Predicting catastrophic failure in barrier coated packaging board and paper after creasing and folding

Proposing a methodology to predict barrier failure after creasing and folding

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Sammanfattning


Utvärderingen började med visuell inspektion av bigade/vikta substrat i ljusmikroskop för att finna barriärdefekter i form av sprickor. Både bra och dåliga prover testades sedan för fetteständighet med hjälp av ett standardtest, dvs TAPPI 454. TAPPI 454 testet visades sig att vara ett effektivt sätt att identifiera barriärdefekter på grund av att penetration av olja vid biglinjen skedde snabbt på de prov som uppvisade sprickor. Även några av de prov som ej uppvisade sprickor i ljusmikroskop klara inte av fetteständighetstest. Resultatet visade att det enda material som kunde bibehålla barriärgenskaper efter bigning och vikning var de PE belagda proven. Detta är antagligen tack vare PE-bestrykningens höga dukulitet och tjocklek.

Vattenångspermeabiliteten, WVTR, uppmättes på de prov som uthärdade fetteständighetstestet. Eftersom PE är en utmärkt vattenångbarriär, var WVTR-mätningar lämpliga för att upptäcka barriärfel. WVTR resultaten för de bigade/vikta proven visade ett något högre värde än de obigade referenserna även om de inte hade sprickor. Det något högre WVTR värdet beror antagligen på att barriärskiktet blev tunnare på grund av töjningen i barriärskiktet under big/vikningen.

En metod för att förutspa skador i barriärbetryckt kartong och papper efter bigning och vikning föreslogs. Definierade big- och vikgeometrier användes i kombination av screening av sprickor i barriärskikten, först genom visuell inspektion i ljusmikroskop och sedan ett standardiserat fetteständighetstest. Proven som passerar screeningen kan sedan bli analyserade för mer exakta och tidskrävande metoder som WVTR.

Figur 1. Schematisk figur över den föreslagna metodiken.
Abstract

Different methods to predict barrier failure in packaging board or paper after converting were investigated. The approach was to compare substrates before and after creasing/folding by applying different barrier tests and to propose a methodology to predict failure in the barrier layer. Different coatings were used to develop and verify the methodology: a hemicellulose based dispersion barrier coating, a dispersion coated PVOH coating and an extrusion coated PE. Creasing was performed according to standard procedure using recommended creasing geometries. Folding of paper was performed by a gentle creasing with a board backing followed by folding the paper between two metal plates with a well defined distance.

The first step in the evaluation was to visually inspect creased/folded substrates by light microscopy to search for coating failures in form of cracks. Both good and bad samples were then tested for grease resistance with a standard test, i.e. TAPPI 454. The TAPPI 454 test showed to be effective to expose barrier failure since oil would penetrate quite fast through the creasing line of cracked samples.

Even some samples that appeared to have no cracks in the light microscope showed failure with the grease test. The results showed that only the PE coated samples could sustain a barrier after creasing and folding. This was probably due to a high ductility of the PE-coating combined with a high thickness.

The water vapour transmission rate, WVTR, of the samples that passed the TAPPI 454 test was then measured on the samples that endured the grease resistance test. Since PE is a good water vapour barrier, WVTR-measurements were proper for detecting barrier defects. The VWTR of the creased/folded samples was slightly higher for the creased samples than the un-creased references despite the absence of cracks. This was probably due to that the barrier layer got thinner as a result of the strains applied on the coating during the creasing/folding operation.

A methodology to predict barrier failure in barrier coated packaging board and paper after creasing and folding was proposed. Well defined creasing and folding geometries were used in combination with screening for cracks in the barrier layer, first by visual inspection in light microscopy and then by a standard grease resistance test. The samples that passed then screening tests could then be analyzed using more exact but also more time consuming methods such as WVTR.

Figure 2. A schematic figure of the proposed methodology.
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1. Introduction

1.1 Background

Year 2016, 1.3 million tonnes of packages were distributed on the Swedish market. Packaging materials based on paper and paperboard accounted for 43% and those based on plastic (excl. PET) for 16% (Naturvårdsverket, 2017). Paper and board materials are to a higher extent recycled than plastics (excl. PET); 82% compared to 47%. Cellulosic based packaging materials such as paper and paperboard are to a great extent made by renewable resources and 99% of all plastics are produced from fossil fuels (European bioplastics, 2017). Among many differences, the cellulosic based packaging materials has a limited ability to prevent e.g. grease, water vapour and oxygen to diffuse through compared to those packaging materials based on plastic and metallic.

Cellulose based packages are made up of cellulose which is the most abundant carbohydrate on Earth and the major building block in all plants (Henriksson, G. Lennholm, H. 2009). Cellulose is built up by β-glucopyranoside molecules in long linear chains with 1->4 glucosidic bonds, the polymer can have a degree of polymerization (DP) up to 15 000 residues linked together (Henriksson, G & Lennholm, H. 2009). Hydrogen bonds and hydrophobic interaction bind these chains bond together which creates cellulose fibrils (Henriksson, G. Lennholm, H. 2009). These cellulose fibrils, in turn bind together making fibril aggregates and in plants these are most abundant in the secondary cell wall (Henriksson, G. Lennholm, H. 2009).

The fibers are obtained by the pulp- & paper industry to make paper, card- and paperboard etc and the raw material is trees (softwood & hardwood). The cellulose is extracted by different methods such as mechanical (stone groundwood/thermomechanical pulp or chemi-thermomechanical pulp) or chemical (kraft-/soda cooking /sulphite cooking) pulping where the cellulose is separated from by-products such as hemicellulose, lignin and salts. The pulp is then used to make paper/paper– or cardboard with or without bleaching and drying of the pulp (Brännvall, E. 2009).

The process to make paper/card- or paperboard of different grades starts with the pulp suspension being distributed evenly across the forming section on the wire in the paper machine (Brännvall, E. 2009). When the pulp loses water a fibre network is formed. Different velocity profiles between the wire and the sprayed pulp will result in orientation introducing anisotropy of the paper products (Wahlström, T. 2009). Equation [1] describes the definition of anisotropy, A is the anisotropy, \( n_{MD} \) is the number of fibers which lie in the machine direction (MD) and \( n_{CD} \) is the number of fibers which is in the cross direction (CD) (Wahlström, T. 2009).

\[
A_{fibre} = \frac{n_{MD}}{n_{CD}} \tag{1}
\]
Along the paper machine the newly formed fibre network is exposed to vacuum dewatering, pressing and drying cylinders (Brännvall, E. 2009). The finished paper/cardboard/paperboard is often coated to make the surface even or and enhance the printability of the product to improve the package appearance and to provide with information (Engström, G. 2009). The coating mainly contains a pigment e.g, clay or calcium carbonate and latex binder (Engström, G. 2009).

The paper-/cardboard which are coated has to be converted into a package before the content is inserted, the converting process involves printing, cutting, creasing, sealing and folding to make the carton ready for use (Söremark, C. Tryding, J. 2009). The creasing is performed to make fold lines where the carton are later folded. The creasing lowers the folding resistance/bending stiffness through delamination within the paperboard, this gives the substrate a different characteristic compared to skip creasing and folds directly. The creasing and cutting are made in the same operation and are made on a flatbed die or a rotary die (Kirwan, M J. 2005 and Lahti et al. 2008).

1.2 Creasing

A rotary die is made up of two pairs of rolls, one for cutting and one for creasing. The cutting rolls are equipped with cutting and creasing rulers. There are two different ways of cutting in the rotary die, crush cutting where the cutting operation is similar to that of a flatbed die, and pressure cutting. The pressure cutting uses shear forces to cut the paperboard which makes use of flat lands of both cylinders which do not get into contact with each other during the cutting. The creasing rolls have a creasing ruler on one of the rolls and a groove on the other one (Kirwan, M J. 2005).

The flatbed die is made up of an upper and a lower plate. The cutting- and creasing rulers are mounted on the upper plate. The lower platen is flat except for the creasing areas, where groove (matrices) are placed at the counter plates. The cutting rule has a sharp edge and the creasing rule has a round edge. When cutting and creasing the lower platen is raised towards the upper platen to let the paperboard and the lower platen to come in contacts with the rule (Kirwan, M J. 2005).

There are several advantages with using rotary die creasing and cutting, lower pressures are needed and they are more economical due to more cycles can be made before a need to re-sharpen the rules (Kirwan, M J. 2005).

When creasing the paperboard is exposed to compression-, shear- and tensile forces within the cardboard, see figure 3. The surface of the board is subjected to the largest tensile strains during the loading which leads to a severe stretch of the top layer (Kirwan, M J. 2005).

![Figure 3. figure of how the forces acts on a substrate during a crease. The notations in the figure corresponds to the different forces: t=tensile, c=compression and s=shear. Sampled from Lahti et al. 2008.](image-url)
The shear forces act parallel to the surface and the compression forces perpendicular to the surface (Kirwan, M J. 2005). The creasing shear forces cause delamination between the fibre layers (Nygårds et al. 2009). The maximal state during loading, compression in the z-direction is of most importance due to creation of shear forces in the board which results in delamination (Nygårds et al. 2009). During unloading normal stresses within the board causes delamination (Nygårds et al. 2009). To achieve as much delamination as possible, the shear forces should deform the layers plastically, which is most easily achieved with well-defined layers with weak interfaces (Nygårds et al. 2009). In these interfaces the bonds should break but not fibers.

The ability to achieve a good crease depends on several factors on the paperboard and the creasing equipment. Concerning the paperboard, the properties of most interest are thickness, anisotropy, tensile strength, elongation, elasticity and compressibility in the Z-direction (Lahti et al. 2008).

Uni-axial tensile testing is a tool that is used to characterize materials. This test is performed in a tensile tester which pulls a test sample at a constant deformation rate while registering the applied force. The stress and strain are then calculated from the length, cross section area and the elongation of the sample. Figure 4 shows a typical stress-strain curve obtained from such a test. The tensile absorption index is the area beneath the stress-strain curve and the ductility of a material is the ability to withstand cracking during plastic deformation (Fellers, C, 2009 and Davis, J R. 2004). The tensile index is the maximum stress to which the material is exposed and defined as the global maximum on the stress-strain curve normalized by width and grammage of the material (Fellers, C, 2009). This mechanical property tells us about the materials strength. In equation [2] the tensile strength (σ) is shown, F is the force, b is the width of the sample and t is the thickness of the sample (Fellers, C, 2009).

\[ \sigma = \frac{F}{b \times t} \]  \[2\]

The strain at break is the maximum elongation before the material breaks and tells us about how much the material can be stretched before breaking (Fellers, C, 2009). In equation [3] elongation (ε) is described, Δl is the difference in length between the length when the material is stretched subtracted with the starting length, l is the length when the material is stretched (Fellers, C, 2009).

\[ \varepsilon = \frac{\Delta l}{l} \]  \[3\]
The tensile stiffness index or elastic modulus is a measure of the linear elastic properties and characterizes the stiffness of the material (Fellers, C, 2009). The elastic modulus is defined in equation [4] which is the slope of the linear part of the stress-strain curve at low strains, i.e. before the plastic deformation starts (Fellers, C, 2009).

\[ E = \frac{\Delta \sigma}{\Delta \varepsilon} \]  

where \( E \) is the elastic modulus, \( \Delta \sigma \) is the difference of the stress in the linear part of the stress-strain curve and \( \Delta \varepsilon \) is the difference in elongation of the same part of the curve (Fellers, C, 2009).

Since paper and paperboard are anisotropic materials which implies that creasing in different directions differs. Creasing in MD and CD, see figure 5, would thus most probably give different results.

The important geometric parameters during creasing are depth and width of the groove, length and width of the creasing ruler. Figure 4 shows how the groove geometry affects the creasing result for one substrate thickness and one creasing ruler. The occurrence of cracking of the substrate is related to sharp- and/or insufficient creasing, areas b, c and d in figure 6 (Lahti et al 2008).

A schematic picture over the laboratory creasing equipment used in the laboratory is showed in figure 7. The parameters for the creasing rule is \( b_r \) which is the width and the length \( H_r \). The parameters for the matrix \( t_f \) which is matrix thickness which corresponds to the depth of the groove in figure 7 and \( b_n \) which is the groove width (Iggesund product catalogue, 2016).

How the behavior of the coating on the cardboard acts during the creasing is of importance for costumers and producers. This includes e.g advertisement and the table of contents on food packages. The print on the package could look undesirable in the edges where the creasing have been performed due to cracks. The amount of cracks during creasing can depend on the mechanical properties of the pigment coating (Rättö. P, Hornatowska. J, 2010).
The creasing is also of importance in another perspective for the food industry. The shelf life of the product within the package depend upon several factor other than just the product itself. The package have to sustain protected atmosphere for the product and defects in the barrier coating could compromise the barrier function of the package and reduce the shelf life.

1.3 Barrier coating & Barrier properties

Barrier coatings are made to protect the packaging material, especially the packaged product itself. Barrier coatings should protect the packaging content from light, grease, water vapour and aromas (Kirwan, M J. 2005). Quite often, the packaging material also needs to be protected moisture from the packaging content in order to keep its mechanical integrity. There are three kinds of different coating operations which are mainly used today: extrusion-, lamination- and dispersion coating. Extrusion coating is a process where a material is processed in an extruder and applied on the substrate (e.g a plastic that is melted in the extruder, formed to a film and solidified on the substrate under pressure) (Kuusipalo et al. 2008). Lamination is a coating process where two or more different webs of flat materials are “glued” together with an adhesive (Kuusipalo, J. Avellan, J. 2008). In dispersion coating, a water based coating/dispersion is applied on the substrate in a similar way as a traditional pigment coating (Kimpimäki, T. 2008).

Dispersion coatings usually contain a water soluble polymer or a dispersed granulate. The polymer is usually combined with a mineral filler, but may also contain thickeners, surfactants, chelating agents etc (Kimpimäki, T. 2008).

The polymers have to form a continuous film to achieve a barrier function. The film formation theory has been investigated for latex binders and been proposed to have three stages: water evaporation, dense packing and coalescence (Kimpimäki, T. 2008). The polymers used in dispersion coatings are latices such as polyacrylate and styrene-butadiene or water soluble polymers such as polyvinylealcohol (PVOH) (Kimpimäki, T. 2008). During application of the dispersion coating onto the substrate the polymer granulates or polymer molecules move freely in the water and packs closer during evaporation (Kimpimäki, T. 2008). As the water is removed the close packed granulates or polymer molecules start to coalescence into a uniform continuous layer (Kimpimäki, T. 2008).

Fillers are used for several reasons. One is economical since the latices and polymers are expansive components (Kimpimäki, T. 2008). The other aspect is that fillers are impermeable which forces the diffusing gas molecules to take a route around the filler, i.e the tortuosity is increased. The tortuosity depends on the alignment and the aspect ratio of the filler. For barrier coatings, platy fillers like clay or talc are preferred since they would give the highest tortuosity (Bollström et al. 2013).
The more aligned the platy particles are in the coating direction the more tortuous path they will make for the diffusing gas molecules (Bollström et al. 2013).

The mass transfer process through a material can be divided into three parts: absorption, diffusion and desorption (Han, H J, Scanlon, M G. 2005). The driving force of mass transfer of a substance through the coating layer is the concentration difference of the substance in the media of the opposite sides of the layer, since the nature strives for equilibrium (Han, H J, Scanlon, M G. 2005). The gas or solute which passes through the coating layer first dissolves into the layer (absorption), the gas/solute passes through the coating layer along the concentration gradient(diffusion) and finally dissolves from the coating layer(desorption) and this three stages is called permeation (Han, H J, Scanlon, M G. 2005). The absorption and desoprtion of the permeant into and out of the package is dependent of how soluble the permeant is in the layer (Han, H J, Scanlon, M G. 2005). The diffusion of a gas or solute in a coating layer follows Fick’s first law (Coulson, J M. Richardsson, J F. 1999), see equation [4] (Auvinen et al. 2008). This law states that the rate of the diffusion is linear to the concentration gradient over the coating layer (Auvinen et al. 2008). \( J \) is the molar flux of the permeant, \( D \) is the diffusion coefficient, \( c \) is the concentration of the permeant and \( x \) is the distance the permeant has travelled. Flux is a measure of the quantity which passes over a specified area during a specific time.

\[
J = -D \frac{dc}{dx}
\]  
\[4\]

After a while, the diffusion reaches a steady state which will last as long as key variables such as the temperature and the concentration gradient remains constant (Auvinen et al. 2008). This steady state is manifested by a constant flux described by Fick’s second law, see equation [5] (Auvinen et al. 2008). \( J_s \) is the steady state flux, \( l \) thickness of the coating layer and \( c_1 - c_0 \) which is the difference in concentration of the permeant (Auvinen et al. 2008).

\[
J_s = \frac{D(c_1-c_0)}{l}
\]  
\[5\]

For gases phases, this steady state flux can be rewritten to the partial pressure of the permeant. This is made by using Henry’s law (in most of cases valid at low permeant concentrations) which states that the concentration of the permeant is equal to the solubility coefficient \( S \) and the partial pressure \( p \), see equation [6] (Auvinen et al. 2008). This together with a paraphrasing using the permeation coefficient \( P \) instead of the solubility \( S \) and diffusion coefficient \( D \), see equation [7] (Auvinen et al. 2008).

\[
c = S \cdot p
\]  
\[6\]

\[
P = D \cdot S
\]  
\[7\]

$$J_s = \frac{p(p_1-p_0)}{l}$$  \hspace{1cm} [8]

There are some parameters which affects the permeability of a gas/solvent. These are, except for the temperature, mostly associated to polymers/conventional barrier materials (Auvinen et al. 2008). The properties which are associated with the polymers are thickness, glass transition temperature, moisture content, molecular weight, polymer density, cross linking and crystallinity and orientation (Auvinen et al. 2008). Methods such as oxygen transmission rate (OTR), ambient oxygen ingress rate (AOIR) or water vapour transmission rate (WVTR) are ways to measure the permeation of oxygen resp. water vapour through a barrier layer (Auvinen et al. 2008).

When measuring water vapour transmission rate a concentration difference is applied over the sample which is attached on an impermeable test cell. The sample forms the boarder between one phase inside the test cell and the other phase outside. The barrier layer of the sample should be towards the more moisture side. The uptake or release of the inner cell side is followed by weighing until the plot of the weight versus days is linear for four days. The water vapour transmission rate is calculated according to equation [9] where $WVTR$ is the water vapour transmission rate, $m$ is the linear part of the slope of when the increase in mass is plotted per hour, $SA$ is the surface area of the sample (ISO2528:1995).

$$WVTR = \frac{240 \times m}{SA}$$  \hspace{1cm} [9]

1.4 Aim

Measurements of WVTR or OTR give a quite good picture of the barrier function of different materials. However, they are quite time consuming and quite expansive, and the application of such methods on a larger test matrix is usually not practically possible. There are is also a need within the packaging industry for quality assurance and screening where faster methods would be possible.

The aim for this thesis is to propose a methodology that predicts when barrier failure occurs after creasing and folding of cellulosic materials as paperboard and paper. In order to be able to study creasing parameters a relatively large number of test points are needed. Therefor the development of a screening procedure is needed as well as the knowledge of how to apply this methodology in terms of benefits and limitations of the screening methods.
2. Materials and method

The material and method is presented in such a way as that a technical description of the method is presented under each section and how the method went on is described under the subheading “approach”. All of the experimental work except for the coating were performed according to ISO187:1990 at 23°C and 50% relative humidity (RH).

2.1 Substrates

The paperboard used for the trials were ‘Invercote G220’ from Iggesund, Paperboard, Iggesund, Sweden. The paperboard contains three layers of bleached chemical pulp (2 thinner outer plies and one thicker inner ply) and had a grammage of 220 g/m² ± 4 % (according to ISO536 and a thickness of 260 μm ± 4 % (according to ISO534) (Iggesund product catalogue, 2015-2016). The outer plies are further coated with three layers of pigment coating on the top side and one layer on the bottom side.

The packaging paper used had a grammage of 60 g/m² (according to ISO536) and a thickness of 52 μm (according to ISO534).

Table 1. Scheme over the different substrates which had been coated by a pilot coater and their respective amount of coating layer.

<table>
<thead>
<tr>
<th>Sample detonation</th>
<th>Substrate</th>
<th>8877 (g/m²)</th>
<th>Layer 1 (g/m²)</th>
<th>Layer 2 (g/m²)</th>
<th>Layer 3 (g/m²)</th>
<th>Total amount of coating layer (g/m²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>304</td>
<td>Paper</td>
<td>6,4</td>
<td>0,37</td>
<td>0,81</td>
<td></td>
<td>1,18</td>
</tr>
<tr>
<td>103</td>
<td>Board</td>
<td>2,1</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>403</td>
<td>Board</td>
<td>2,1</td>
<td>0,56</td>
<td>0,41</td>
<td>0,41</td>
<td>1,38</td>
</tr>
</tbody>
</table>

The paper and board had been pilot coated with a primer (8877, BIM Kemi, Gothenburg, Sweden) and a hemicellulose based barrier. The paper was coated two layers of hemicellulose based barrier while the board had been coated with three layers of hemicellulose based barrier in a pilot coater according to table 1.

To obtain further references, PVOH coated paperboard and extrusion coated PE paperboard was used. In both cases, Invercote G220 was used as substrate. The PVOH was applied in a pilot coater with a dispersion of PVOH in three layers giving a total applied grammage of 12 g/m².
The PE was applied on the board and packaging paper at Iggesund Paperboard, Strömsbruk, Sweden with a applied amount of 20 g/m². A glossy PE was applied on the packaging papers by a smooth chill roll. The packaging paper and board was corona treated before the PE was applied.

2.2 Coating

Since the coated amounts of the pilot coated materials were rather low, additional hemicellulose based barrier coating was applied with a laboratory bench coater, K202 control coater, from RK Print Coat Instrument Ltd, United kingdom. The coating was applied in the CD direction of the paper and board. The coating was made on a K202 control coater with the speed of 4 m/s and the coated substrates were dried for 120 s at 105 °C. The substrates used are listed in table 1. Three coating layers were applied using yellow (6 microns wet deposit) and red (12 microns wet deposit) rods in different combinations. Half of the paperboards were coated according to this scheme (bottom coating layer -> middle coating layer -> top coating layer): yellow, yellow and red (GGR) while the other half were coated: yellow, red and red (GRR). The yellow rod was used for the first layer in both cases since the barrier coating showed tendency to contract after the application of the first layer.

The barrier coating slurry’s dryness was tested before it was applied on the paperboard/paper. The average dryness of the coating slurry were: 24.54 %. This were tested in an IR Balance and the data sample were one for each day (the coating took three days), the number of replications were three.

The coated paper and paperboard was sent to MoRe Research to analyze the lithium ion content in the barrier coating and thus calculate the applied hemi coating. The calculated total application of hemi coating were: 304 GGR had coating grammage of 9,1 g/m², 304 GRR had a grammage of 10,5 g/m² and 403 GGR and 103 GRR had both a grammage of 7,8 g/m².

2.3 Casting of self-supporting films

The Hemi-dispersion was diluted to 10 % and then casted into plastic petri dishes which had been covered with a teflon film produced by Sigma-Tech, Bytaç® surface protection laminate (Item: Z27876-9). The amount of hemi-dispersion which was poured into the petri dishes were 10 g ± 0.1 g and the casted films were dried in 23 °C and 50 % RH for three days.

2.4 Creasing

The creasing were performed on a Marbach Werkzeugbau creasing machine, see figure 8. Creasing parameters were chosen for a paperboard with a thickness of 270 μm. According recommendation in Holmen Iggesunds product catalogue, i.e a ruler hight/width of 23,4 mm/0,70 mm.

The ruler were the same for all creasing while the matrix were varied.
The creasing was made by first marking up the fiber direction with lines using a pencil. The sheet was then cut into 10x10 cm² pieces. The thickness of the pieces was measured using a Lorentzen&Wettre Thickness tester. Then the piece of paper/paperboard was inserted on the matrix within the creasing machine and then the samples were creased in the machine during ~5 s. In cases, where little material was available, pieces of 5x10 cm were cut out and sealed to the matrix by tape. After creasing, the samples were folded once.

First, Invercote G220 from another batch than the barrier coated samples were investigated in CD and MD in order to see how the paperboard behaved during creasing by changing the matrices and keeping the ruler constant. The creased paperboard was inspected for crack using light microscope(both in surface and cross section). From these first results paperboard from Invercote G220 from the same batch as the barrier coated was validated to ensure that the paperboard gave the same results as the other batch of Invercote G220.

Creasing of hemicellulose based barrier coated Invercote G220 was based on the results from the validation. The creased samples were analyzed using the same procedure as the uncoated. PE and PVOH coated Invercote G220 was also investigated and the experimental was based on the same type of validation.

2.5 Folding

A sack manufacturer was contacted for guidance to make a fold as similar to how folding is performed in an industrial process. He suggested that a careful crease should be applied with a wide matrix before folding. The paper was therefore creased and folded with an Invercote Creato 200 board as a backing. The Invercote Creato 200 had a grammage of 200 g/m² ± 5 % (according to ISO536) and a thickness of 200 μm ± 5 % (according to ISO534) which implies that the paper and the Invercote Creato 200 had an average thickness of 252 μm together

The folding was made with the paper and the paperboard beneath it was folded together in a slot of 1,05 cm, see figure 9. This was done by folding the paper inside the paperboard and putting it in the slot, then a ruler with the same thickness as the slot pushed the sample through the slot.
Paper was first investigated without barrier coating to see how the material behaved, the creased and folded samples were inspected for cracks by light microscope. With results from the investigation of the paper without barrier coating the barrier coated paper was creased and folded.

2.6 Visual inspection of cracks

The creased and folded paper with and without barrier coating was inspected with a light microscope with magnification of 15x. Both the surface (in-plane) and the cross section were inspected for the creased board without barrier coating while only the surface of the creased barrier coated board and the folded paper (with and without barrier coating) was inspected. If a sample showed excessive cracking, only one of sample was analyzed in the microscope while samples that were judged “good enough” for grease tests were analyzed in four samples in the microscope. Samples without barrier were analyzed in four replicates.

The description of the cracks/deformation was done by commenting on the crack size (tiny, small), if cracking severe or not and if cracks were along the crease, figures 10a and b, or on the crease, figure 10c. The folded samples where noted as a crack in the folding line, see figure 11a and 11b, or in the “bend” which are noted for cracks inside of the folding, example of this is shown in figure 11c. For creased board without barrier, the cross section evaluation was as important as the surface analysis. Figures 10 a to c shows examples of cross section analysis where 12a shows a good crease, 12b a bursted crease and 12c a bad crease.

![Figure 10](image)

*Figure 10. In plane pictures of three creased paperboard with hemicellulose based barrier taken in a light microscope. Arrows indicates cracks.*
The folded and creased samples were then inspected for cracks in a light microscope and from these results a selection of which samples for the grease resistance test were made. The best sample was the one with none or the one with the least cracks and the worst sample was the sample with most cracks, but not the samples where the paper or board had bursted. The best and the worst samples from the visual inspection were further investigated with a grease resistance test.

2.7 Grease resistance test

A modified TAPPI standard 454-test was used to characterize the grease resistance. The TAPPI 454 test prescribes the use of 5.0 g of dry sand on the paper/board surface and the oil is poured on the sand. The sample is placed with a free bottom and a mirror below the sample in order to register when the oil has penetrated the sample. In order for a sample to pass the test, the oil should not penetrate through the sample during 30 minutes. The method also prescribes 10 samples for each test point.

The sand, Mursand 0/4, Råsjö Kross AB, Sweden, was subsequently sieved and dried in an oven at 105 °C for at least 24 h and cooled in a desiccator for at least one hour before testing. The oil used for the test here was a refined rape seed oil manufactured by Eldorado, Estonia.
The colour is a fat soluble Sudan red G (LOT#: BCBD3070V) manufactured by Sigma tech, India. The amount of sand used here was 2.0 g ± 0.1 g and the amount of coloured oil was 440 μL. The number of repetitions were four. The sand was applied on the sample barrier facing up with a metallic cylinder with an inner diameter of 18 mm. On the top of the pile of sand a small pit was made with a pipette tip and the oil was poured into the pit carefully using an automatic pipette. The stop watch was switched on as the last drop of oil left the pipette the and the watch was stopped when the first spot of penetrated oil was visible on the backside of the sample or when the time reached 1800 s. An example of a time sequence as the oil penetrates a sample is shown in figure 14.

![Image](image1)

*Figure 14. The time sequence of the penetration of the coloured oil on a sample. Arrows indicates penetration of the oil.*

2.8 Scanning electron microscope

Scanning electron microscope (SEM) pictures were taken by RISE Research Institutes of Sweden, Stockholm, Sweden. The SEM analysis was performed in SEM from Hitachi; (ModelSU3500), using an accelerating voltage of 10 kV and a chamber pressure of 40 Pa. The magnification was 50x, 100x, 250x, 500x and 1000x. Ten samples, i.e creased and folded barrier coated board and packaging paper, were selected based on the grease resistance test results.
2.9 Water vapour transmission rate

The water vapour transmission rate was tested according to ISO2528:1995, with two repetitions per test point. The test area was masked with an aluminum film and the samples was exposed to an area with 5 cm². The barrier was exposed to a test environment of 23 °C and a RH of 50 % on the outside while the inside of the test cell were filled with 50ml of dried silica gel to achieve an environment of 0 % RH. Testing continued for five days in order ensure steady state conditions.

2.10 Tensile testing

The uniaxial tensile testing was performed using a Zwick screw material tester of model Roell Z005, with a load cell of 1 kN. The tensile testing was performed according to ISO1924-3:2011, the rate of elongation was 100 mm/min and the number of repetitions varied between the samples. Two different materials were tested, PE and casted hemicellulose based barrier.

A sample length of 10 cm was used for the PE samples while a sample length of 4 cm was used for the casted hemicellulose based films. The casted hemicellulose based films were just tested in one direction since they were likely to be isotropic. The PE was extrusion coated and supposedly anisotropic and the PE film was obtained by removing the PE layer from the extrusion coated board or from plastic tape. The PE was therefore tested in both MD and CD and four different versions were tested: one with little fibers attached to it (PE1), one sample with even fewer fibers (PE2) and two samples with no fibers at all (PE3 & PE4). Four different samples were tested as a result of different methods used to remove the film from the board. The PE films with fibers had their fibers removed by rubbing with the thumbs using water to soften the fibers, the PE films without fibers were extruded on a plastic tape. PE3 was a glossy PE and was made by a smooth chill roll.

The thickness of the casted hemicellulose based barrier was measured using a STFI thickness tester with a measuring length of 8 cm. The thickness of the PE films was measured using a Lorentzen & Wettre thickness tester, with a 10 repetitions per test point. The grammage was measured by cutting out a sample with a known area and determining the weight. The repetition was 10 per test point.
3. Results

In this section a presentation of the results are shown. Error bars in the graphs corresponds to 95 % confidence limits unless other is mentioned.

3.1 Creasing

In this section the results of the creased paperboard is represented. The graphs are a summary of the data of the visual inspection found in Appendix I.

Figure 17 shows the good (pass) and bad creases (reject) of a Invercote G220 board without barrier coating at different matrix thicknesses and groove widths. The graph shows that there is an area with combinations of matrix thickness and groove width that gives acceptable creases, just as prescribed by Lahti, J. Hatanpää, I. Lathinen, K. (2008) except for high matrix thicknesses and large groove widths. Results from the creasing test also showed that the area of acceptable crease is larger when creasing in MD than when creasing in CD. The validation gave similar results as the creasing test, see Appendix II.

3.2 Folding

In this section the results of the creasing and folding of the packaging paper is represented. The graphs are a summary of the data from the visual evaluation of the creased and folded paper during the screening that can be found in Appendix I.

Figure 17. Scheme of creasing in CD and MD using a non-barrier coated Invercote G220.
In figure 18, the behavior of the folding line of the uncoated paper is shown. Within the marked ellipse, the fold line of the samples looks more like “crease” for paperboard, compared to the samples outside the ellipse. The samples showed a sharper fold line compared to a more “round” one as schematically indicated in the graph.

**Figure 18.** Scheme over how the fold line for the paper looks after creasing.

The screening of the folding, in figure 19, indicated that the paper also had a boundary where the creasing and folding is OK or not. Results for the creasing and folding screening also showed that the area for acceptable creasing and folding is when creasing and folding in MD compared to CD creasing and folding.

**Figure 19.** Scheme of creasing/folded in CD and MD using a non-barrier coated paper.
3.3 Grease resistance

The grease resistance test are represented in table 2-3. For further details and all data set and full set of data see, Appendix III. The selected dataset for the grease resistance test were based on previous results from test/validation for the paperboard and the screening for the paper are found in Appendix IV. PVOH in MD and CD, PE in MD and PE paper MD and CD where not screened as described earlier. The samples were simply chosen as with the same creasing/folding geometries as with the hemicellulose based barrier coated samples, i.e 103 coated with GRR in CD for the board and 304 coated with GGR in CD and MD for the packaging paper. The visual evaluation of this dataset are to be found in Appendix II. All references (uncreased samples coated with PE, PVOH and hemicellulose based barrier coated samples) had a penetration time (s) of at least 1800. This indicates that all the samples showed a good enough oil barrier before creasing and folding.
The results of the paperboard showed that only two of the samples endures the grease test after creasing. These were the PE coated samples (both in CD and MD) with a matrix thickness 0,5 mm and a groove width of 1,0 mm. The hemicellulose based coated samples showed oil penetration after a short period of time while PVOH coated samples showed oil penetration after somewhat longer times.

<table>
<thead>
<tr>
<th>Material</th>
<th>MD/CD</th>
<th>GRR/GGR</th>
<th>Groove width (mm)</th>
<th>Matrix thickness (mm)</th>
<th>Best/Worst</th>
<th>Time (s)</th>
<th>Conf. T</th>
</tr>
</thead>
<tbody>
<tr>
<td>403</td>
<td>CD</td>
<td>GRR</td>
<td>0,9</td>
<td>0,5</td>
<td>Best</td>
<td>35</td>
<td>28</td>
</tr>
<tr>
<td>403</td>
<td>CD</td>
<td>GRR</td>
<td>1,5</td>
<td>0,8</td>
<td>Worst</td>
<td>40</td>
<td>34</td>
</tr>
<tr>
<td>403</td>
<td>MD</td>
<td>GRR</td>
<td>1,5</td>
<td>0,5</td>
<td>Best</td>
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<td>47</td>
</tr>
<tr>
<td>403</td>
<td>MD</td>
<td>GRR</td>
<td>1,4</td>
<td>0,6</td>
<td>Worst</td>
<td>10</td>
<td>0</td>
</tr>
<tr>
<td>403</td>
<td>CD</td>
<td>GGR</td>
<td>1,0</td>
<td>0,6</td>
<td>Best</td>
<td>15</td>
<td>16</td>
</tr>
<tr>
<td>403</td>
<td>CD</td>
<td>GGR</td>
<td>1,3</td>
<td>0,3</td>
<td>Worst</td>
<td>18</td>
<td>8</td>
</tr>
<tr>
<td>403</td>
<td>MD</td>
<td>GGR</td>
<td>1,6</td>
<td>0,6</td>
<td>Best</td>
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<td>44</td>
</tr>
<tr>
<td>403</td>
<td>MD</td>
<td>GGR</td>
<td>1,0</td>
<td>0,3</td>
<td>Worst</td>
<td>10</td>
<td>0</td>
</tr>
<tr>
<td>103</td>
<td>CD</td>
<td>GRR</td>
<td>1,0</td>
<td>0,5</td>
<td>Best</td>
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<td>72</td>
</tr>
<tr>
<td>103</td>
<td>CD</td>
<td>GRR</td>
<td>1,3</td>
<td>0,3</td>
<td>Worst</td>
<td>33</td>
<td>20</td>
</tr>
<tr>
<td>103</td>
<td>MD</td>
<td>GRR</td>
<td>1,7</td>
<td>0,6</td>
<td>Best</td>
<td>85</td>
<td>94</td>
</tr>
<tr>
<td>103</td>
<td>MD</td>
<td>GRR</td>
<td>1,0</td>
<td>0,3</td>
<td>Worst</td>
<td>8</td>
<td>8</td>
</tr>
<tr>
<td>103</td>
<td>CD</td>
<td>GGR</td>
<td>1,0</td>
<td>0,5</td>
<td>Best</td>
<td>73</td>
<td>38</td>
</tr>
<tr>
<td>103</td>
<td>CD</td>
<td>GGR</td>
<td>0,8</td>
<td>0,5</td>
<td>Worst</td>
<td>13</td>
<td>8</td>
</tr>
<tr>
<td>103</td>
<td>MD</td>
<td>GGR</td>
<td>1,7</td>
<td>0,6</td>
<td>Best</td>
<td>60</td>
<td>34</td>
</tr>
<tr>
<td>103</td>
<td>MD</td>
<td>GGR</td>
<td>1,0</td>
<td>0,4</td>
<td>Worst</td>
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<td>120</td>
</tr>
<tr>
<td>PE</td>
<td>CD</td>
<td>NA</td>
<td>1,0</td>
<td>0,5</td>
<td>Best</td>
<td>1800</td>
<td>0</td>
</tr>
<tr>
<td>PE</td>
<td>CD</td>
<td>NA</td>
<td>0,8</td>
<td>0,5</td>
<td>Worst</td>
<td>125</td>
<td>177</td>
</tr>
<tr>
<td>PE</td>
<td>MD</td>
<td>NA</td>
<td>1,0</td>
<td>0,5</td>
<td>Best</td>
<td>1800</td>
<td>0</td>
</tr>
<tr>
<td>PE</td>
<td>MD</td>
<td>NA</td>
<td>0,8</td>
<td>0,5</td>
<td>Worst</td>
<td>13</td>
<td>8</td>
</tr>
<tr>
<td>PVOH</td>
<td>CD</td>
<td>NA</td>
<td>1,0</td>
<td>0,5</td>
<td>Best</td>
<td>840</td>
<td>917</td>
</tr>
<tr>
<td>PVOH</td>
<td>CD</td>
<td>NA</td>
<td>0,8</td>
<td>0,5</td>
<td>Worst</td>
<td>108</td>
<td>93</td>
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<tr>
<td>PVOH</td>
<td>MD</td>
<td>NA</td>
<td>1,0</td>
<td>0,5</td>
<td>Best</td>
<td>1265</td>
<td>1157</td>
</tr>
<tr>
<td>PVOH</td>
<td>MD</td>
<td>NA</td>
<td>0,8</td>
<td>0,5</td>
<td>Worst</td>
<td>273</td>
<td>458</td>
</tr>
</tbody>
</table>

Table 2. The grease resistance test results for the barrier coated paperboard which has been creased.
Table 3. The grease resistance test results for the barrier coated paper which has been creased and folded.

<table>
<thead>
<tr>
<th>Material</th>
<th>MD/CD</th>
<th>GRR/GGR</th>
<th>Groove width (mm)</th>
<th>Matrix thickness (mm)</th>
<th>Best/Worst</th>
<th>Time (s)</th>
<th>Conf. T</th>
</tr>
</thead>
<tbody>
<tr>
<td>Paper CD</td>
<td>GRR</td>
<td>2,0</td>
<td>0,7</td>
<td>Best</td>
<td>20</td>
<td>18</td>
<td></td>
</tr>
<tr>
<td>Paper CD</td>
<td>GRR</td>
<td>1,5</td>
<td>0,6</td>
<td>Worst</td>
<td>23</td>
<td>20</td>
<td></td>
</tr>
<tr>
<td>Paper MD</td>
<td>GRR</td>
<td>2,1</td>
<td>0,9</td>
<td>Best</td>
<td>55</td>
<td>133</td>
<td></td>
</tr>
<tr>
<td>Paper MD</td>
<td>GRR</td>
<td>1,4</td>
<td>0,6</td>
<td>Worst</td>
<td>13</td>
<td>8</td>
<td></td>
</tr>
<tr>
<td>Paper CD</td>
<td>GGR</td>
<td>2,0</td>
<td>0,7</td>
<td>Best</td>
<td>50</td>
<td>23</td>
<td></td>
</tr>
<tr>
<td>Paper CD</td>
<td>GGR</td>
<td>1,7</td>
<td>0,5</td>
<td>Worst</td>
<td>33</td>
<td>35</td>
<td></td>
</tr>
<tr>
<td>Paper MD</td>
<td>GGR</td>
<td>2,0</td>
<td>0,7</td>
<td>Best</td>
<td>25</td>
<td>38</td>
<td></td>
</tr>
<tr>
<td>Paper MD</td>
<td>GGR</td>
<td>2,4</td>
<td>1,0</td>
<td>Worst</td>
<td>8</td>
<td>15</td>
<td></td>
</tr>
<tr>
<td>PE CD</td>
<td>NA</td>
<td>2,0</td>
<td>0,7</td>
<td>Best</td>
<td>1800</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>PE CD</td>
<td>NA</td>
<td>1,7</td>
<td>0,5</td>
<td>Worst</td>
<td>1380</td>
<td>1337</td>
<td></td>
</tr>
<tr>
<td>PE MD</td>
<td>NA</td>
<td>2,0</td>
<td>0,7</td>
<td>Best</td>
<td>1800</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>PE MD</td>
<td>NA</td>
<td>2,4</td>
<td>1,0</td>
<td>Worst</td>
<td>1800</td>
<td>0</td>
<td></td>
</tr>
</tbody>
</table>

The grease resistance test of the creased and folded packaging paper, see table 3, showed similar results as the creased paperboard. The only samples that showed passed the modified TAPPI 454 test was the PE coated samples. Also here, the best results were obtained when creasing and folding in MD compared to CD. The hemicellulose based barrier coated paper showed an almost imidiate oil penetration.
3.4 Water vapour transmission rate

A summary of the results of the WVTR measurements is presented in table 4. The full data set can be found in Appendix V. The creased and folded samples showed a slightly higher WVTR than the uncreased and unfolded references. The creased board sample with PE was creased in CD with a matrix thickness of 0.5 mm and a groove width of 1.0 mm. The folded PE coated paper was first creased in MD with a matrix thickness of 0.7 mm and a groove width of 2.0 mm before folding.

Table 4. Water vapour transmission results of the creased/folded paperboard/paper and their references.

<table>
<thead>
<tr>
<th>Test</th>
<th>WVTR (g/cm(^3)*day)</th>
<th>Std.dev</th>
</tr>
</thead>
<tbody>
<tr>
<td>Referance paperboard</td>
<td>3.3</td>
<td>0.0</td>
</tr>
<tr>
<td>Creased paperboard</td>
<td>6.0</td>
<td>2.8</td>
</tr>
<tr>
<td>Referance paper</td>
<td>3.3</td>
<td>0.9</td>
</tr>
<tr>
<td>Folded paper</td>
<td>5.3</td>
<td>0.9</td>
</tr>
</tbody>
</table>
3.5 Tensile testing

The results for the tensile testing, table 5, showed that for the extruded PE was unisotropic; the elastic modulus maximum stress and elongation at break was larger in MD than in CD. The elastic modulus increased with fibers on the PE while the elongation at break decreased with fibers. This was evident in both directions. Data for tensile properties for PVOH was obtained from Javed, A. (2016). Both heat treated (cured) and non-heat treated PVOH was measured by Javed et al. (2016) and both showed large elongation at break, a high elastic modulus and a high maximum stress. The hemicellulose based barrier film had the lowest elongation at break, i.e only 2%, the highest elastic modulus and a maximum stress between the maximum stresses of PE and PVOH. The complete data for the testing can be found in Appendix VI.

*Table 5. Results for the tensile testing for three different barrier materials.*

<table>
<thead>
<tr>
<th>Thickness (μm)</th>
<th>PE1 (MD)</th>
<th>PE1 (CD)</th>
<th>PE2 (MD)</th>
<th>PE2 (CD)</th>
<th>PE3 (MD)</th>
<th>PE3 (CD)</th>
<th>PE4 (MD)</th>
<th>PE4 (CD)</th>
<th>PVOH (Javed et al. 2016)</th>
<th>PVOH Cured (Javed et al. 2016)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conf T</td>
<td>1,3</td>
<td>1,5</td>
<td>1,1</td>
<td>2,0</td>
<td>2,3</td>
<td>NA</td>
<td>NA</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Basis weight (g/m²)</td>
<td>32,9</td>
<td>24,0</td>
<td>20,0</td>
<td>12,9</td>
<td>160,4</td>
<td>NA</td>
<td>NA</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Conf T</td>
<td>0,5</td>
<td>4,0</td>
<td>2,7</td>
<td>1,8</td>
<td>5,5</td>
<td>NA</td>
<td>NA</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Elongation at break(%)</td>
<td>3,0</td>
<td>4,8</td>
<td>60,1</td>
<td>41,8</td>
<td>155,9</td>
<td>194,4</td>
<td>59,0</td>
<td>50,8</td>
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<td>279,0</td>
</tr>
<tr>
<td>Conf T</td>
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<td>0,3</td>
<td>4,7</td>
<td>7,9</td>
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<td>69,9</td>
<td>10,2</td>
<td>8,4</td>
<td>0,3</td>
<td>20,0</td>
</tr>
<tr>
<td>Maximum stress (MPa)</td>
<td>7,1</td>
<td>5,7</td>
<td>5,0</td>
<td>3,9</td>
<td>7,8</td>
<td>6,8</td>
<td>7,7</td>
<td>5,8</td>
<td>16,9</td>
<td>54,9</td>
</tr>
<tr>
<td>Conf T</td>
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<td>0,2</td>
<td>0,3</td>
<td>0,3</td>
<td>0,4</td>
<td>0,5</td>
<td>0,2</td>
<td>0,8</td>
<td>3,5</td>
<td>4,6</td>
</tr>
<tr>
<td>Elastic modulus (Mpa)</td>
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<td>257,2</td>
<td>83,9</td>
<td>79,3</td>
<td>77,4</td>
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<td>82,4</td>
<td>75,3</td>
<td>2046,6</td>
<td>911,0</td>
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<tr>
<td>Conf T</td>
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<td>24,3</td>
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<td>14,8</td>
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<td>4,0</td>
<td>5,4</td>
<td>61,4</td>
<td>38,0</td>
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</table>
3.6 SEM pictures

In this section pictures are scanning electron microscope (SEM) pictures are taken of the creased and creased/folded paper and paperboard with different barrier coatings. This pictures had a magnification of 100x, 250x, 500x and 1000x.

Figure 20. SEM pictures taken of a creased paperboard using a matrix thickness of 0.3 mm and a groove width of 1.3 mm. The board had been coated with hemicellulose based coating. Arrows indicates cracks. The magnifications from left to right are 100x, 250x and 500x.

Figure 21. SEM pictures taken of a creased paperboard using a matrix thickness of 0.5 mm and a groove width of 1.0 mm. The board had been coated with hemicellulose coating. Arrows indicates cracks. The magnifications from left to right are 100x, 250x and 500x.

In figure 20-21, cracks in the crease and along the crease are shown on the paperboard with the hemicellulose based barrier. In figure 20 where 0.3x1.3 (matrix thickness x groove width) showed larger and more severe cracks visually where material seems to have been removed in the crack compared to the cracks in figure 21. In figure 21, the cracks seems to be smaller without as much material being removed around the cracks.
Figures 22 and 23 shows SEM images of creased samples of PE coated paperboard. The pictures show some kind of defect in the paperboard which has been creased with the matrix thickness of 0,5 mm and groove width of 0,8 mm. If the origin of this defect is from the creasing operation or if it’s just a small pinhole in the barrier layer can not be concluded from these images. The picture regarding matrix thickness of 0,5 mm and a groove width of 1,0 mm showed no cracks or defects.
Figure 24. SEM pictures taken of a creased PVOH coated paperboard using a matrix thickness of 0.5 and a groove width of 0.8. Arrows indicates cracks. The matrix thickness was 0.5 mm and the groove width was 0.8mm. Arrows indicates cracks. The magnifications were 100 and 500x at the top and 500 and 550x in the bottom.

Figure 25. SEM pictures taken of a creased paperboard which are coated with PVOH at a magnification of 100x. The matrix thickness was 0.5 mm and the groove width was 1.0 mm.

Figures 24 and 25 shows creased PVOH coated paperboard. In the upper figure a small defect appears. It is hard to determine from these images of the defect originates from the creasing operation or if the defect are was created during the coating operation, e.g by trapped air bubbles. The image in Figure 25 showed no evidence of cracks but a tendency of pin-holes or trapped air bubbles.
Figure 26. SEM pictures taken of a creased and folded paper which are coated with hemicellulose based barrier. The samples were creased with a matrix thickness of 0.7 mm and a groove width of 2.0 mm. The arrows indicate a crack. The magnification was 100x for the left image and 500x for the right image.

Figure 27. SEM images of creased and folded paper coated with hemicellulose based barrier coating, creased with a matrix thickness of 1.0 and a groove width of 2.4 mm. The magnifications from left to right are 50x, 100x and 500x.

Figures 26 and 27 above shows SEM images of crease and folded paper with the hemicellulose based barrier coating. Both samples showed cracks within the barrier coating layer.

Figure 28. SEM images of a creased and folded PE coated packaging paper using a matrix thickness of 0.5 mm and a groove width of 1.7 mm. The magnification of the image of the left was 50x and the image on the right 100x.
Figure 29. SEM pictures taken of a creased and folded PE coated paper using a matrix thickness of 0.7 mm and a groove width of 2.0 mm. The magnification was 50x.

The surface of creased and folded PE coated packaging paper is shown in figures 28 and 29. None of these samples showed any evidence of cracks or failure.
4. Discussion

The purpose of this work was to propose a screening methodology to determine how barrier coated products withstand creasing. Failure of the barrier layer might result in cracks that will compromise the barrier properties.

The first thought was to use pilot coated material with the hemicellulose based barrier coating. This proved to be more difficult than expected since the applied amount of the pilot coating was rather low, i.e. a few grams. Samples were therefore laboratory coated. However, the first creasing tests showed cracking and the folded paper looked wrinkled. The grease test and the SEM analysis also showed that all of the hemicellulose based barrier coated samples had cracks in them leading to leakage of oil during the grease test. This rescheduled the master thesis to involve conventional barriers as PE and PVOH. Even if the PVOH did not show cracking, creasing caused oil leakage during the grease test. Considering that all uncreased samples passed the grease tests, this is an illustration of how problematic it is to obtain a barrier that would sustain converting operations. The original thought was to use measurements of oxygen transmission rate, OTR, at the end to determine the impact of failure on the barrier properties, but it was not considered meaningful since the hemicellulose based barrier and the PVOH coating did not withstand the grease test. It is therefore not likely that any of them would pass an OTR measurement.

PE proved to endure the grease resistance test. With PE the theory that cracks in the barrier coating could be predicted using a grease resistance test could be validated. The crease and folded paper and paperboard samples underwent a WVTR and showed a small increase in transmission rate, indicating a mildly compromised barrier due to creasing. WVTR was chosen over OTR since PE is an excellent water barrier but a rather poor oxygen barrier. These measurements could thus be used to confirm the methodology.

The results regarding the creasing of paperboard (test and validation) and creasing/folding of paper (screening) indicates that the boundary for MD creasing is larger than a CD-crease. The reason for this can be due to that a MD crease which is parallel to the MD has less fibers to “deform” and the layers are easier to delaminate. The CD crease is more severe due to that crease is done perpendicular to the MD where there are more fibers compared to that in CD. This boundary is solely dependent of how the mechanical properties are in MD respective CD of the subject which are creased.
When visually inspecting the paperboard which has been creased and the paper which has been creased and folded and comparing their respective cracks formations a conclusion can be made. The crack formations differs between the creased/folded paper and the creased paperboard see figure 20 & 26, this is due to different loads from the creasing.

The different loads is due to that the paper was creased the coating faced the creasing rule and when the paperboard was creased the coating faced down towards the matrix.

Why a bad and good crease sample was subjected for a grease resistance test was to show that there is a boundary where the barrier coating cracks and not. This can be seen for example for the paperboards PE samples where the creased good samples endured the grease test and the bad samples didn’t. For this material, PE of the paperboard, has a critical boundary of where the barrier fails and this should be somewhere in between those samples. These critical boundaries for respective material should show a larger boundary in MD-crease than in CD due to different mechanical properties in MD and CD, same argument as for the validation/test of the creasing.

Why a critical boundary was not found for the other barrier coatings for the paperboard are due to that the other barrier coatings couldn’t endure the stresses and strains applied on the surface during the creasing operation. This problem has its origin within the mechanical behaviour of the coatings and the thickness of the coatings. The hemicellulose based barrier showed a low ductility with a low strain at break and this resulted in cracking during creasing and folding. These cracks were also visible both in light microscopy and in SEM images. The tensile results for the PVOH (Javed et al. 2016) were from casted films compared to the PVOH coatings applied in a pilot coater. Even if cracks were not visible in the microscopic investigations, the grease tests revealed failure in the coating compromising the barrier properties. The oil penetration only took a little longer time than for the creased hemicellulose based coating. However, the tensile results showed however, that PVOH was a more ductile material than PE. The reason for that the PVOH failure might be due to two reasons. The first is that the pilot coated PVOH is less ductile than the casted coated PVOH in the study of Javed et al (2016) causing failure during the creasing operation. The second possibility is that the rather thin PVOH layer, 12 g/m² compared to 20 g/m² for PE, did not withstand the stresses and strains imposed on the layer during creasing and folding.

The creased PVOH paperboard had samples which didn’t show any cracks visually but did get penetrated during the crease resistance test. In figure 22 shows that the PVOH samples had some kind of defect which could be caused by trapped air. These defects might cause failure during the creasing operation leading to oil penetration.
The results for the WVTR shows a difference between the references and the creased/folded samples, the creased/folded samples shows a slightly higher value. This could be explained by that during the creasing and folding the barrier coating is exposed to strains which stretches the barrier coating which makes it thinner. A thinner the barrier coating results in a higher the permeation, due to a shorter diffusion distance for the molecules (in this example water vapour) through the barrier coating. If the creased/folded samples would have cracks in the barrier coating the diffusing molecules would have crossed the barrier and had given rise to a significant higher WVTR value. This would also most probably have been seen in the grease resistance test.

4.1 Suggested method
The suggested method to discover cracks in creased/folded barrier coated paperboard/paper was proposed here. This suggested method was based on the results presented above.

Four samples that are creased or creased/folded, the samples are investigated visually using a light microscope. If no cracks are visible, a grease resistance test will determine if the barrier coated paperboard/paper. If the creased or creased/folded samples Endures the grease resistance test the barrier is intact and does not have any cracks. The grease resistance test could be performed as described in TAPPI454 or a similar adjustment which has been done in this master thesis.

4.2 Possible sources of error
Due to that the direction of the rod coating of the hemi dispersion is made perpendicular to machine direction might affect the results. In the industry is the coating direction in the same way as the machine direction. Even if the magnitude of error of this is likely to be of minor importance in a laboratory coating operation, it needs to be considered.

A possible error source is the inhomogeneity of the particle size of the sand used in the grease resistance test. When performing the tests, the smaller sand grains accumulated in the bottom of the aluminum foils which the sand were dried in. The sands particle size will influence the time of how the oil passes through it and reaches the surface of the barrier. The smaller the particles are the longer it takes for the oil to pass through it. The sands purpose in the test is just to prevent the oil to come all over the sample and pollutes the backside of the sample. Even if the time of how the oil passes through the sand depending of the particle size, this does not matter for the hemicellulose based barrier due to the immediate penetration through the barrier, but for samples like the PVOH it can cause big errors when it took the oil approximately 10-15min to penetrate the barrier. A factor which reminds of the sand particle size issue is also that when preparing the coloured oil, where there were undissolved colour particles. These particles did settle but when preparing some samples some undissolved particles pipetted and this oil took longer to be absorbed into the sand than oil with undissolved colour particles.
This problem can be solved by filtering the coloured oil after preparing it. Further possible error sources with the grease resistance test is the determination of when the barrier coating is penetrated. This is a problem due to that in some cases it was hard to see when the oil penetrated. This is just a major concern of the method as such and due to the simplicity of the method itself. The only thing which could be said with the grease resistance test is if the sample gets penetrated or not.

A possible error during the casting of the hemibarrier based films is that the dried hemicellulose based films showed a phase separation (the two phases has different colours, see figure#) and this will most probably influence the tensile testing. During the tensile testing all of the samples did break in the same phase (the lighter phase) which concludes that the tensile testing of the hemicellulose based films just give tensile properties for the lighter phase and the darker phase has different mechanical properties. The darker phase is probably stronger than the lighter phase due to that the lighter phase breaks first.

4.3 Further work
The hemicellulose based barrier coating is very hydrophilic and when applied your thumb against the coated surface the hemicellulose coating started to dissolve due to the moisture from the fingers. When performing the experimental the climate was according to ISO187:1990, which was 23 °C and 50 % RH, this will influence the highly hydrophilic coating as well. One thing that would be of interest for the manufacturers of the hemicellulose based dispersion is to see how the hemicellulose based coating behaves when a barrier layer such as PE is applied on the hemicellulose based coating.

A thing which there was not time for during the master thesis, was to try the method on a PVOH coated board with a higher grammage (20 g/m²). An OTR analyzis would be of most interest if the samples passes the grease resistance test. Would the OTR measurements deviate and how much compared to an uncreased sample. The measured values would probably deviate, but comparing the magnitude of deviation from the OTR and the PE measurments would perhaps raise new questions.

A further development of the methodology could be verification by using more materials. The method could be improved with a camera connected to a computer with a software that indicates when the oil penetrates the sample in the grease resistance test. An easier way to indicate cracks visually instead of using a microscope could be to use a marker pen with a soft tip. The colour of the marker pen would maybe leave a spot which indicates a crack and this would perhaps be a quicker way to identify cracks than using light microscopy.
This methodology could be used to screen new barrier materials. For instance could new materials from development projects be coated onto a board. The method could be used to see if the material endures the strains caused by creasing. Whether the sample endures the grease resistance, decisions can be made on how to continue the work. If the material did not pass the screening, focus could be on to improve the materials ductility.

A continued development of this methodology could be to build a library of creased barrier coatings. Here data about the barrier coating including the results from the proposed methodology could be stored. The gathered data could be analyzed to find patterns and relationships to get a better understanding about creasing of barrier coatings.
5. Conclusion

Since all samples passed the grease test before creasing and folding, it is safe to conclude that the reason for failure of barrier properties here are due to failure during creasing and folding.

The proposed method to predict the failure in barrier coated paperboard and paper after creasing and folding has been validated by the PE extruded paper and paperboard. A visual inspection in a light microscope of the creased or creased/folded material distinguishes quickly if a barrier coating has cracks or not. This visual inspection worked well and with all the subjects which had a barrier coating with hemicellulose based coating showed cracks already in the microscopy which was confirmed by the grease resistance test. In case that there are doubts of visual cracks a grease resistance test could be used to verify this. PVOH illustrates this quite well. Even if cracks were not found visibly in the PVOH, the oil penetrated the barrier layer after creasing. The only material which could resist penetration from the oil after creasing were a few samples of PE. To see if the method works and that PE has no cracks WVTR was performed to validate the method and the WVTR validates the method.
Acknowledgements

During this master thesis have I come in contact with a lot of people that should be thanked for their involvement. First of all I want to thank Gunnar Forsgren and Peter Rättö which are my supervisors for a superb guidance and support during the project. I want to say thanks to all of the employees at Holmen AB, Iggesund Paperboard which I have come in contact with during my visits there. I want to especially say thanks to Esko Pakarinen and Mikael Thorell for good company during the laboratory part, Brita Timmermann and Lena Blomström for showing and instructing me with the creasing machine. I want to say thanks to Birgitta Gustafsson and Asif Javed at Karlstad’s University for their respective assistance. Also want to say an extra thanks to Peter Rättö which have taken SEM pictures on some of the samples and to Kristina Junel at RISE for helpful information.
References

Literature


Standards


TAPPI454 – Turpentine test for voids in glassine and greaseproof papers.
## Table 6. Results for the visual inspection of the packaging board and the paper.

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<td>Substrate type</td>
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Appendix II

Figure 30. Scheme of creasing in CD and MD using a non-barrier coated Invercote G220.
Appendix III
Table 7. The grease resistance test results for the packaging board with different barrier coatings.

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Table 8. The grease resistance test results for the paper with different barrier coatings.

<table>
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<tr>
<th>Matrix thickness(mm)</th>
<th>Groove width(mm)</th>
<th>Material</th>
<th>MD/CD</th>
<th>GRR/GGR</th>
<th>Best/Worst</th>
<th>#</th>
<th>Time(s)</th>
<th>Average</th>
<th>Std. Dev</th>
<th>Conf.T</th>
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<td>Groove width(mm)</td>
<td>Material</td>
<td>MD/CD</td>
<td>GRR/GGR</td>
<td>Best/Worst</td>
<td>#</td>
<td>Time(s)</td>
<td>Average</td>
<td>Std. Dev</td>
<td>Conf.T</td>
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</table>
Appendix IV

Figure 31. Schemes of the data sets which were tested during the packaging board and paper with their respective barrier.
## Appendix V

### Table 9. The weight and corresponding elapsed time during the WVTR test for the references, creased and folded PE samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Time(h)</th>
<th>Weight(g)</th>
<th>Time(h)</th>
<th>Weight(g)</th>
<th>Time(h)</th>
<th>Weight(g)</th>
<th>Time(h)</th>
<th>Weight(g)</th>
<th>Time(h)</th>
<th>Weight(g)</th>
<th>Time(h)</th>
<th>Weight(g)</th>
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<td>172,138</td>
<td>48</td>
<td>172,14</td>
<td>72</td>
<td>172,143</td>
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<td>171,942</td>
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<td>179,14</td>
<td>72</td>
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</table>

### Table 10. The calculated slope and WVTR-values for the references, creased and folded PE samples.

<table>
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<th>Sample</th>
<th>Slope</th>
<th>WVTR(g/cc*day)</th>
<th>Average</th>
<th>Range</th>
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</thead>
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<td>3,33</td>
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<td>2,828</td>
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<tr>
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<td>4</td>
<td>6</td>
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<tr>
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<td>4,66</td>
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<td>5,33</td>
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Appendix VI

Table 11. The thickness measurements of the casted hemi film and the different PE’s.

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<th>Hemi</th>
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<th>PE2</th>
<th>PE3</th>
<th>PE4</th>
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<td>28,0</td>
<td>17,0</td>
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<tr>
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<td>47,3</td>
<td>42,0</td>
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<td>45,0</td>
<td>27,0</td>
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<td>28,0</td>
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<td>42,2</td>
<td>28,6</td>
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Table 12. The weight measurements and grammage calculations of the casted hemi film and the different PE’s.

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<th>PE2</th>
<th>PE3</th>
<th>PE4</th>
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<td>Grammage (g/m2)</td>
<td>Weight (mg)</td>
<td>Grammage (g/m2)</td>
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Table 13. The calculated and measured tensile properties of the casted hemi film.

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<th>Elastic modulus MPa</th>
<th>Maximum stress N/m</th>
<th>Elastic modulus MPa</th>
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Table 14. The calculated and measured tensile properties of PE1 in MD.

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<th>Maximum stress</th>
<th>Elastic modulus</th>
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| Std.dev | 0.6 | 36.0 | 56.0 | 0.8 | 117.9 |
| Conf.T  | 0.3 | 17.3 | 27.0 | 0.4 | 56.8  |
Table 15. The calculated and measured tensile properties of PE1 in CD.

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<th>Elastic modulus</th>
<th>Maximum stress</th>
<th>Elastic modulus</th>
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Std.dev: 5,1, 13,0, 5,6, 0,3, 13,2
Conf.T: 4,7, 12,0, 5,1, 0,3, 12,2

Table 16. The calculated and measured tensile properties of PE2 in MD.

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Std.dev: 5,1, 13,0, 5,6, 0,3, 13,2
Conf.T: 4,7, 12,0, 5,1, 0,3, 12,2
Table 17. The calculated and measured tensile properties of PE2 in CD.

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Table 18. The calculated and measured tensile properties of PE3 in MD.

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Table 19. The calculated and measured tensile properties of PE3 in CD.

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<th>Elastic modulus</th>
<th>Maximum stress</th>
<th>Elastic modulus</th>
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Table 20. The calculated and measured tensile properties of PE4 in MD.

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<th>Elastic modulus</th>
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<th>Elastic modulus</th>
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Table 21. The calculated and measured tensile properties of PE4 in CD.

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