The idea was to add a known amount of fluorescent tracer to the coating colour and to use a fluorometer to measure the signal on the cylinder and the produced paper and relate the result to coating thickness.

Luminescence is the emission of light from electronically excited species, and fluorescence is a particular case, see Figure 18 (Valeur, 2002, Lakowicz, 2006). When a molecule absorbs a photon, the molecule is brought to an electronically excited state. It returns to the ground state with the emission of a photon. The lifetime of the excited state for fluorescence is $10^{-10}$-10$^{-7}$ s (Valeur, 2002).
Figure 18. Jablonski diagram, showing various processes by which the molecule to return to the ground state (Valeur, 2002).

If a substance showing fluorescence is introduced into the coating colour, the intensity of fluorescence can be used as a measure of the amount of coating chemicals present on the cylinder surface at any given moment.
3 Experimental

3.1 Materials

3.1.1 Coating chemicals

3.1.1.1 Adhesive and release agent

The chemicals used in the trial on the pilot machine were an adhesive called Ekasoft B15 and a release agent called Ekasoft R95 from Eka Chemicals, Sweden. The former is a standard cationic PAAE (polyamidoamine-epichlorohydrin) adhesive and the latter is an alcohol ethoxylate mineral-oil-based release agent.

3.1.1.2 Optical brightener

The optical brightener used was Blankophor Flüssig 01 from Kemira. The brightener absorbs radiation between 330 nm and 370 nm and fluoresces at 420-470 nm.

3.1.2 Creping equipment

A new creping method for laboratory purposes was needed to be able to perform adhesion studies. Adhesion tests are commonly performed with peeling equipment, but a peeling method is not optimal for assessing the adhesion forces acting on the Yankee cylinder. The separation between paper and metal takes place more in the paper, than if the paper is scraped off with a blade. When the paper is scraped off, the separation usually occurs in the coating layer, as in the industrial production. The creping equipment described in this work used a scraping technique resembling the industrial process. The equipment consisted of a wagon with a weight on top which applied a line load of 3 kN/m upon the metal strips (the same line load was used in pilot trials in Paper I). The metal strips, on which paper adhesion occurs, had an average surface roughness (Rz) of 0.3 µm – 0.4 µm and a width of 20 mm. The scraping tests were performed in a temperature- and humidity-controlled room and, to prevent corrosion, acid-proof steel was chosen for the metal strips, although the surface of a Yankee cylinder is usually made of
cast iron. The stainless steel used in this work was less reactive and probably gave a slightly lower adhesion than that on an industrial Yankee cylinder.

The wagon was pulled forward by a material tensile tester (Zwick/Roell Z005, Zwick Roell AG, Germany) at a speed of 2 m/min. The speed was low compared to that used in an industrial process and was limited by the tensile tester. The different trials can however still be compared to each other.

![Image](image.png)

**Figure 19.** Left hand side: the creping wagon attached to the tensile tester. Right hand side: creping wagon with a paper adhered to the metal strip.

*Figure 19* shows on the left hand side the blade scraping the metal strip in the absence of any paper sample applied and *Figure 19* shows on the right hand side a sketch of the equipment with a paper adhered onto the metal strip. The blade is mounted under the wagon and has a fixed blade angle of 26°. The creping angle and bevel angle could be adjusted by changing blades with different bevel angles. A Yankee cylinder has a large diameter of about 5 m and the tangential surface can therefore be assumed to be flat. The total angle is \( \alpha + (90 - \beta) + \gamma = 180^\circ \), see *Figure 20*. 
3.1.3 Pulp

In the trials, four different pulps were used:

1. The main pulp used was Södra Black 85Z, a bleached softwood kraft pulp, supplied by Södra Cell, Sweden. The pulp was totally chlorine free (TCF) bleached with the steps: Q-OP-Q-PO (Q= EDTA or DTPA, O=oxygen and P=hydrogen peroxide). The pulp was produced from roundwood chips from a mixture of 70% spruce and 30% pine. More information about the pulps is given in Table 1.

2. Södra Green softwood kraft was also TCF bleached, including the steps: Q-OP-(Q-Paa)-PO (Paa – peracetic acid), and contained 70% spruce and 30% pine. This pulp consisted mainly of sawmill chips from south eastern Sweden.

3. Eucalyptus Eurograndis pulp from Veracel.

4. Södra Gold Eucalyptus, which is an elemental chlorine free (ECF) bleached kraft pulp of Eucalyptus Grandis 75% and Globulus 25%.
Table 1. Furnish data for the four pulp types from Södra.

<table>
<thead>
<tr>
<th>Furnish</th>
<th>Fiber length before beating (mm)</th>
<th>Fiber width (µm)</th>
<th>Fines (%)</th>
<th>Brightness ISO (%)</th>
<th>Kappa number after cooking</th>
<th>Kappa number after O₂ (mg/g dry pulp)</th>
<th>Hemicellulose (mg/g dry pulp)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SW roundwood</td>
<td>14.5</td>
<td>2.2</td>
<td>6.0</td>
<td>86</td>
<td>29</td>
<td>12</td>
<td>169</td>
</tr>
<tr>
<td>SW sawmill chips</td>
<td>14.1</td>
<td>2.63</td>
<td>6.0</td>
<td>86</td>
<td>28</td>
<td>9.1</td>
<td>169</td>
</tr>
<tr>
<td>Euca. Eurograndis</td>
<td>20.1</td>
<td>0.78</td>
<td>4.3</td>
<td>90</td>
<td>-</td>
<td>-</td>
<td>-164</td>
</tr>
<tr>
<td>Euca. Grandis(75)/Globulus(25)</td>
<td>29.3</td>
<td>0.75</td>
<td>10.0</td>
<td>90</td>
<td>16</td>
<td>8.0-9.0</td>
<td>204</td>
</tr>
</tbody>
</table>

For the trials at different pH levels, the pulp produced from softwood roundwood chips was used. 0.1M sulphuric acid was used to lower the pH and 0.1M sodium hydroxide was used to increase the pH.

3.2 Methods

3.2.1 Yankee cylinder coating

3.2.1.1 System for making replicas

The RepliSet-Gt1 system from Struers was used to make replicas of an industrial Yankee cylinder surface. Based on a fast curing (4 min at 25°C), a two-component silicone rubber with good releaseability was applied to the surface with a dispensing gun. The cartridges contain both polymer and curing agent. These were mixed in a static mixing nozzle during application onto a release paper. The release paper was immediately pressed against the Yankee cylinder for a few minutes to allow the silicone to cure. The replicas were then evaluated in a Scanning Electron Microscope (SEM). The method is described in detail in Paper 1.

Images of the replicas were taken using a Hitachi low vacuum SEM S3000N microscope in a high vacuum position. The replica from the commercially operated tissue machine was coated with gold as a different SEM instrument was used.

3.2.1.2 Sampling of the coating

The tissue machine used in the trial was a pilot dry crepe tissue machine in a C-Former configuration at Metso Paper in Karlstad, Sweden. The Yankee cylinder was heated up the day before the trial and the old coating layer was removed with sandpaper. The machine was run at a speed of 1000 m/min and the machine settings were those intended for toilet tissue with a grammage of 18 g/m². The
pulp used was a softwood kraft pulp (Södra Black 85Z) refined in a conical refiner, Conflo JC-01 Valmet with MX filling (20 kW/ton). The total flow of the diluted coating chemicals was about 4.8 l/min and had a dry content of 0.4%.

Three samples were collected at each trial point: a paper sample, a coating sample at the cleaning doctor and a coating sample between the cleaning doctor and the spray boom, see Figure 21. The coating scrapings were taken from different layers on the cylinder while the machine was in operation.

![Figure 21. The Yankee cylinder with the sampling positions.](image)

The samples were taken at five different dosages of spray chemicals. The ratios of adhesive to release chemicals were: 1:5, 2:4, 3:3, 4:2 and 5:1. The tissue machine was running continuously, and paper and coating samples were collected 30 minutes after each coating composition was changed.

### 3.2.1.3 Chemical analysis

The carbohydrate and nitrogen contents were determined on the coating samples taken from the pilot tissue machine and on the paper. The samples were sent to three different external laboratories for analysis.

The carbohydrate analyses were performed using ion chromatography or gas chromatography on samples that were hydrolysed in sulphuric acid. To determine the carbohydrate content, Laboratory 1 used the Tappi standard T249, which involves gas/liquid chromatography using alditol formation, Laboratory 2 used HPAEC-PAD (High Performance Anion-Exchange Chromatography with Pulsed Amperometric Detection) after hydrolysis in sulphuric acid, and Laboratory 3 also used ion chromatography with pulsed amperometric detection.
The nitrogen content of the tissue samples was determined by chemiluminescence using a COSA TN110 nitrogen analyzer. The nitrogen content was, however, much higher in the coating scrapings and, in order to improve the accuracy, the Dumas method was used on these samples and a Thermo Flash EA1112 combustion analyzer was employed.

3.2.2 Measurements of coating thickness

Initially a method similar to that described by Nuyan et al. (2007) was used to measure the coating thickness but due to the difficulty in making magnetic measurements at exactly the same time as the surface of the coating layer was measured, this method was rejected. The coating layer is very thin compared to the paper and a slight difference in sampling point could strongly affect the result.

For the coating thickness measurement, equipment developed in 1997 by Granlöf was used. The equipment was updated but was based on the same theory.

The cylinder used to represent the Yankee cylinder was a drying cylinder on a laboratory scale with a diameter of 80 cm and a width of 70 cm. The cylinder was heated to 100°C - 102°C and rotated at a speed of 0.18 m/s. The coating was sprayed with an air-driven commercial spray nozzle for spraying paint. The optical brightener was used at three different concentrations, 0.1 g, 0.5 g and 1.0 g mixed in 500 g distilled water with 13.25 g of the adhesive Ekasoft B15. The dry brightener content was thus 1.7 %, 9.96 % and 14.8 % of the dry coating chemicals sprayed onto the cylinder. The chemicals were mixed just before the trials and were sprayed onto the cylinder in two, four, six and eight layers, each layer consisting of approximately 7.1 g coating/m².

The equipment for measuring the fluorescence of the coating on the dryer consisted of an UV-LED (boxed into the measuring head, see Figure 22) and electronics to emit light pulses, a measuring amplifier and software to log the signal. The diode emitted ultraviolet radiation at a wavelength of 370 nm.
The light passed through the window and illuminated the coating on the cylinder. The particles in the brightener emitted light that passed not only through the window but also through another window made of polycarbonate, which absorbs UV-radiation, so that only the visible light generated by the fluorescence was collected and transported via the optical cable. (The box can be pressurized with industrial air to prevent moisture and dust from covering the lens.) The signal was led to the amplifier, and measurements were made alternately on illuminated/non-illuminated objects and the difference between them was recorded, in order to eliminate background light and noise. The outgoing signal was low-pass filtered to obtain a limiting frequency of about 1 Hz and led to a computer. The log programme used was a Labview-application that logged the signal to a log file on the computer. The signal was measured at a frequency of 1 kHz.

3.2.3 Laboratory evaluation of creping and adhesion forces

3.2.3.1 Papermaking

The dry pulp was soaked in water over night and defibrillated to 50 000 revolutions. The pulps were beaten in a PFI mill (Hamjern A/S, Hamar) according to ISO 5264-2. The pulp was diluted with de-ionized water and the stock concentration was determined according to ISO 4119 except that four 100 g samples were used instead of one 500 g sample. Water was added up to a concentration of 0.2% dry pulp. The SR value (PTI Austria) was measured according to ISO 5267-1. The papers were made in a Formette Dynamic Sheet Former (DSF) at a target grammage of 40 g/m² (it varied between 39 g/m² and 56 g/m²) and machine direction/cross direction (MD/CD) anisotropy of 2.2. This
grammage was chosen to obtain a paper of approximately 30% dryness (in the moisture tests it varied between 24% and 37% dryness) during the application onto the metal strips. At a more suitable grammage for tissue paper, around 20 g/m², the paper was found to be too dry and hard to handle when applied onto the metal. The paper was cut into machine direction strips with approximate dimensions of 40 mm * 200 mm. The paper strips were sealed in plastic bags and stored overnight. Papers dried under restraint were produced at each sample point so that the grammage could be measured and tensile tests could be performed in MD and CD.

3.2.3.2 Adhering of paper to metal

The metal strips were placed on a heating plate to reach a temperature of 100-105°C. In total, 0.54 g/m² of dry coating was sprayed onto the metal strips. The last coating layer sprayed had a concentration of 0.38% dry adhesive (the same concentration as used in the previous pilot trial). The moist papers stored in the plastic bags were placed on the metal strips. A thick polished metal sheet was placed on the paper and a 10 kg roll weight was moved back and forth once on the sheet to press the paper under hot conditions. The metal strip, paper and polished metal sheet were then transferred to a bench and a 2.6 kg weight was placed on top of the pile for one minute while the samples cooled. The weight was used to prevent curling and cockling of the paper during the cooling of the metal strips. The weight and the polished sheet were then carefully removed and the metal strip, complete with adhered paper, was left to dry at room temperature for at least three hours. The samples on the metal strips were placed in the climate room, at 23°C and 50% humidity, for at least 24 hours before creping, for the samples to acclimatize to the temperature and humidity.

3.2.3.3 Creping

The wagon was placed on the table between guiding bars. The wagon was attached to the tensile tester with a stiff cord rigged through a pulley, the wagon was pulled forward at a speed of 2 m/min, causing the blade underneath to scrape off the adhered paper. The force needed to scrape off the paper was measured and logged to a computer, and the distance travelled by the tensile tester’s head was measured. After the paper was scraped off, the test was repeated on the same metal strip without paper. This allowed the friction between the blade and the strip to be assessed, and the result was subtracted from the result from the first test in order to calculate the adhesion force.
3.2.4 **Paper characterisation**

3.2.4.1 **Softness**

The surface softness of the tissue paper produced in the pilot trial was determined using the surface tester developed by Hollmark (1976, 1983).

3.2.4.2 **Paper characterisation**

The structure of the paper was also studied with the OptiTopo method (Hansson and Johansson, 1999). This is a photometric stereo method for determining the topography of a paper surface where the paper is illuminated from both the left-hand and the right-hand side. Two images were captured with a CCD (Charged Coupled Device) camera. The spatial intensity of the reflected light was used to calculate a partial derivative which was integrated to give the surface height. The mean standard deviation of the surface height at different wavelength intervals was then calculated. The surface height differences were indicative of the crepe structure.

3.2.4.3 **Crepe wavelength measurements**

The creped tissue paper consists of micro crepes folded into macro crepes. The macro crepe forms a sinusoidal wave pattern. This wavelength of the sinus form is important for the paper quality and the ambition was to measure this on-line, but the OptiTopo instrument is too slow for use on-line in a tissue machine. *Figure 23* shows the typical appearance of the structure of the tissue paper.

![Figure 23. The crepe structure of the tissue paper showing macro and micro crepes. The length of the macro crepes is here 100 µm.](image)

A modification of Brewster’s method (1993) was used. The paper from the trials described in Paper I was used for the initial measurements and commercial papers were used in the main trials to validate the method. The paper was illuminated with a light beam from the optical fibre onto the paper surface. The paper was pulled under the sensor head with the help of two metal bars under the paper which were moved with an engine translator at a maximum speed of 0.5 mm/s, which is much
slower than a commercial tissue machine which runs at about 30 m/s. The light source used was a broad band Light-Emitting Diode (LED) with a central wavelength of 1300 nm in the initial trials and 980 nm in the main trials, and a bandwidth of 40 nm. Measurements were made with and without lenses and also with collimated light. The intensity of the signal should vary with the paper structure. The diameter of the beam was smaller than the expected crepe wavelength of the paper. *Figure 24* shows the sensor equipment with microscope lenses.

![Figure 24](image.png)

*Figure 24. Photograph of the sensor head with the tissue paper moving in the directions of the arrow.*

The light was directed onto the paper at angles of 0°, 10° and 45° to the paper surface and the light reflected at the same angles from the paper web was collected, *Figure 25*. At 0°, the sensor was placed directly at the lens instead of collecting the light through a second optical fibre.

![Figure 25](image.png)

*Figure 25. Set-up for the angular measurements of the surface structure. The paper was attached to a metal plate by magnets. The plate can be moved in a horizontal direction while the measurements are made by the measuring device to the right.*
To verify the method, measurements were made on four commercial papers with different surface structures. The multiple signal classification (MUSIC) method was used to estimate the wavelength for each paper. More details of the method are given by Schmidt and Bienvenu (Bienvenu, 1979, Schmidt, 1979, Schmidt, 1986).

3.2.4.3 Tensile strength

Tensile strength was measured in a Zwick Roell (Z005) materials tester according to the ISO 1924-3 standard.

4 Results and discussion

4.1 Yankee cylinder coating

In this study, the remaining coating layer taken from a pilot Yankee cylinder was analysed chemically and morphologically. The ratio of adhesive to release agent was varied as shown in Table 2.

<table>
<thead>
<tr>
<th>Trial</th>
<th>Adhesive</th>
<th>Release agent</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3</td>
<td>3</td>
<td>6</td>
</tr>
<tr>
<td>2</td>
<td>4</td>
<td>2</td>
<td>6</td>
</tr>
<tr>
<td>3</td>
<td>5</td>
<td>1</td>
<td>6</td>
</tr>
<tr>
<td>4</td>
<td>2</td>
<td>4</td>
<td>6</td>
</tr>
<tr>
<td>5</td>
<td>1</td>
<td>5</td>
<td>6</td>
</tr>
<tr>
<td>1:2</td>
<td>3</td>
<td>3</td>
<td>6</td>
</tr>
</tbody>
</table>

The carbohydrate content (originating from fibres) and nitrogen content (from the adhesive) were determined. The adhesive was the only component containing nitrogen, therefore nitrogen was used as an indicator of adhesive content. Replicas of the surface were made when the machine was stationary after using 5 and 3 parts of adhesive in the coating. The replicas were thereafter studied by SEM.

Figure 26 shows the contents of cellulose, hemicellulose and other substances. The carbohydrate content was calculated from the monomers according to Meier (1958). The total carbohydrate content was greater in the paper than in the coating samples. The outer coating layers contained almost as much carbohydrates as the paper. The inner layer had less, but still a fair amount of carbohydrates. This cured
coating layer contained mainly residuals from the pulp and very little of the added coating chemicals.

The nitrogen content, i.e. the amount of adhesive, was higher in the samples taken after the cleaning doctor (the inner layer of the coating) than in the samples collected at the cleaning doctor (the outer layer of the coating) see Figure 27. More adhesive seemed therefore to be present close to the Yankee surface, in agreement with the literature (Hättich, 1999), and only a small amount of adhesive was transferred to the paper. It is desired to cut off the paper in the coating layer, and a small amount of adhesive on the paper is therefore beneficial.
Figure 27. Nitrogen content as function of amount of adhesive applied to the cylinder. A total of 6 parts of adhesive and release together where applied in each trial point.

More adhesive was presumably present in the inner coating layer because of the tendency, encouraged by heat, for the adhesive material to cross-link which results in a cured inner layer, depending on the temperature and moisture gradient in the coating layer. When the wet paper web comes into contact with the Yankee surface, the outer layer of the coating is cooled and dampened by the paper. Since the doctor blade acts only on the outer layer of the coating, the adhesive should be more cured in the inner layer.

The Yankee coating was patchy with uneven coating layers. After the outer layers had been scraped off by the doctor blade, the residual coating contained blade streaks and also fibre fragments, Figure 28. The SEM images of the cylinder surface showed that the surface was very uneven.
4.2 Coating thickness measurements

In this project, a method of determining the coating thickness based on fluorescence measurements was tested on a laboratory scale. An optical brightener was mixed in the coating sprayed onto a metal cylinder, and an UV-LED was used to provide ultra violet radiation and to collect the light emitted due to fluorescence. A thicker coating layer should generate a stronger signal.

In a pre-study, the equipment was tested when the cylinder was stationary. The measurements were made on one fluorescent paper (unknown optical brightener) and one non-fluorescent paper. The dryer cylinder used had a more glossy surface than a normal Yankee dryer so, in order to determine whether or not this affects the measuring device, tests were also made using a mirror and a piece of black fabric. Figure 29 shows the average values obtained for the different surfaces, with error bars showing the 95% confidence interval. The values for the standard and fluorescent papers differed greatly, suggesting that the equipment was able to detect fluorescence. The difference between the mirror and black fabric was very small and the shiny cylinder is therefore assumed not to affect the results.
In a second pre-study, coatings containing different amounts of brightener were sprayed onto papers in thicker layers than used in the actual trials, again with the cylinder stationary. The papers all showed visible fluorescence when placed under a UV lamp. Figure 30 shows the results obtained in a test using standard paper with no coating, brightener, brightener plus adhesive and finally a layer of brightener.
The equipment was affected by fluorescence, *Figure 30*, but when the cylinder started to move, no reflected light was measured, *Figure 31*. No significant differences in signal strength could be seen for different amounts of brightener and coating layers. The cylinder was smoother than an actual Yankee cylinder and it was possible that the adhesive with optical brightener had not adhered to the cylinder and that almost no coating was left on the cylinder to emit fluorescence. The amount of optical brightener being used in the industry also has to be investigated, due to product safety.

![Figure 31. The amount of light emitted due to fluorescence (voltage) for different numbers of coating layers with error bars showing 95% confidence intervals.](image)

The averages and confidence intervals were calculated after outliers has been removed using a method that employs the quartiles and the differences between the quartiles (Scheaffer and McClave, 1995). The 95% confidence intervals overlap, and there is no significant difference between the points.
4.3 Laboratory creping equipment and adhesion

4.3.1 Creping equipment

This creping method gives the force needed to scrape a paper strip from a metal surface. A tensile tester measures the displacement of the wagon and blade while the force required to scrape off the paper was logged to a computer. By using this method different parameters can be varied, such as creping angle, different coatings and pulp quality etc.

Each adhesion test resulted in a curve similar to that shown in Figure 32. The solid curve represents the test with the adhered paper and the dashed curve is the blank test with only metal. Initially the two curves coincide. This section refers simply to the metal strip without any applied paper. For an initial distance of, approximately 0.8 mm, the force increases linearly due to stretching of the cord before it began to pull the wagon forward. When the blade made contact with the adhered paper the force increased significantly.

![Figure 32: Results obtained with the tensile tester.](image)

The average force to scrape just the metal was calculated from the measurements between the two marks on the dashed curve (the first mark was chosen at the point where the measurements was stabilized after stretching the cord). To develop the method, three different ways of analyzing the curve were investigated, see Paper 3.
The preferred method was to find the highest peak in the test curve and subtract the average force for the metal. This method gave the narrowest confidence interval for the test points.

This creping equipment was designed so that the creping angle could be changed. In this work, four different angles were compared, Figure 33. A creping angle of 50° resulted in a much higher adhesion force than angles of 70°, 89° and 100°. There is a significant difference between the results obtained at a creping angle of 70°, and these obtained at the two larger creping angles. A decreasing creping force with increasing creping angle was also reported by Ramasubramanian and Shmagin (2000), using a rotating laboratory device with a creping blade. At the smallest creping angle, the scraped-off paper was trapped by the blade. When the creping angle is less than 90°, a pocket is created between the blade tip and the drying cylinder where the paper can be easily trapped. Instead of scraping off the paper from the metal, the paper is then compacted and sheared off. When the creping angle is increased from 50° to 70°, some paper is still stuck in the pocket, and this contributes to a higher creping force. At the two larger angles there is no difference in creping force. At a creping angle larger than 90° the paper falls off the blade tip and the paper is separated from the metal by a cut in the coating layer, instead of by shearing off the paper. In tissue mills, the creping angle is normally not less than 90°.

![Figure 33. Adhesion force between paper and metal at creping angles of 50°, 70°, 89° and 100°. The error bars shows the 95% confidence interval.](image)

The mechanical properties of the wrinkled parts of the creped papers were evaluated and the average elongation for the trial points is shown in Table 3. The length of the creped parts of each paper was measured and only the creped part
was placed between the clamps in the tensile tester. The crepe ratio was calculated as:

\[
\text{crepe ratio} = \frac{\Delta L}{\Delta L + L}
\]

Eq 2

\Equation 2\ corresponds to \Equation 1\ in chapter 2.3.1, which is expressed in tissue machine terms, and \(\Delta L\) is the change in length when the paper is fully extended at maximum load and \(L\) is the initial length of the creped paper. The crepes were stretched out at the same time as the paper was breaking. Therefore, not only the unfolding of the crepes was measured, but also the straining of the paper. The elongation was calculated as:

\[
\text{elongation (\%)} = \frac{\Delta L}{L} \times 100
\]

Eq 3

<table>
<thead>
<tr>
<th>Creping angle</th>
<th>Elongation (%)</th>
<th>Crepe ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>50</td>
<td>6170±1050</td>
<td>0.98±0.01</td>
</tr>
<tr>
<td>70</td>
<td>550±130</td>
<td>0.83±0.03</td>
</tr>
<tr>
<td>89</td>
<td>140±10</td>
<td>0.57±0.02</td>
</tr>
<tr>
<td>100</td>
<td>280±40</td>
<td>0.72±0.04</td>
</tr>
</tbody>
</table>

Table 3. Elongation (%) at failure with 95% confidence interval and crepe ratio for papers creped at angles of 50º, 70º, 89º and 100º.

The papers scraped at creping angle of 50º had an elongation more than ten times greater than the papers separated off at the other angles. The measurements for this angle were made on shorter test strips, and this could affect the stretch. The paper was wrinkled to both smaller crepe folds and larger folds (Hollmark, 1972) because the blade held the paper between the metal strip and blade.

4.3.2 Beating

The pulp was beaten to different levels to see the effect on the adhesion. \Figure 34\ shows the impact of beating on the creping force for the SW roundwood pulp. In the diagram, the creping force is plotted versus the tensile strength index in the MD and the beating levels correspond to SR values: 14.5º, 15.8º, 16.8º and 19º. Beating clearly increased the creping force. Beating leads to internal- and external fibrillation of fibers and the formation of fines (Norman, 1992). Fibrillation increases the flexibility and contact surface of the fiber and therefore also the possibility of bonding. The fines will be mixed into the coating layer on the metal
plate when the paper web adheres and will contribute to the adhesion between paper and metal surface. More bonds between fibers lead to a stiffer fiber network and an increased creping force. The force needed to scrape off the paper from the metal includes therefore overcoming adhesion and the force to buckle the paper itself. The higher the grammage, the greater is the contribution of the tensile stiffness to the creping force. When the pulp is beaten, the area for fiber bonds increases and the tensile strength increases (Gigac and Fisėrová, 2008) due to a larger number of hydrogen bonds (Persson, 1996) between the fibers.

Figure 34. Creping force versus tensile index for the SW roundwood pulp at different beating levels: unbeaten (SR 14,5°), 190 rotations (SR 15,8°), 570 rotations (SR 16,8°) and 1000 rotations (SR 19°). The error bars show the 95% confidence interval.
4.3.3 Different pulps

In the study with different types of fibres in the pulp, the paper made of Eucalyptus Grandis/Globulus showed a higher adhesion force than the other papers made from Eucalyptus, softwood (roundwood chips) and softwood (sawmill chips), Figure 35. Note that the pulps were not beaten in this experiment.

![Figure 35. Mean adhesion and standard deviation for two types of softwood and two types of Eucalyptus pulps.](image)

The pulp from SW sawmill chips had the lowest adhesion and showed a lower creping force than the SW from roundwood chips. The SW roundwood chip pulp had shorter and more slender fibres than the SW sawmill chips, and this contributes to a greater contact area and consequently a greater adhesion. Eucalyptus with 75% Grandis and 25% Globulus showed a much higher creping force than the other pulps. One reason for the difference between the two eucalyptus pulps could be the higher content of hemicellulose in Eucalyptus Grandis(75)/Globulus(25). Higher hemicellulose content could make it easier to adhere to the metal (Fuxelius, 1967, Grigoriev et al., 2008). This pulp also had more fines contributing to a larger contact area and better adhesion. The grammage of the paper made of Eucalyptus Grandis(75)/Globulus(25) was higher than that of Eucalyptus Eurograndis and this can explain up to 1/3 of the difference in creping force between these two pulps (Boudreau, 2013).
The SW sawmill chips had the highest moisture content when the paper adhered to the metal, and this could have affected the result in Figure 35. The drier the paper when adhering to the metal surface, the stronger is the drying tension in the paper and the paper is cockled. This prevents the bonding regions on the fibers from coming close to the coating chemicals on the metal. Rewetting of the cylinder coating is affected by the moisture content in the paper and this in turn affects the tackiness of the coating. A paper with higher moisture content is more flexible and sticks more easy to the metal. The small difference in dryness in this trial should make no difference and definitely not the significant difference observed between SW roundwood chips and Eucalyptus Grandis(75%)/Globulus(25%).

Table 4 shows a comparison of the different pulp samples and how the parameters according to theory influence the creping force. The basic data for this comparison are found in the Tables and Figures in this report, and the theoretical influence of a certain parameter is given as minus, zero or plus. As can be seen, the Eucalyptus Grandis/Globulus pulp received significantly more plus than the other pulps and the SW sawmill pulp received the lowest amount of plus. The other two pulps received one plus each. The individual rating of the four pulp samples is in good agreement with the creping force values obtained in the experiment and given in the final column of Table 4. Thus, this simple comparison between the different paper grades can relatively well explain the results obtained for the paper samples in this project.

Table 4. Different factors affecting the creping force. + indicates a positive contribution to an increase in creping force.

<table>
<thead>
<tr>
<th>Furnish</th>
<th>Grammage</th>
<th>Dryness</th>
<th>SR</th>
<th>Fines</th>
<th>Hemicellulose</th>
<th>Total</th>
<th>Creping force (N/m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SW sawmill</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>1230</td>
</tr>
<tr>
<td>Eurograndis</td>
<td>+</td>
<td>0</td>
<td>+</td>
<td>-</td>
<td>0</td>
<td>+</td>
<td>1310</td>
</tr>
<tr>
<td>SW roundwood</td>
<td>+</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>+</td>
<td>1350</td>
</tr>
<tr>
<td>Eucalyptus</td>
<td>+++</td>
<td>0</td>
<td>++</td>
<td>++</td>
<td>++</td>
<td>+++</td>
<td>2110</td>
</tr>
<tr>
<td>Grand/Glob</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The force measured with the scraping equipment and tensile tester is probably related to both the adhesion between paper and metal and the buckling of the paper. Hence the grammage is affecting the paper stiffness and the buckling in the creping action.
4.3.4 Effect of pulp pH

In the study, the adhesion values were compared for papers made from SW roundwood pulp at pH 4.8, 6.6 and 7.9. *Figure 36* shows no significant difference in creping force with pH. Note that the error bars, 95% confidence intervals, overlap. The adhesive used in this study was not particularly reactive and there were few azetidinium groups in the adhesive to cross-link, and this gives a fairly stable adhesive even if the pH changes, as is supported by Grigoriev et al. (2008). In the industry, the coating layer builds up over time to reach a steady state. In this study, the coating layer in contact with the paper web corresponds to only one rotation with the Yankee cylinder. Thus, the papers with different pH have probably had insufficient time to affect the coating layer as much as in a commercial process. This is probably the reason for the lack of effect of pH on the adhesion in this study.

*Figure 36*. Mean creping force and standard deviation at different pH level for the SW roundwood pulp.
4.4 Crepe wavelength measurements

In this project, a simple optical method was used to measure the surface structure of a moving paper web. The goal was to obtain a clear signal from the structure of the creped paper to provide the basis for a future development of an on-line method. The trials were made in cooperation with Acreo AB.

Initially an optical fibre emitting light and collecting the reflected light was used. A second trial was made with collimated light (the rays are parallel) and a third trial with focusing lenses. The optical fibre and focusing lens gave about the same result, but it was easier to use the focusing lens due to the longer distance to the paper. The collimated light gave no result at all, probably because too large a collimator used. These measurements were made perpendicular to the paper. There were a lot of disturbances in the data and no clear period could be extracted. The scatter in the signal was probably due to reflection inside the paper disturbing the reflection from the crepe structure on the outside of the paper.

New trials were made at angles of 10° and 45° from the paper surface and the reflected light was collected at the same angle (Figure 25). Figure 37 shows the Fourier transformed result of the trials. Measurements at 45° were also made when the paper was stationary, as a reference to assess the noise.

With a macro-creping length of about 100 μm and a velocity of 0.5 mm/s, a peak would be expected at about 5 Hz in the Fourier transformed diagram.
(Petermann, 2008). The curve obtained at an angle of 45° has many peaks, but the largest showed up around 5 Hz. This angle was therefore used in the subsequent trials.

Four industrial papers have been studied with a LED with a central wavelength of 980 nm. The papers used had a crepe wavelength between 200 µm and 300 µm and were travelling at a speed of 5 mm/s. This gives a peak in the pseudo spectrum between 1.67 Hz and 2.5 Hz. Each data set was first low-pass filtered with a cut off frequency at 5 Hz before the MUSIC method was applied. The MUSIC method estimated the frequency, and the pseudo-spectra for four papers can be seen in Figure 38.

![Figure 38. Pseudospectra for papers A, B, C and D.](image)

The frequencies were recalculated to wavelengths and the results are shown in Figure 39, together with the results of measurements made with off-line commercial equipment based on image analysis (Bonday, 2010) and an optical microscope. For papers A, B and C the results obtained correlated well with the results of the commercial method. Paper D showed a difference and the confidence interval was larger. This paper is a TAD paper, from which it is difficult to get a clear crepe wavelength. TAD paper has one structure from the through-air drying and another creped structure from the Yankee cylinder.
This LED method of measuring the crepe wavelength probably has a good potential to work on-line. Whether the higher speed used in the industry affects the result needs to be investigated further. The sampling takes a few seconds and the mathematical analyses a few additional seconds. The total time should be sufficiently short to be able to run the equipment on-line in a tissue machine.

5 Conclusions

The chemical compositions of the coating layer on the Yankee cylinder and of the final tissue paper have been studied together with morphological investigations of the surface of the Yankee coating. One result was that the adhesive content was significantly lower in the outer coating layer than in the inner coating layer. This suggests a gradient in the coating layer, where more adhesive is present deeper in the coating. The coating layer consists mainly of carbohydrates and this was patchy, and there were imprints of fibres and fibre fragments embedded in the coating. The blade streaks in the coating layer show that only the higher layers of the patchy coating touch the blade. The coating was not transparent and the coating thickness measurements therefore had to be based on a technique not requiring a clear coating layer.

Equipment intended for on-line measurement of the coating thickness on a Yankee cylinder was studied on a laboratory scale. A fluorescent optical brightener
was added to the coating chemicals and the intensity of the light emitted as a result of UV radiation was measured using a UV-LED. The signal from the trial was scattered and there was no correlation between the amount of brightener and the intensity of the emitted light. The equipment clearly responded to the optical brightener, but the technique requires further investigation before it can be used on a tissue machine. Trials are for example required to see whether the adhesive itself disturbs the measurement method, and most important to investigate the reason for the disturbances in the measurements when the cylinder is rotating.

New creping equipment was development together with a new adhesion method to be used on a laboratory scale. The equipment together with a tensile tester scrapes off the paper and measures the force needed. The creping blade can easily be changed if a different creping angle is desired. The study showed that the adhesion strength increased with increasing grammage and increasing degree of beating of the pulp. It was also found that a pulp with more fines or with higher hemicellulose content had higher adhesion strength.

Measurements of the crepe wavelength of the tissue paper have been made with a simple probe using an optical fibre at the surface of the paper. The probe has been used alone, with a focusing lens perpendicular to and at an angle to the paper and also with collimated light. The fibre with a lens and illuminated at an angle of 45° was most promising. Readings were made on commercial papers. The signal contained a lot of information and to distinguish the most common wavelength for each paper, mathematical analyses were made with the MUSIC method. The measured wavelength was close to the measurements made by commercial equipment (less than 3% difference for DCT (Dry Creping Technology) papers) and this technique has a good potential for use on-line.
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New methods for evaluation of tissue creping and the importance of coating, paper and adhesion

The creping process is the heart of tissue paper manufacture. To control the process better, on-line measurements of paper structure and coating thickness are sought after. The creping is highly dependent on the adhesion of the paper to the Yankee dryer. To be able to measure the adhesion, laboratory creping equipment was also required. Different pulp parameters affect the adhesion and some of them have been investigated in this work.

The coating on the Yankee cylinder consisted mainly of fiber fragments and could not be considered as transparent, which had to be considered when choosing a method to measure coating thickness. A method based on the light emitted from an optical brightener in the coating when subjected to UV-irradiation was used, but has to be further improved before it can be used on-line.

A new laboratory creping method was developed to determine the adhesion between paper and metal, and the force needed to scrape off the paper with a doctor blade was measured. The highest creping force was obtained for papers made of pulp with a high drainability, high fines content and high hemicellulose content.

An optical method using reflected light to measure crepe wavelength on-line was developed. The paper travelled under a sensor and the light collected was mathematically analyzed to determine the most common wavelength.

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