In-plane Wetting in Packaging Material and its Consequences for Cap Performance

CENTRE FOR ANALYSIS AND SYNTHESIS, ENGINEERING NANOSCIENCE | LUND UNIVERSITY
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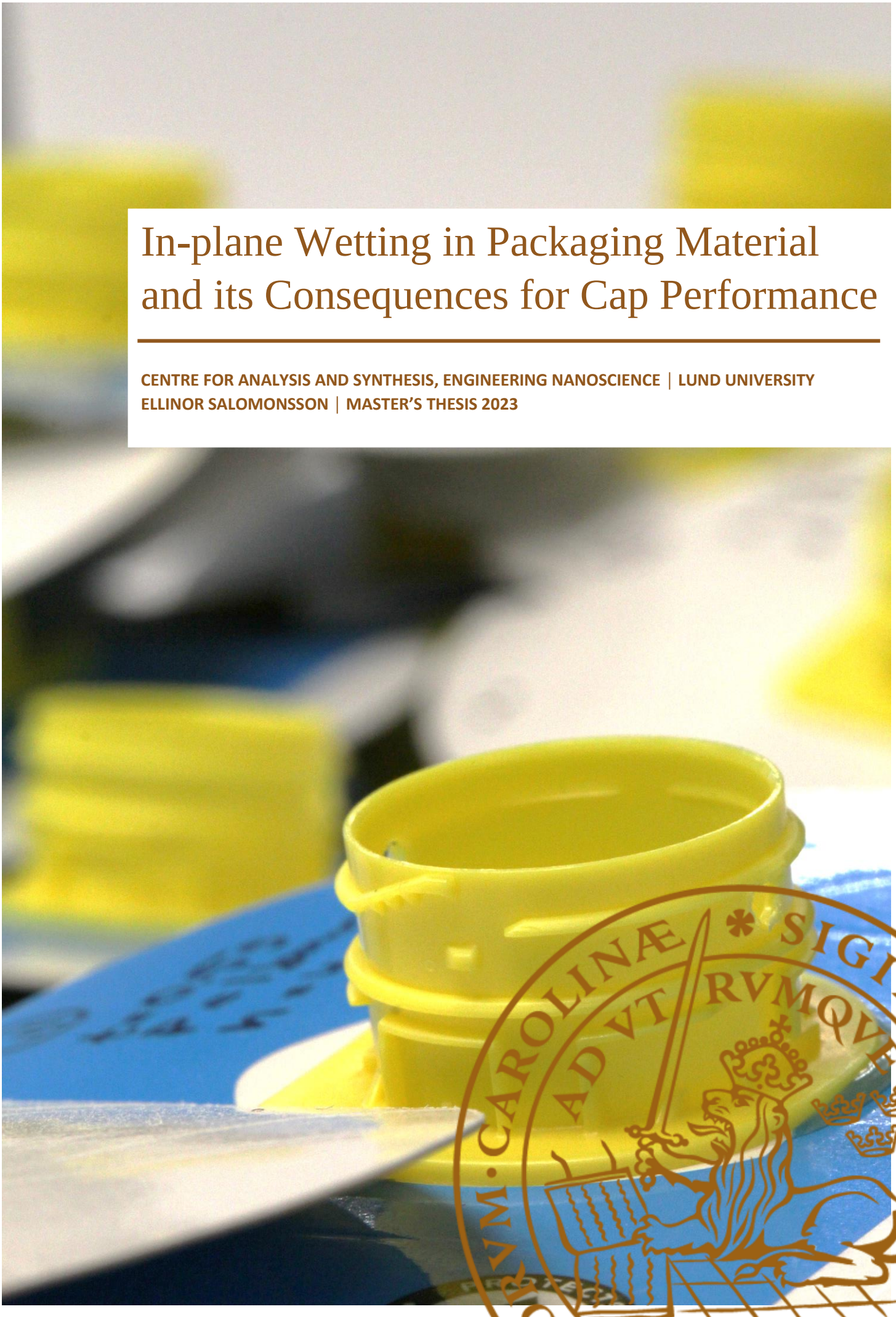

In-plane Wetting in Packaging Material and its Consequences for Cap Performance

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Abstract

Aseptic packaging solutions enable long time storage without refrigeration and are an important part of securing food supply globally. The package material consists of multiple layers: A paperboard for robustness, polymers for adhesion as well as shielding the board from liquid, and a barrier protecting the product.

This thesis consists of two parts regarding in-plane wetting into the paperboard. The first part is investigating the current test method evaluating in-plane wetting by the raw edges due to the aseptic hydrogen peroxide bath during production. In the aseptic process of the filling machine, packaging material is exposed to sterilizing hydrogen peroxide which can penetrate the raw edges of the material where the paperboard is exposed. If this is extensive enough, breakage of the material can lead to the need to halt production for cleaning the machines. Later in the filling process, residual water can also cause soaking of the material by the edges or where the cap is to be attached (around the pre-laminated hole) if defects are present in the décor layer. The second part of the thesis aims to investigate a possible correlation between the amount of in-plane wetting around the pre-laminated hole and the amount of force needed for cap detachment.

For the investigation regarding in-plane wetting around the raw edges due to the hydrogen peroxide bath, promising results that eventually might lead to a chemical exchange were obtained and observations regarding how the existing test set-up could be optimized were made. For the second part of the thesis, the results indicate that there exists a correlation between the amount of in-plane wetting around the pre-laminated hole and cap detachment force when measuring on material with board still soaked. The force needed decreases for increasing amount of wetting which somewhat correlates with the expectations. For material that had been dried before cap application, no clear trends were found.

Keywords: Tetra Pak®, Aseptic, Paperboard, Pre-laminated hole, Edge wicking, In-plane wetting, Hydrogen peroxide, Cap detachment

Sammanfattning

Aseptiska förpackningslösningar möjliggör långtidsförvaring i rumstemperatur och är en viktig komponent för att säkra livsmedelstillgången värden över. Förpackningsmaterial består av flera lager: Ett kartonglager för robusthet, polymerer för fästförmåga mellan lagren samt för att skydda kartongen mot vätska och en barriär som skyddar produkten.

Det här masterarbetet består av två delar som båda är relaterade till vätskeinträngning i kartongen. Den ena delen undersöker den existerande testmetoden för att mäta vätskeinträngning på grund av det aseptiska väteperoxidbadet. I den aseptiska tillverkningsprocessen i fyllmaskinen exponeras förpackningsmaterialet för steriliserande väteperoxid som kan leda till vätskeinträngning i kanterna av materialet där kartongen är exponerad. Även senare i processen kan överblivet vatten på ytan leda till absorbtion vid kanterna av materialet eller där korken senare ska fästas (runt det förlaminerade hålet) om det finns defekter i dekorlagret. Den andra delen av arbetet handlar om att undersöka om det finns ett samband mellan mängden vätskeinträngning runt det förlaminerade hålet och hur stor kraft som behövs för att rycka loss korken från materialet.

För studien gällande vätskeinträngning på grund av väteperoxidbadet i fyllmaskinen, uppnåddes lovande resultat som längre fram skulle kunna leda till ett byte av kemikalie i testmetoden och observationer gällande hur den existerande testmetoden kan optimeras kunde göras. Resultaten för den andra delen av arbetet visar på att det finns ett samband mellan mängden vätskeinträngning runt det förlaminerade hålet och hur stor kraft som behöver användas för att rycka loss korken för material som fortfarande har blöt kartong. Kraften som behövs minskar drastiskt för ökande mängd vätskeinträngning, vilket till viss del stämmer överens med förväntat resultat. För material som torkat innan korkappliceringen kunde inga lika tydliga trender observeras.

Nyckelord: Tetra Pak®, Aseptisk, Kartong, Förlaminerat hål, Vätskeinträngning, Väteperoxid, Korklossning

Acknowledgements

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First, I want to thank Tommy Garting for being my supervisor at Tetra Pak®. Thank you for giving me the freedom and time to tackle this project in my own way, but always being available when I needed you. Regardless of how lost I felt before discussing something with you, it always felt like nothing was impossible afterwards.

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Aron Molnar, thank you for lending me your time and helping me realize the fixtures with your CAD skills. The brain storming session we had together with Tommy when the first edition fixture didn't work out is one of my favourite memories from this spring.

I would also like to thank Lena Hansson. Not only have you been a great support during this semester, but you also took me under your wings from the start in 2020. You are a huge reason for me feeling at home at Tetra Pak®.

A big thank you to Malin Isgren for inviting me to your team in 2020 and for continuing to let me be a part of it during this master thesis.

I want to thank Cecilia Bäckman for spending hours teaching me how to perform the test method for edge wicking, Hanna Markström for helping me locate caps for the project, and Ingrid Isacsson for always being available when I needed a coffee break in the sun as well as helping me with the cover page.

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I never would have made it to the master thesis to begin with if it weren't for my family. Thank you for all the support, and a special thank you to my dad for early on letting me know that paper packaging is the most interesting line of work there is.

And now to my second family. Thank you for these incredible five years and for being the best friends one could ever hope for.

Finally, to my husband Henric. Thank you for standing by my side, always.

Contents

List of Abbreviations

AKD – Alkyl ketene dimer

CD – Cross direction

DoE – Design of experiments

ES – Edge soaking

EW – Edge wicking

GSM – Grams per square meter

HP – Hydrogen peroxide

MD – Machine direction

mN – Millinewton

PE – Polyethene

PLH – Pre laminated hole

PM – Packaging material

PPH – Pre punctured hole

TM – Test method

1. Background

To avoid the need for refrigerator storage and to increase the shelf life of a product, aseptic packaging methods were developed. When dealing with aseptic packaging, penetration of liquid into the paperboard might occur during the sterilization process or later in the form of residual water on the material during filling. At Tetra Pak® one talks about two different scenarios: edge wicking (EW) when the in-plane wetting occurs by the exposed edges of the packaging material, and edge soaking (ES) when the in-plane wetting occurs by the pre laminated hole (PLH) where the cap is to be attached.

The current test method (TM) for evaluating packaging material (PM) for EW includes a hydrogen peroxide (HP) bath which Tetra Pak® would want to replace with something else, since HP is a hazardous chemical in many ways. At the same time, there are doubts regarding if it really is necessary to evaluate EW and if so, is HP the right way to do it at all? A part of this project will therefore focus on the actual need for the existing EW TM using HP.

The second, and main part of this thesis, focuses on ES and whether there is an increased risk of cap detachment for packages where large degree of ES has occurred during filling machine interaction. Customers have expressed a concern regarding the possibility of cap detachment for high degrees of ES and that this could create a functionality problem. A method for studying this possible correlation needs to be designed and the force needed for cap detachment should be measured for packaging material exposed to various amounts of ES prior to cap attachment. The study is performed on flat PM since it previously has been shown difficult to produce packages with varying degree of ES in a controlled way.

2. Objectives

The current test method for measuring EW in the paperboard uses an HP bath and the reason for this is somewhat unclear. HP is a dangerous chemical, and Tetra Pak® would like to replace this with a more lenient one if possible.

- Part of the study focuses on understanding what the purpose of using HP to induce EW is and if there is an actual need to perform the measurements this way.
- Depending on the outcome of the previous objective, aim to find a suitable replacement chemical for HP.
- Provided that the TM is still relevant, aim to gain knowledge about potential time, temperature, and concentration dependency of the method.

It is also of interest to define threshold values for what degrees of ES around the PLH that can be accepted with no risk of cap detachment.

- An experimental set-up for measuring the force needed to detach the cap from flat PM using a tensile tester needs to be designed.
- A correlation between the degree of ES and the force needed to detach the cap should be determined if such a correlation exists.
- A variety of different kinds of PMs should be examined to find possible differences in this correlation, e.g., bleached, and unbleached paperboard.

3. Theory

3.1 Aseptic packaging material and production

Aseptic packaging requires sterilization of both the package and the product to be stored prior to filling, and this enables storage at room temperature for long periods of time without additives in the product [1]. Tetra Pak® performed its first successful aseptic filling process for carton packages as early as 1961 [2], and today aseptic packaging still is a very important component to be able to fulfill the company purpose which is "We commit to making food safe and available, everywhere and we promise to protect what's good: food, people and the planet." [3]. In 1961, the aseptic filling system utilized a combination of HP and heat for sterilization [2], and a similar process is used today.

3.1.1 Filling machine

In the current processing systems, the PM goes through a 30% HP bath heated to 70 ºC prior to forming it into a tube and eventually the the final shape of the package, see Figure 1. The material is kept in the HP bath for as long as needed to become sterile and is then dried using hot air or pressure rollers [1]. The process just described is used for roll fed production, which is when a reel of PM is supplied and goes through the filling machine as a web, is folded into a tube that is filled with product, which then is sealed below the liquid line to minimize foaming, and then cut up into separate packages. Tetra Brik® aseptic, which is a popular and commonly used package type, is an example of a type of packaging that uses this process [4]. An alternative to roll fed production is blank fed production, where the packaging material is cut up into separate packages prior to going into the filling machine. However, since this type of production is not part of the scope for this study, no further details regarding blank fed production will be discussed here.

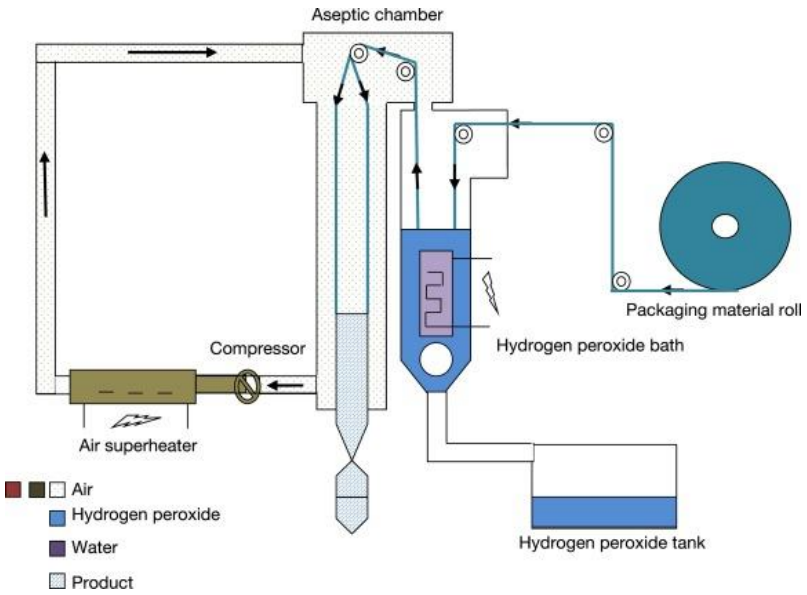

Figure 1 Schematic of a filling machine for roll fed production. A tube of material is formed within the aseptic chamber and is then filled with product and sealed below the liquid line. Image retrieved from [4].

3.1.2 Package material

PM is not one homogeneous material but consists of multiple layers, all with different purposes to keep the package intact and the stored product safe. What differentiate aseptic PM from non-aseptic PM is the inclusion of a barrier to protect the stored product for a long time. An illustration of the different layers that traditional aseptic PM consists of can be seen in Figure 2.

Figure 2 The layers of traditional aseptic PM.

3.1.2.1 Paperboard

The paperboard gives the stability and robustness of the package. A relevant property of the paperboard is bending force, which is measured in millinewton (mN), and is related to the board thickness. The paperboards can be of different thicknesses and vary in other properties such as bleached or non-bleached (duplex). Paper is a porous material that consists of a planar and randomly ordered network of cellulose fibres, and empty space, so called pores. The cavity within the fibres is typically in the range of 1-1000 nm while the pores between the fibres are a few μm. The fact that paper is a porous material means that liquid can flow through pore space, which is an important property during papermaking [5]. A 3D printed model of a paperboard can be seen in Figure 3. The fibres are linked to one another by hydrogen bonding and the bonds controls the mechanical properties of the paperboard [6]. Between the outer polyethylene (PE) layer and the paperboard, there is a clay coat layer with the purpose to smoothen the surface to enable printing, since it usually is not possible to print directly on the rough paperboard (Internal: [7]). The paperboard is not something that Tetra Pak® produces itself, but instead buys from external producers.

Figure 3 3D printed model of cross-section of a paperboard with clay coat layer on top showing both the cavities within the fibres (green circle) and the pores (red circle). The model is not to scale.

3.1.2.2 Barrier

The barrier is traditionally made of aluminium, and it covers the entire inside of the paperboard including the potential hole where the cap is to be attached. The purpose of the barrier is to protect the product from light and oxygen among other things [4] to ensure that the product stays safe, nutritious, and without change in taste for up to more than six months on the shelf [1]. The barrier-covered hole is called a PLH, which can be seen in Figure 4. The same kind of barrier covered hole, but smaller, that is used for portions packs with straws are called pre punctured hole (PPH).

Figure 4 Packaging material with PLH (circled) where the cap is to be attached during package production.

3.1.2.3 Polymers

The different PE layers act as adhesives in some cases and as a protective film for liquids during production and storage in some cases [8]. The amount of polymer used for the different layers is measured in grams per square meter (GSM) which is related to the thickness of the layer. The outer most layer is called the décor layer.

3.2 Edge Wicking and Edge Soaking

Cellulose fibres are hydrophilic by nature which means that the surface has a low contact angle to water (< 90 º) which leads to wetting [5], see schematic in Figure 5. A crucial property of the paperboard is its resistance to penetration of liquid into the plane of the board [9]. This penetration needs to be avoided since water breaks the hydrogen bonds between the fibers which enable movement between them and therefore lowers the strength of the board [6].

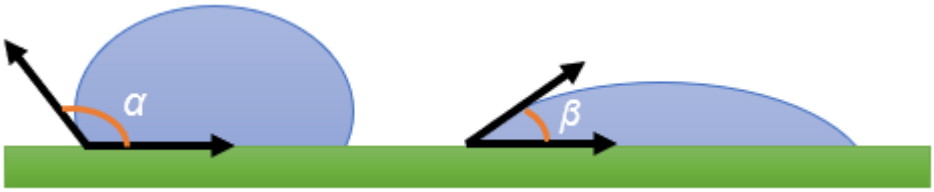

Figure 5 Schematic of the concept contact angle where angle α is larger than angle β which means it is more hydrophobic and less wetting will occur on the surface.

As previously mentioned, polymer layers are used as the main protector of the paperboard from liquid, but this polymer film does not cover the raw edges of the PM [10]. The transportation of water within the paperboard is believed to occur by the following four mechanisms:

- Diffusion transport of vapor in the pores
- Capillary transport of liquid in the pores
- Surface diffusion in the pores
- Water transport through the fibers [11].

The transportation of water is often dominated by one of these mechanisms [12]. For porous material in general, the most common mechanism is capillary transport. However, for boards used for cartons, diffusion of water within the fibers dominates when no external pressure is applied [11]. The permeability typically differs between the different directions of the board and machine direction (MD) usually has a higher water permeability than cross direction (CD) [5].

To achieve good resistance against liquid penetration, the board is treated with additives to increase the contact angle, increase its hydrophobicity, and inhibit capillary transport. This process is called sizing [11]. Dual sizing using rosin and alkyl ketene dimer (ADK) is commonly used in paperboards intended for cartons. The sizing agents lower the surface energy of the paperboard by reducing the amount of hydrogen bonds that can be formed between the hydroxyl groups of cellulose and hemicellulose, that paper mainly consists of, and water. The dominating mechanism for AKD sizing is believed to be the formation of β-ketoester between the active part of the AKD molecule and the hydroxyl group of the cellulose [13]. The reaction can be viewed in Figure 6 below.

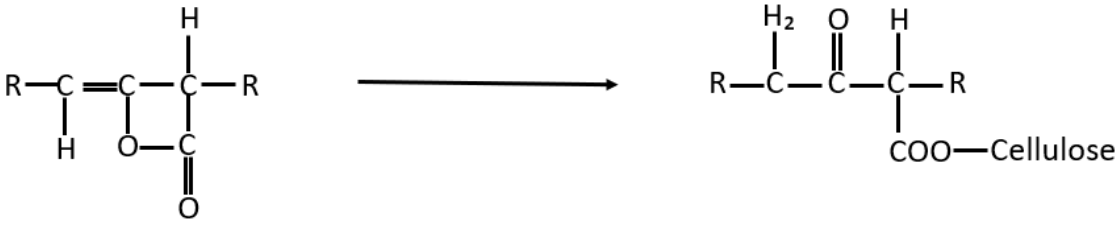

Figure 6 The most common chemical reaction between sizing agent AKD and cellulose in paperboard.

With good coverage of sizing agents, their hydrophobic parts will not be as mobile and will be directed out from the paperboard, thus increase the contact angle of the surface [8]. The EW TM that Tetra Pak® has in its portfolio are measurements of this sizing. During production of aseptic packages, the PM is exposed to a HP bath, as well as residual water in following steps. If the sizing of the paperboard is not good enough and/or if there are defects in the overlaying polymer layer, liquid can penetrate the paperboard where it is exposed [9] such as by the raw edges or around the PLH. EW is a commonly used name in the industry for the this in- plane wetting, but within Tetra Pak®, a differentiation between when the liquid penetration occurs at the raw edges of the PM (EW) and when it occurs around the PLH (ES) is made. There are many different parameters that is believed to influence how much ES that will result from production and many of them are due to the different settings of the filling machine, but of course also due to the amount of sizing that has been performed on the paperboard in combination with the quality of the lamination process (Internal: [14]).

Contrary to EW that can lead to a functionality problem during filling, ES is mostly a cosmetic issue that might lead to the finished packages losing their fresh look, but it is rarely an issue of integrity of the finished package. However, customers and end consumers have reported problems with caps detaching from the packages already before opening it. The concern that this might be due to extensive ES during production needs to be taken seriously and is therefore the main activity of this thesis.

3.3 Hydrogen peroxide

HP has a strong activity against bacteria, yeast, molds, viruses, and bacterial spores and is therefore commonly used in sterilization applications, not only within aseptic packaging, but also within healthcare etc. [15]. HP decomposes into water and nascent oxygen ([O]), which is the monoatomic form of oxygen. The nascent oxygen is very reactive and is the reason why the process of disinfection using HP is very fast [16]. HP is a very hazardous chemical that, in moderate to high concentration, can cause health and safety hazards. It is corrosive for both eyes, skin, and the respiratory system as well as a strong oxidizer which means that it can cause or intensify fire [17]. The mentioned properties are the reason why Tetra Pak® prefers to exclude the usage of HP for quality testing PM if possible.

3.4 Current evaluations

3.4.1 Edge Wicking

One way to evaluate the amount of EW that occurs for different PMs is by placing specimens in 33% HP heated to 70 ºC for 10 minutes. The material is weighed before and after the bath and the result is reported in kg/m^2 (Internal: [18]). The amount of EW can also be evaluated by observing how deep into the specimen liquid has penetrated and reported as a value in mm (Internal: [19]).

3.4.2 Edge Soaking

When evaluating ES, finished packages can be collected and visually examined before attachment of the cap. There is no determined relation between the degree of ES and non-cosmetic issues that possibly could follow (Internal: [20]).

3.4.3 Cap Detachment

The quality of cap attachment can be evaluated by measuring the amount of force that must be added to the cap to be able to remove it from the package. This is performed to ensure that there is no risk of the cap coming off when lifting the package by the cap or when opening the package. Current methods utilize a tensile tester to pull the cap using a constant rate until it detaches from the package (Internal: [21]).

3.5 Design of Experiments

Design of Experiments (DoE) is a powerful statistical method to evaluate the effect of multiple input factors as well as the interactions between the input factors. By using this method, one can determine the key factors of a process and how these factors depend on one another in a less time-consuming way than with the traditional *one factor at the time* approach [22]. DoE will be an important and frequently used tool throughout this master thesis and is utilized both when investigating EW and ES.

4. Methods

4.1 Edge Wicking

4.1.1 Interview study

The relevance of the existing TMs for EW is examined initially by gathering information from different parts of the company. By meeting with people from different departments, with different experience and various competences in combination with documented information from internal systems, the purpose as well as the need of the current EW TMs are evaluated.

4.1.2 Experimental

Three small studies using DoE are performed to investigate the effect of time, temperature, and HP concentration on the current EW TMs for three different materials. Three different values for each parameter are investigated and a center point with two repetitions is used. The number of experiments performed for each DoE is therefore 10. The temperatures, times, and concentrations used are listed in Table 1 below.

Table 1 The values used for the investigated parameters in the DoE.

Parameter	Values tested
Exposure Time	8 min, 10 min, 12 min
Temperature	30 ºC, 50 ºC, 70 ºC
HP Concentration	0%, 16.5%, 33%

The convention when using DoE is picking one value higher and one value lower than the reference, and in this case the values from the existing TMs are used as reference. However, since it is preferable to avoid heating the HP to higher temperatures than 70 ºC due to safety reasons, two values lower than the reference will be used for this parameter. The same reasoning is used for the HP concentration where the highest value is 33% which is what is available in the lab. Ideally, the experiment would be conducted in a randomized order to avoid biases from the operator's increasing skills and other time-dependant effects such as fluctuations in temperature over time, but due to practical reasons this is not possible. Since it would be very time consuming to change the temperature of the liquid between every experiment it is decided that the experiments within each temperature block are to be performed together. To be able to distinguish the temperature dependency from the learning curve of the operator to at least some extent, the experiments are performed in the order: 30 ºC, 70 ºC and lastly 50 ºC. The experiments are performed for one bleached material and two different duplex materials since the absorption of liquid is known to be different for bleached and duplex. The properties of the paperboards in the materials used are listed in Table 2 below. For each experiment in the studies, five specimens with the dimensions 25 x 75 mm are used, see Figure 7.

Table 2 The properties of the materials used in the EW study.

Property	Duplex I	Duplex II	Bleached
Paperboard thickness	150 mN	320 mN	150 mN
Paperboard type	Duplex	Duplex	Bleached

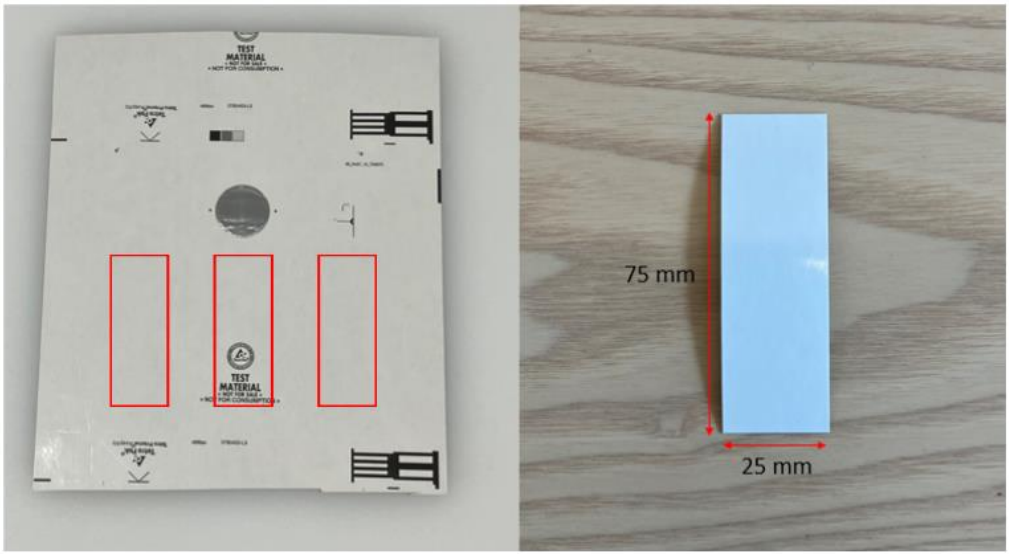

Figure 7 How specimens are cut and the dimensions.

The specimens are weighed both before and after being exposed to the heated bath and the results noted after each experiment are the mass of the liquid absorbed by the material, the average depth of penetration as well as the maximum penetration depth. The average penetration depth is obtained by taking the average of all five specimens and the maximum penetration depth is obtained from the specimen that has the deepest penetration. A loupe with a scale is used for measuring distances. The resolution of the scale is 0.1 mm. The test set-up as well as an example of a set of specimens after the heated bath can be seen in Figure 8 below. Colouring of the solutions simplify the measurement of the penetration depth.

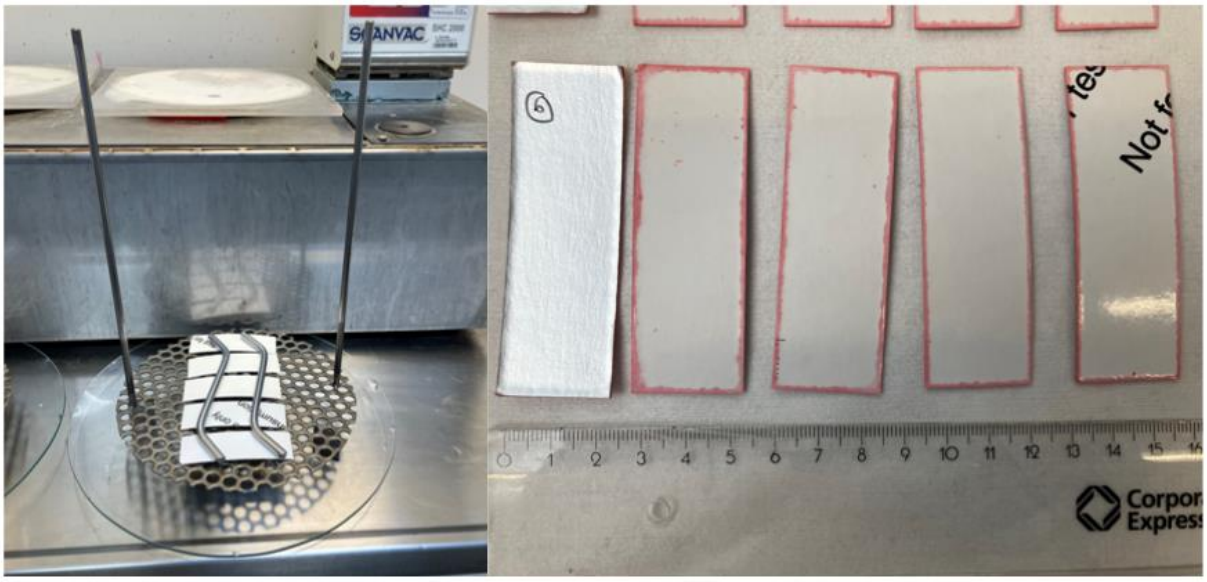

Figure 8 Set up during heated bath and specimens after heated bath. The set of specimens are placed on a tray and lowered down in the heated bath. Metallic rods are used to keep the specimens in their place during the experiment.

4.2 Edge Soaking

4.2.1 Sample creation

The specimens are cut into a circular shape with the PLH in the center using an automatic cutting table, see Figure 9. The diameter of the specimens is 100 mm and a notch in the MD is introduced by the cutting table to be able to keep track of the directions in following steps. The variations for where on the specimen the cap is applied can be minimized due to this, which is important since the frame of the cap is not uniform. To induce ES, existing defects must be present in the décor layer by the edges of the PLH, and these are created manually using a needle in four different places, see Figure 10.

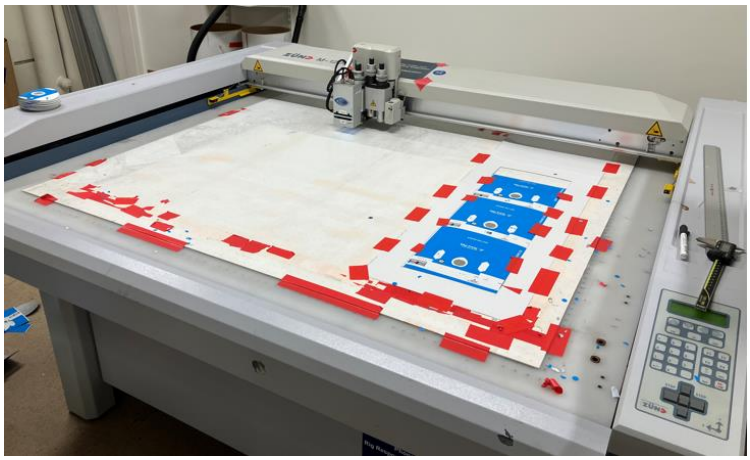

Figure 9 Automatic cutting table used for cutting specimens.

Figure 10 Specimen with four marks around the PLH showing where the defects are created.

4.2.2 Creating and quantifying edge soaking

To induce various degrees of ES into the PM, equipment that forces water into the material by using compressed air is used (ESEE). The ESEE is a rebuilt tool with the purpose of creating various degrees of ES on flat PM. The set-up can be seen in Figure 11 and the chamber is assembled using four screws before filling it with water. Both the pressure and the exposure time can be varied to customize the size of the ES. The chamber must be emptied of water and the screws must be removed prior to each specimen exchange.

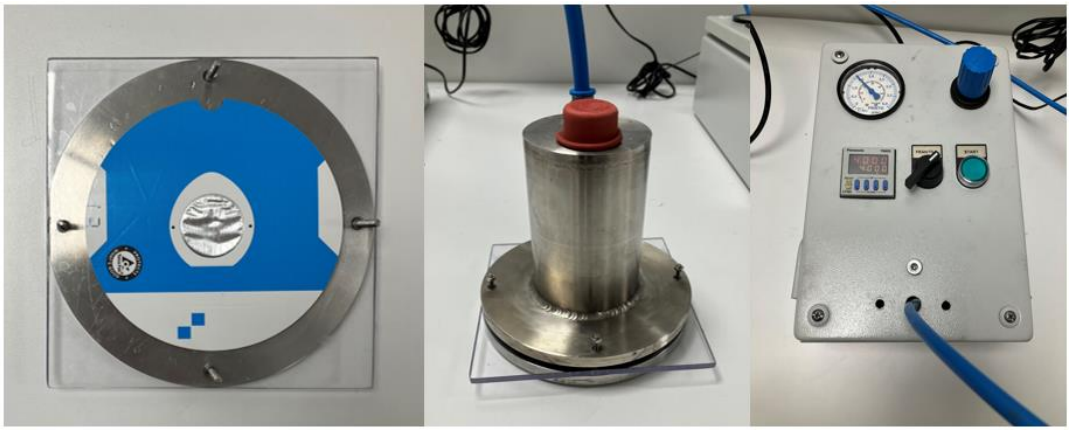

Figure 11 The ESEE tool used for inducing ES in flat PM. The chamber is filled with water and by varying the parameters pressure and exposure time, different degrees of ES can be obtained.

The degree of ES is then quantified using an IR-camera and image analysis performed using ImageJ to measure the soaked area. The area is used as a quantification of how much ES the specimen has and is to be compared with the force measured for cap detachment on the same specimen. The IR-camera has a sensitivity in the near infrared region (950 nm - 1700 nm) and by using this together with a filter, water is detected in the samples with good enough contrast so that the area easily can be measured. A ruler is used to set the scale of the obtained images, and an example of how the measurement is performed can be seen in Figure 12. In addition to area measurements, all specimens are weighed both before and after inducing ES to obtain the mass of the absorbed water as an alternative method for quantification.

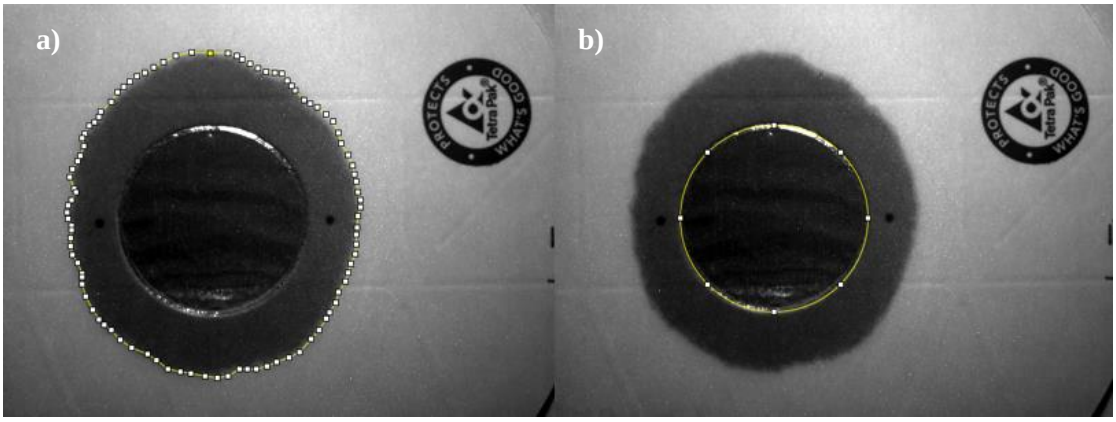

Figure 12 Area measurements performed using ImageJ on images obtained using an IR camera that detects water. The area of the PLH in b) is subtracted from the area in a) to obtain the ES area.

4.2.3 Cap attachment

Following, caps are attached to the PLH, see Figure 13.

Figure 13 Specimen with cap applied at the PLH.

To reduce the operator dependency of the cap application, a YuMi robot is used. Following sequence is executed by the robot:

1. The robot arm moves from the starting position to the cap.
2. The robot arm closes on the cap frame.
3. The robot arm moves to the nozzle providing hot melt.
4. Hot melt is applied in a in a circle on the frame, see Figure 15.
5. The robot arm is quickly moved to the specimen while rotating the cap 180 º.
6. The cap is applied on the specimen using a force of 20 N for 10 seconds.
7. The robot arm lets go of the cap frame and goes back the starting position.

Using the YuMi robot enables the attachment of caps to be done in the same way for all specimens, in a repetitive manner. Parts of the sequence can be seen in Figure 14. Examples of the uncertainties that can be eliminated, or to the least minimized, are the amount and the temperature of the hot melt used, where on the cap the hot melt is applied and where on the specimen the cap is applied, time between applying hot melt and attaching the cap to the PM, pressure used for attachment etc. The parameters used for the attachment can be viewed in Table 3 below.

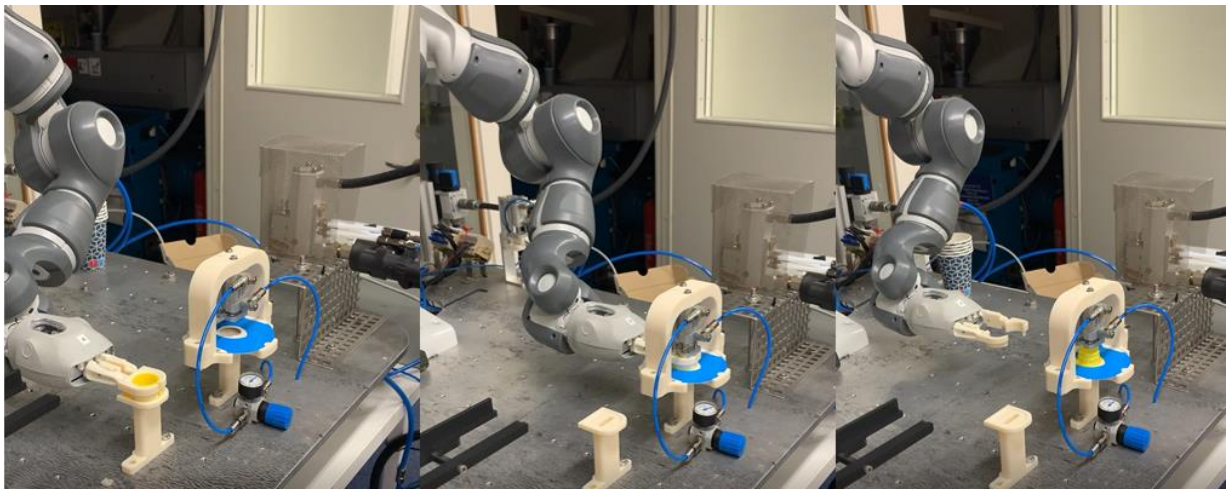

Figure 14 Cap attachment performed by YuMi robot. a) The arm closes on the cap frame. b) The cap is applied to the specimen. c) Robot arm lets go of the cap and returns to staring position.

Table 3 Parameters used for cap attachment.

Parameters	Value
Temperature of hot melt	205 ºC
Quantity of hot melt	0.15 g - 0.18 g
Time between applying hot melt and cap attachment	1.2 s
Pressure used for cap attachment	20 N

Initially some difficulties appear when depositing the hot melt on the cap frame. During the test run, the hot melt deposition on the first few caps works just fine, but after a few depositions, the amount of hot melt decreases substantially until completely ceasing. After discussing the problem together with colleagues with a lot of hot melt experience, the temperature of the hot melt tank and tube leading to the nozzle is changed, and together with exchanging filters in the hot melting system, this solves the problem, and even distribution of hot melt can be performed for many caps without problems occurring. The hot melt distribution before and after these changes can be seen in Figure 15 below.

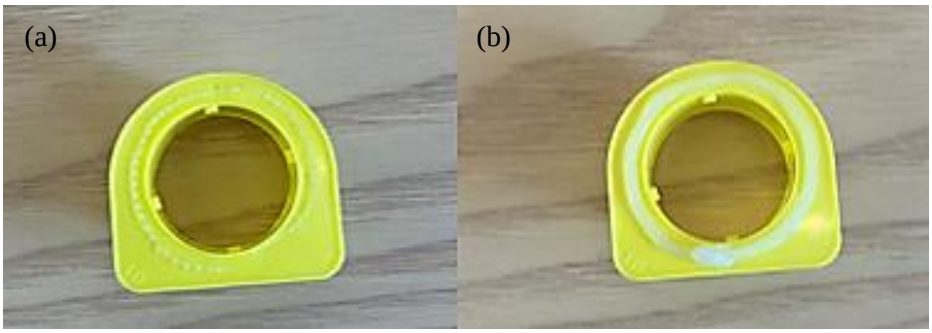

Figure 15 HeliCap™ 26 Pro with hot melt (a) before filter exchange and optimization of temperature settings and (b) after filter exchange and optimization of temperature settings.

4.2.4 Cap detachment

Cap detachment is performed using a tensile tester that pulls the cap at a constant speed and records the forces needed for detaching the cap. Fixtures for holding the PM specimen as well as attaching to the cap are designed and 3D printed in collaboration with a colleague with competence within CAD. The speed used for the testing is 300 mm/min, see exploratory activities in section 4.2.7.3.

4.2.5 Sample size

Three scenarios are investigated:

1. Attaching and detaching the cap while the board in the specimens is still wet from ES.
2. Drying the specimens overnight using a 60 ºC oven followed by placing them in a climate-controlled environment (23 ºC and 50% RH) for two weeks to let the paperboard acclimate prior to attaching the caps, see Figure 16.
3. Drying the specimens in the 60 ºC oven overnight and then attaching the caps without letting the material acclimate.

Around 22 specimens for each scenario are created with the ambition to have the degree of ES evenly distributed among them.

Figure 16 Specimens placed in climate-controlled environment for two weeks to acclimate the paperboard.

4.2.6 Material

Two types of PM are used in the study. One which has a bleached paperboard with a thickness of 150 mN and a 16 GSM décor layer and one with an unbleached paperboard (duplex) with a thickness of 150 mN and a 16 GSM décor layer. The caps used are the HeliCap™ 26 Pro and it can be seen in Figure 17 below. The HeliCap™ 26 Pro is a tethered cap that allows the lid of the cap to stay attached to the frame after opening, according to directives from the EU. However, this has no effect on this study since the entire lid is removed from the cap frame

prior to attachment to the specimen. The glue that is used for the cap attachment is the hot melt adhesive CapFix 600V.

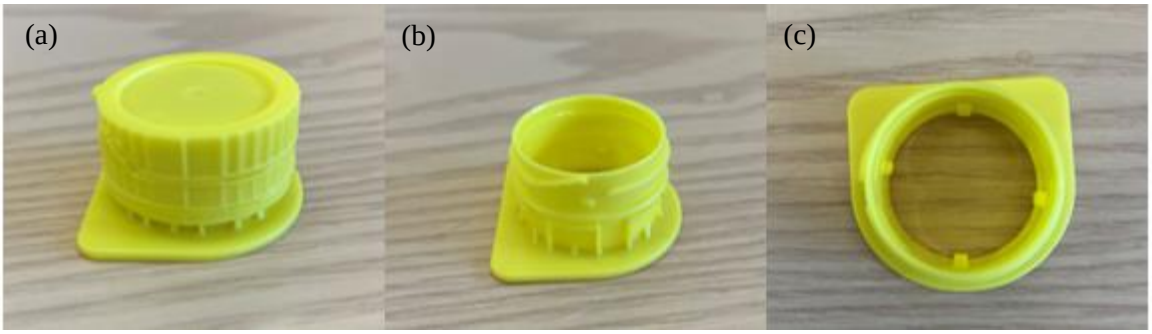

Figure 17 HeliCap™ 26 Pro (a) with lid (b) with the lid removed and (c) showing the knobs used for attaching to the fixture.

4.2.7 Exploratory activities

4.2.7.1 Edge soaking over time

To get an overview of how fast the ES dries out after being induced into the specimen a small pre-study is performed. The specimens are weighed, and three degrees of ES (low, medium, and high) are induced in bleached and duplex PM by varying the exposure time and the pressure used. After creating ES using ESEE, the specimens are weighed and photographed after 15 min, 30 min, 45 min, 1 h, 2 h, 3 h, 6 h, and then at an interval of 24 h (or 72 hours over weekends) for 19 days. This is done to monitor the drying of the sample and investigate if there is initial swelling of the sample that leads to an increase in the soaked area during the first period after inducing ES. Photographs of the soaked specimens are therefore only taken during the first six hours. No complete measurement of the areas is performed for this initial study, but measurements in two directions (CD and MD) are made and used for comparison, see Figure 18. The aim of monitoring the change in area and mass is to decide which method would be most suitable for quantifying the degree of ES: measuring the mass of the absorbed water or the area of the soaked material.

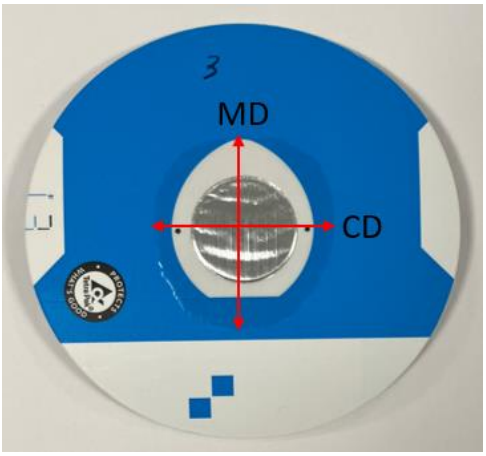

Figure 18 Specimen with edge soaking present and the directions of how it was measured is marked.

One can observe that the mass of the specimens does not decrease substantially during the first hour for either of the material types, see Figure 19 and Figure 20.

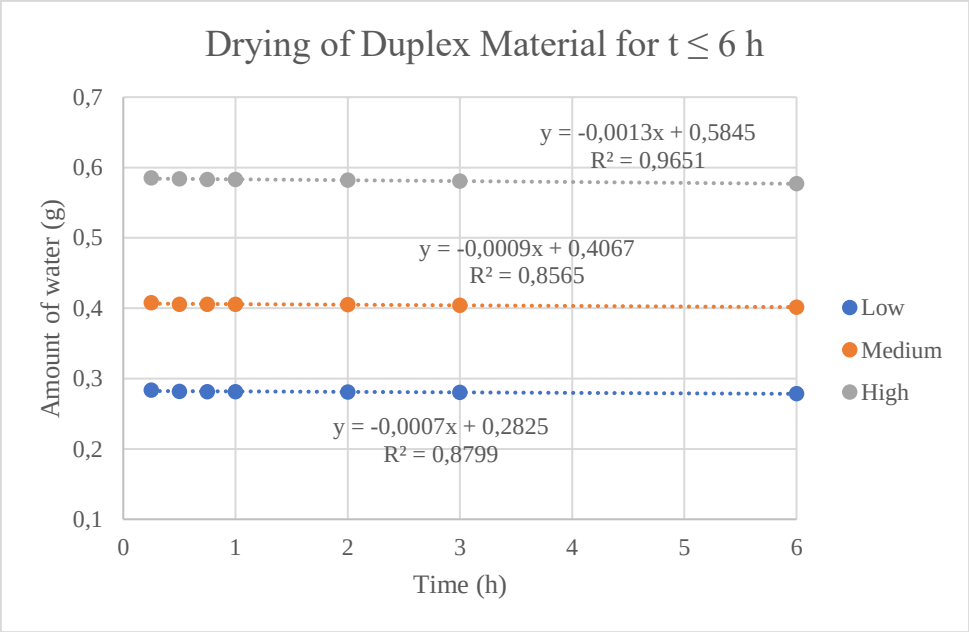

Figure 19 The results from monitoring the drying of the specimens of duplex material during the first 6 hours after inducing ES.

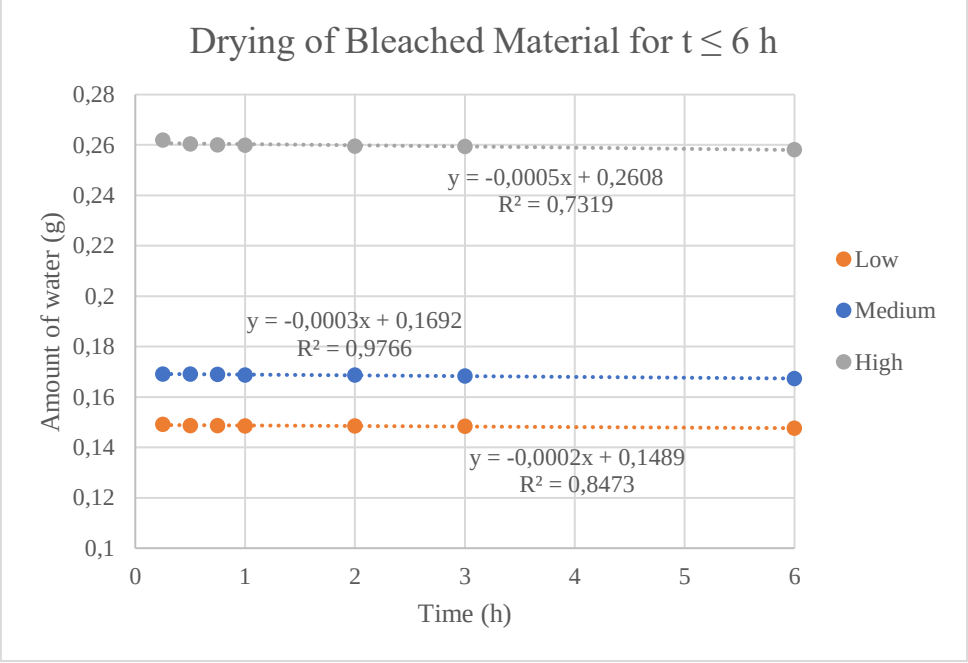

Figure 20 The results from monitoring the drying of the specimens of duplex material during the first 6 hours after inducing ES.

For both materials the drying is a linear course initially, see Figure 21 and Figure 22. The *k* values are somewhat higher for the duplex material than the bleached and for both materials the *k* values are higher for high degree of ES. It is however noteworthy that the *m* values correspond

to the amount of water absorbed and that the values are much higher for the duplex material than for the bleached even though the same settings for the ESEE is used for both materials. The fact that the *k* values are higher for the duplex material might therefore not have to do with the drying process of the material itself, but rather that it absorbs liquid more easily and therefore has a steeper moisture reduction. After the initial linear trend, the moisture content reaches a plateau value. This can be compared to a reference value of how much moisture the specimens absorb just by being put in a climate-controlled environment. Ten specimens without ES for each material type is placed in climate-controlled environment and are weighed at the same interval as the specimens with ES. These references are demonstrated by the solid red lines in Figure 21 and Figure 22 respectively.

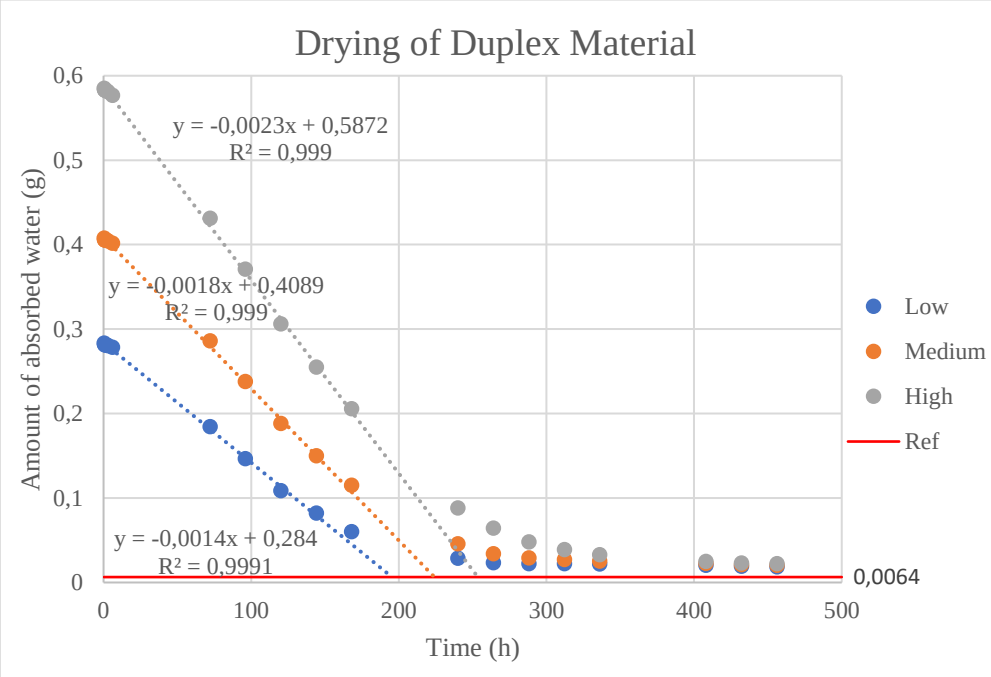

Figure 21 The results from monitoring the drying of the specimens of duplex material. During the first 144 h, linear behavior can be observed. A reference is used to demonstrate how much moisture the specimens absorb just by being put in a conditioned environment.

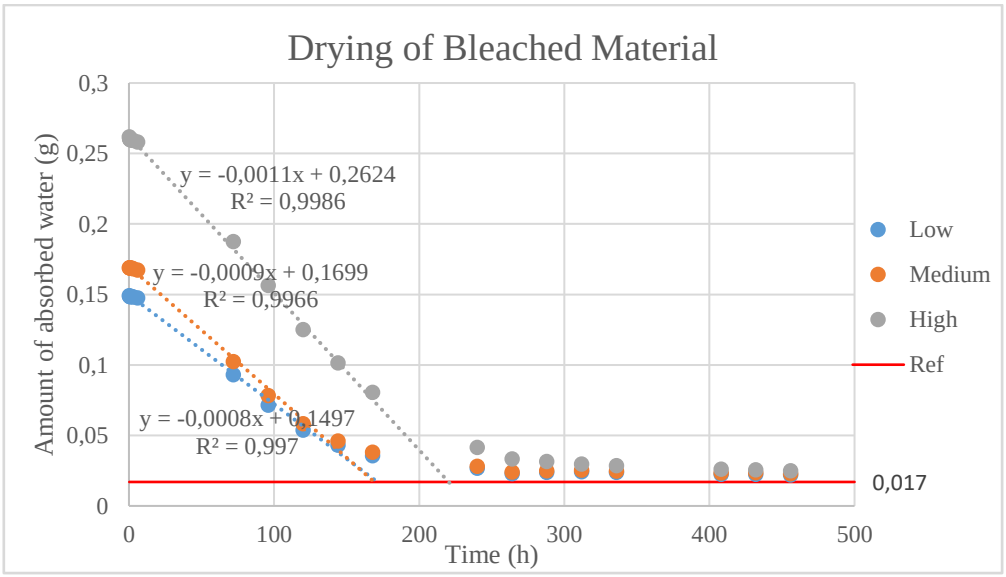

Figure 22 The results from monitoring the drying of the specimens of bleached material. During the first 144 h, linear behavior can be observed. A reference is used to demonstrate how much moisture the specimens absorb just by being put in a conditioned environment.

The area of the soaked material increases initially for both materials and for the duplex material a clear trend can be observed for all degrees of ES in this interval, see Figure 23. This might be due to initial swelling of the material since large amounts of water are being introduced very quickly. For bleached material the trend is not as clear, see Figure 24. However, the amount of water introduced in the bleached material is much lower than for the duplex material since it absorbs less water for the same exposure time and pressure. The fact that less water is introduced to the bleached material could therefore be a reason for the lack of a clear trend as in the case of duplex. If the area of the soaked material is to be used as a quantification method, it might be reasonable to not perform the measurements directly after inducing the ES but instead wait for 30-45 minutes to allow for the water to be evenly distributed in the paperboard.

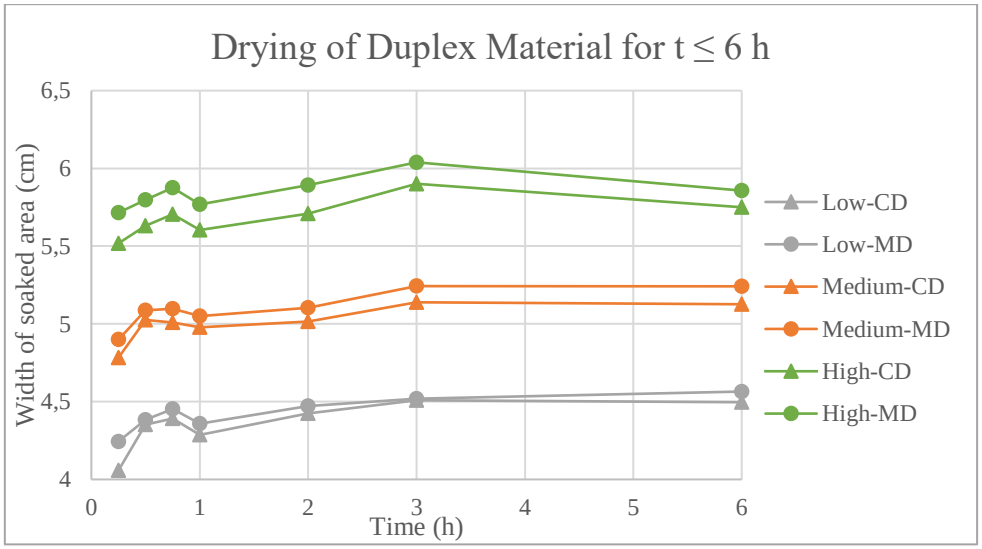

Figure 23 The results from monitoring the soaked area of the specimens of duplex material for three different degrees of ES.

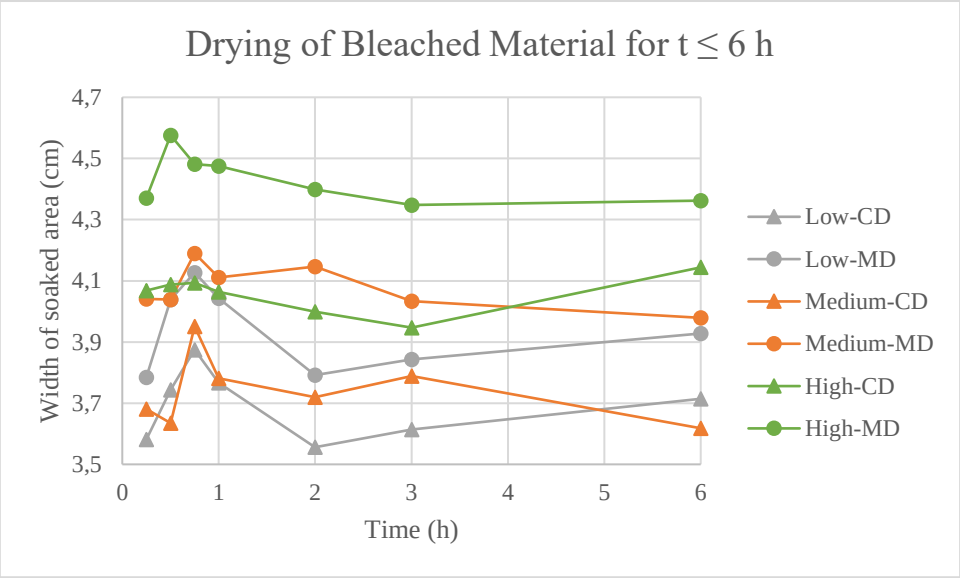

Figure 24 The results from monitoring the soaked area of the specimens of bleached material for three different degrees of ES.

4.2.7.2 Mass vs area

To further investigate the relationship between the mass of the water absorbed and the area of the soaked material, a small pre-study where specimens of varying degree of ES are weighed and measured using the IR camera is performed. The specimens are weighed before and just after performing the ES and the area is measured after approximately 45 minutes according to the results in section 4.2.7.1. A linear relationship between the mass of the absorbed water and the soaked area around the PLH in bleached PM could be obtained as can be seen in Figure 25. Similar results were observed for duplex material.

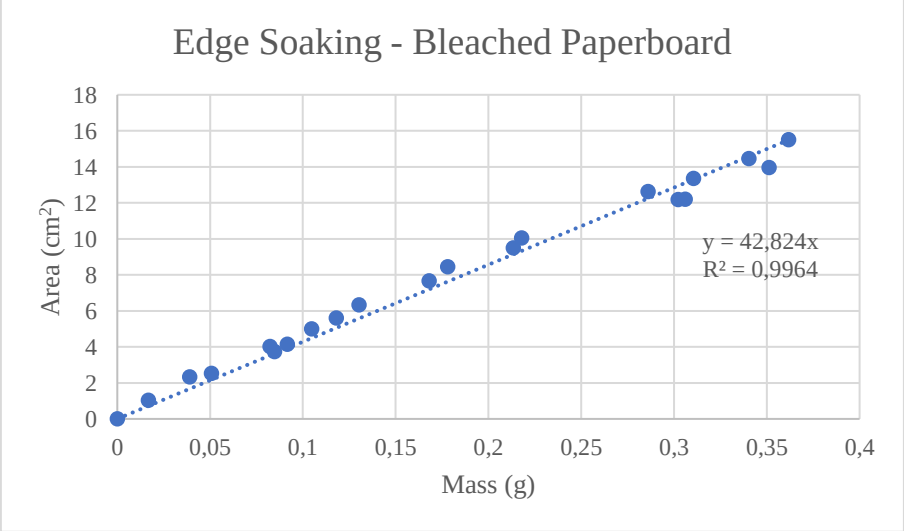

Figure 25 Linear relationship between mass of absorbed water and soaked area when performing ES.

4.2.7.3 The effect of pulling rate on force measurements

Small studies using DoE are conducted for both bleached and duplex material. The factors investigated are the degree of ES (None, smaller than frame, size of the frame, larger than frame) as well as the rate used for detaching the caps (100 mm/min, 300 mm/min, 500 mm/min). Using α=0.15 only the degree of ES has a significant effect on the force needed to detach the cap for both materials and one can therefore assume that the rate for cap detachment can be chosen freely without affecting the result of the measurement in the investigated interval. 300 mm/min was chosen as the rate for the following measurements. Figure 26 shows the pareto chart of the duplex material, but similar results is obtained for bleached.

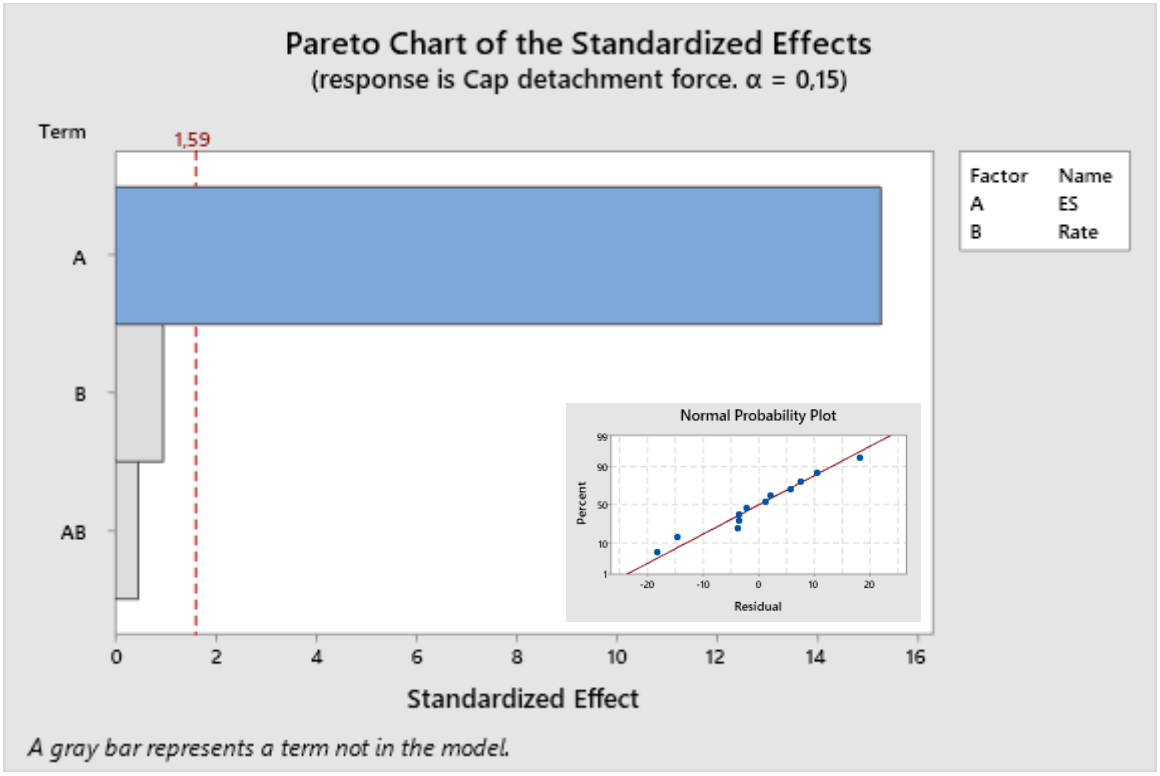

Figure 26 Pareto chart for what factors affect the cap detachment force. Only the degree of ES has a significant effect for α=0.15.

5. Method development for Cap Detachment

5.1 First edition

Since the aim of this project only is to investigate the relationship between the degree of ES and force needed for cap detachment, not necessarily mimic conditions close to reality, a test set up that is as simple as possible is the first approach. Using a 180 º pulling angle is deemed the simplest case (straight upwards). When using this angle there are less options for what direction the cap can be pulled in since the cap frame is not uniform, see Figure 15Figure 29. An additional reason to use a 180 º pulling angle is that one could argue that this can be used to mimic the forces applied when lifting the package by the cap, which can happen during transport or by the end consumer.

Since there is no TM available for measuring the force needed for cap detachment on flat PM, a fixture that can be used together with a tensile tester is designed in collaboration with a colleague with competence within CAD. The fixture consists of a plate that can be attached to the base of the tensile tester and a lid with a hole that can be attached to the plate using screws to apply pressure. Three versions of the lid are designed, where the difference between them is the size of the hole. The dimensions of the holes are 10-, 20-, and 30-mm larger diameter than the widest part of the cap frame and their exact measurements are compiled together with the dimensions of the specimens in Table 4 below.

Table 4 Relevant dimensions of the cap detachment test setup.

	Measurements
Diameter Sample	100 mm
Widest part of cap frame	42.54 mm
Diameter hole – Lid 1	52.54 mm
Diameter hole – Lid 2	62.54 mm
Diameter hole – Lid 3	72.54 mm

The reason for designing the three alternative lids at once is that the size of the hole might affect the force measurements and the limitation regarding time makes it a more efficient way to have a few options from the start since it can take a few weeks to get the designs printed. The hole should not be large enough to enable bending of the specimen under the applied force. Therefore, high pressure from the lid is needed and a hole large enough for the cap to be removed from the specimen without problems, but small enough to not enable bending. Unfortunately, due to miscommunication in the 3D printing process, only two of the three lids are produced, which are Lid 1 and Lid 2 from Table 4. The lids can be seen in Figure 27 below.

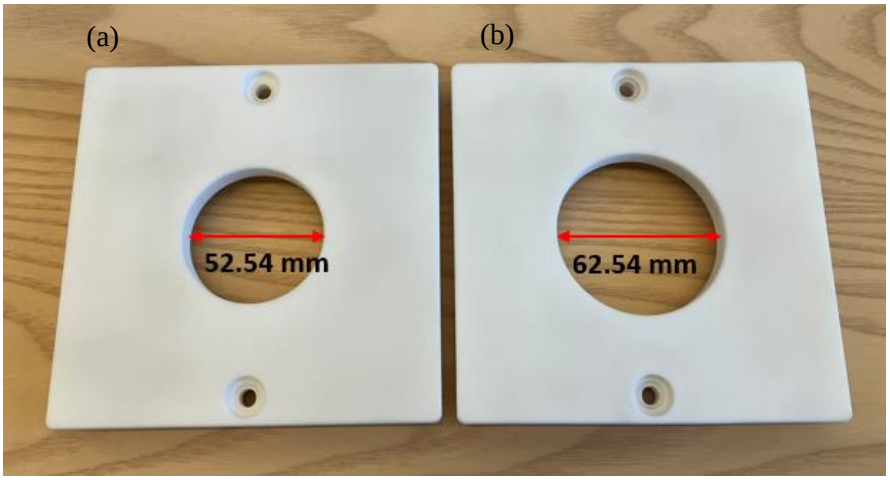

Figure 27 The lids created for cap detachment with a hole diameter of (a) 52.54 mm and (b) 62.54 mm.

In addition to the base plate and the lids, a fixture that is attached to the cap as well as the load cell of the tensile tester is designed. A visual representation of the test set up can be seen in Figure 28 below.

Figure 28 Visual of the 3D-printed fixtures created to measure cap detachment force.

Unfortunately, the model does not work for detaching the caps for unsoaked material where the forces are high. The part of the cap that the fixture attaches to breaks due to the high forces and the fixture detaches from the cap frame without removing it from the specimen. The broken knobs of the caps can be seen in Figure 29 below.

Figure 29 HeliCap™ 26 Pro with broken knobs from using the first edition of cap detachment fixtures.

For specimens with large degree of ES, same order of magnitude as cap frame and larger, and where both the cap attachment and detachment are performed while the paperboard still is wet the measurements could be performed using this first edition test set-up. This indicates that the force needed for cap detachment is lower for large degrees of ES, at least while the material is still wet. The forces needed are between approximately 50-80 N for both PM types. The breakage occurs between the décor layer and the clay coat, see Figure 30.

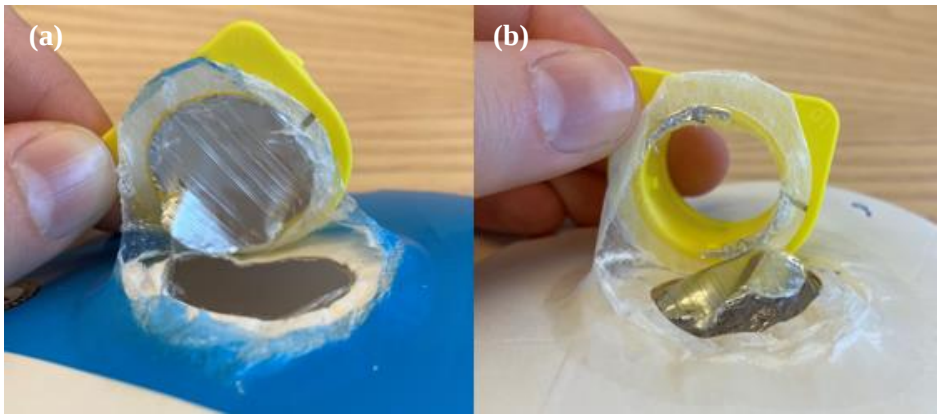

Figure 30 Specimens with large degree of ES after cap detachment. (a) Bleached material (b) Duplex material.

Since the parts of the cap that the first edition fixture was attached to are not robust enough to withstand the large forces from the 180 º pull, a new method needs to be designed.

5.2 Second edition

A fixture using a 180 º angle but utilizing a more robust part of the cap for attachment is designed and printed. The part of the cap that the fixture is attached to can be seen in Figure 31 below. Unfortunately, this fixture also does not work for the unsoaked specimens, and the fixture detaches from the cap without removing it from the specimen during force measurements. However, it is possible that this fixture could be a suitable alterative with a few modifications, but due to the time limit, this was not achievable within this study.

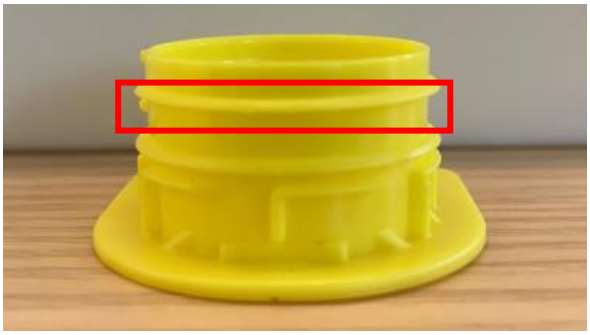

Figure 31 HeliCap™ 26 Pro with the part attaching to the second edition cap detachment fixture marked.

5.3 Third edition

Since the parts of the cap that the first edition fixture is attached to are not robust enough to withstand the large forces on their own, a new method needs to be designed. Even though the initial plan was to perform the measurements using a 180 º angle, a test set up using a 90 º angle instead, is designed. The prospect is that the large forces applied will be more evenly distributed on the cap using a 90 º angle and that this might enable cap detachment without breaking the cap. In addition to a more even force distribution on the cap, the forces that the PM is exposed to should be localized on a smaller part of the specimen. The third edition of the fixtures is based on the same principle as the first (the fixture attaches to the inside knobs), and the same

lids are used as for the first and second edition test set-up. However, a new base plate as well as a new cap attaching fixture is designed to enable a 90 º pull. The set-up can be seen in Figure 32 below.

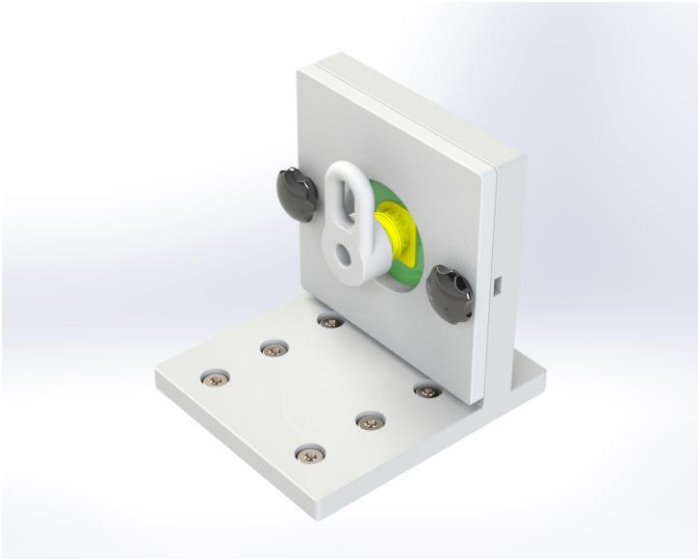

Figure 32 Visual of the third edition cap detachment test set-up using a 90 º pulling angle.

Unfortunately, neither using a 90 º angle makes it possible to detach the cap from the specimen for unsoaked material and the knobs breaks for forces around 210-250 N. However, for very small degrees of ES, the fixture works well (cap detachment forces of around 65-80 N) for specimens with wet paperboard.

5.4 Fourth edition

Since the problem of the fixtures is that the cap is not robust enough to withstand the high forces needed for cap detachment from specimens with no ES, an attempt to modify the existing third edition fixture is performed. By drilling a hole through both the fixture and the cap, a peg can be pushed through them, and the third edition fixture will no longer depend on the inside knobs of the cap frame. To be able to control that the holes on the cap are placed at the same location for all caps, the first version of the cap fixture is used as a mold for where the holes should be placed to increase the repetitiveness. The set-up for drilling holes in the caps as well as the fourth edition cap fixture can be seen in Figure 33 below.

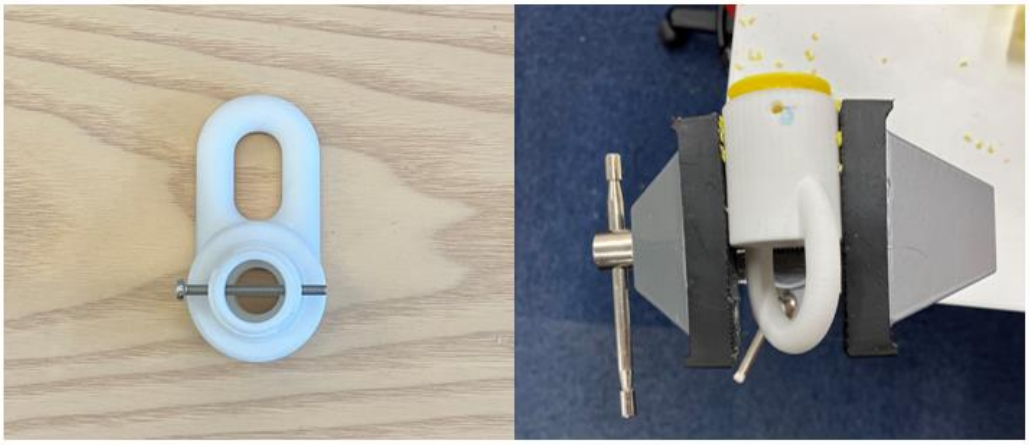

Figure 33 Final cap detachment fixture using a pin and the old fixture used as a mold to enable accuracy of the hole placement when drilling.

This version of the fixtures was able to remove the cap for specimens without ES and is therefore used as the test set-up for cap detachment. The final set-up mounted in the tensile tester as well as a schematic of the test set-up can be seen in Figure 34. Lid 2 is used since the hole of Lid 1 is small enough that the cap jams to the edge of it when removed which disturbs the measurements.

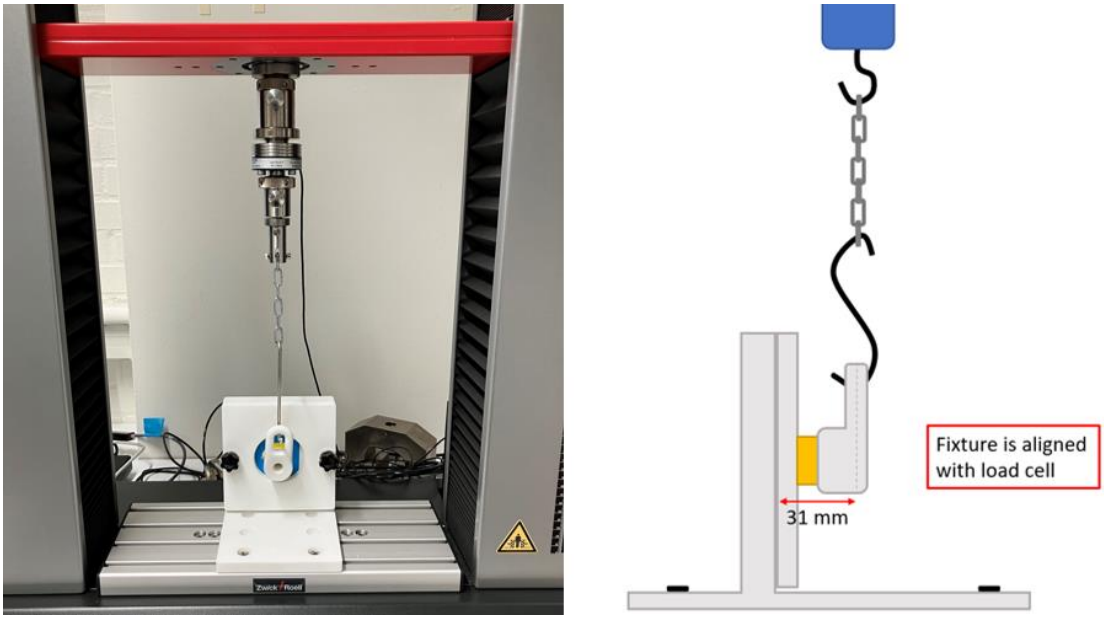

Figure 34 Final test set-up in the tensile tester and schematic with measurement.

6. Results

6.1 Edge Wicking

The following information can be compiled when meeting with colleagues from Physical Modelling, Material Analysis and Base Materials together with internal reports from the company.

EW testing is used as a measurement of the sizing of the paperboard, and the HP bath is used to mimic when material stays in the aseptic bath in the filling machine for a longer time than usual during a short stop in production (Internal: [23]). If this is the best way to evaluate the sizing is unclear and perhaps it would be enough to use water (possibly with some surfactant). The TMs are generally not used in converting factories since EW is a paperboard quality and is therefore usually not evaluated in the factories. Even if the converting factories wanted to perform the TM at some point, special equipment such as fume hood is needed to handle HP which generally is not available in the factories. However, the TMs are performed regularly by the producer of the paperboards to ensure that the boards deliver according to the Tetra Pak® specifications and are therefore deemed relevant. This to ensure that there is no risk of breakage of the PM reel when resuming production after a short stop. If this were to happen, the system would have to be shut down to reattach the material lane. There would also be a risk of product leakage due to tube burst, which would require thorough cleaning and sterilization of the aseptic environment before resuming production. All these possible outcomes have great economic consequences and need to be avoided.

In addition to the paperboard producer, the TMs are also frequently performed at the department of Material Analysis and are used for evaluating new PMs during development in various projects in the company. It is therefore of interest to exchange the HP for a more lenient chemical, but this activity would have to be performed in collaboration with the paperboard producer and the department of Base Materials. The scope of the study that would have to be performed for this kind of chemical exchange is far larger than what can be done within this master thesis, but the results from the small experiments performed to investigate time, temperature, and HP concentration dependency of the TMs are presented in following sections.

6.1.1 Duplex I

For the duplex material with 150 mN paperboard, the results of the EW experiment can be seen in Table 5. As can be observed in Figure 35, showing a pareto chart for how the mass of the absorbed liquid is affected by the investigated parameters, the temperature is the parameter with most impact. Using α=0.15 both temperature and HP concentration affects the mass of the absorbed liquid, but temperature to a much higher degree. Time does not affect the result, which is interesting since, prior to this study, it has been believed that this would have a very large impact on the result when performing EW testing.

Table 5 The results from the DoE on edge wicking for duplex I material.

Time (s)	Temp. (ºC)	HP Conc. (%)	Mass (g)	Maximum Depth (mm)	Average Depth (mm)
720	30	0	0.0654	0.6	0.5
600	50	16.5	0.0917	0.8	0.7
720	70	33	0.2776	2	1.5
480	30	0	0.0522	0.5	0.4
480	70	33	0.188	1.6	1.1
720	70	0	0.1716	1.3	0.9
480	70	0	0.1289	0.7	0.5
720	30	33	0.077	0.7	0.5
600	50	16.5	0.0915	0.9	0.6
480	30	33	0.0694	0.6	0.4

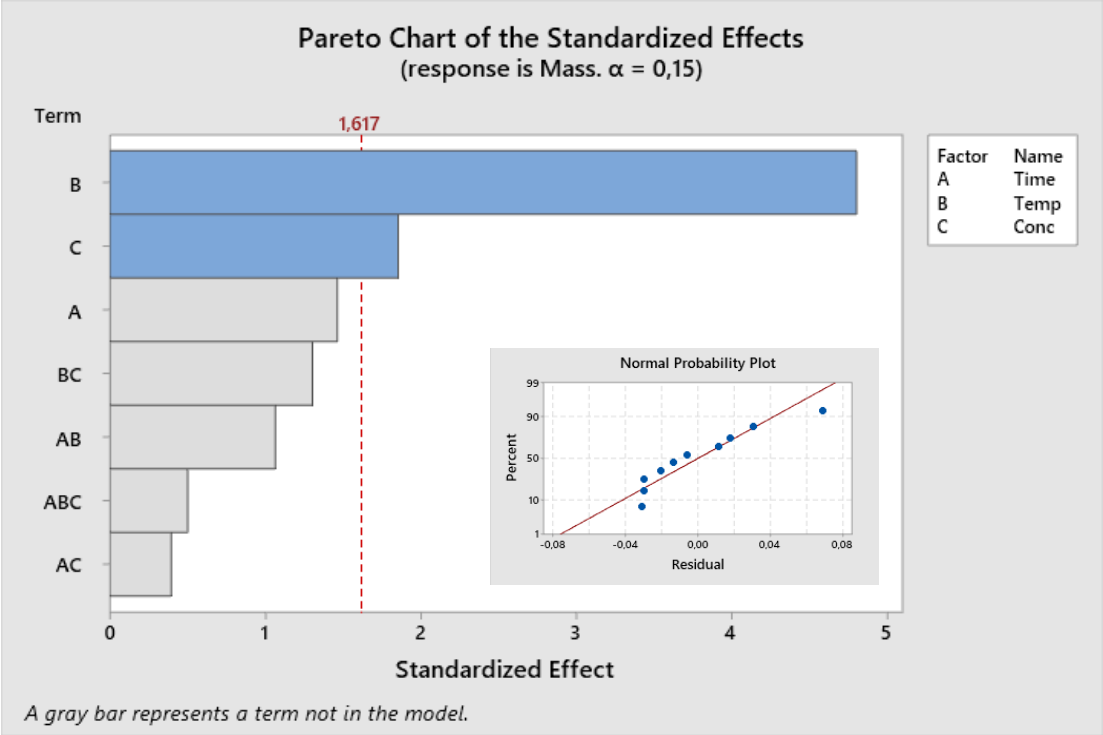

Figure 35 Pareto chart for what factors affect the mass of the absorbed liquid in duplex I material. The most prominent parameter is temperature, and substantially less prominent, but still significant, is the HP concentration. Time does not affect the mass of the liquid absorbed for α=0.15.

For the distance measurements, both average and maximum depth, all the parameters investigated as well as confounding parameters for temperature and concentration, and also time and temperature, have a significant effect using α=0.15, see Figure 36 and Figure 37. However, the residuals have a somewhat peculiar distribution. When studying the values for average and maximum depth in Table 5, one can observe that the variations in the obtained values are not that large. It is a very limited interval of values, and in addition to this it is not possible to obtain a better resolution than 0.1 mm. This makes the measurements very discontinuous in comparison with the mass of the liquid that can be measured with 0.001 g resolution. It is therefore not surprising that the limited DoE cannot separate the effect of the

different parameters when it comes to distance. It should however be noted that for both maximum and average depth, as well as for the mass of the absorbed water, the temperature has the highest effect followed by the concentration of the HP.

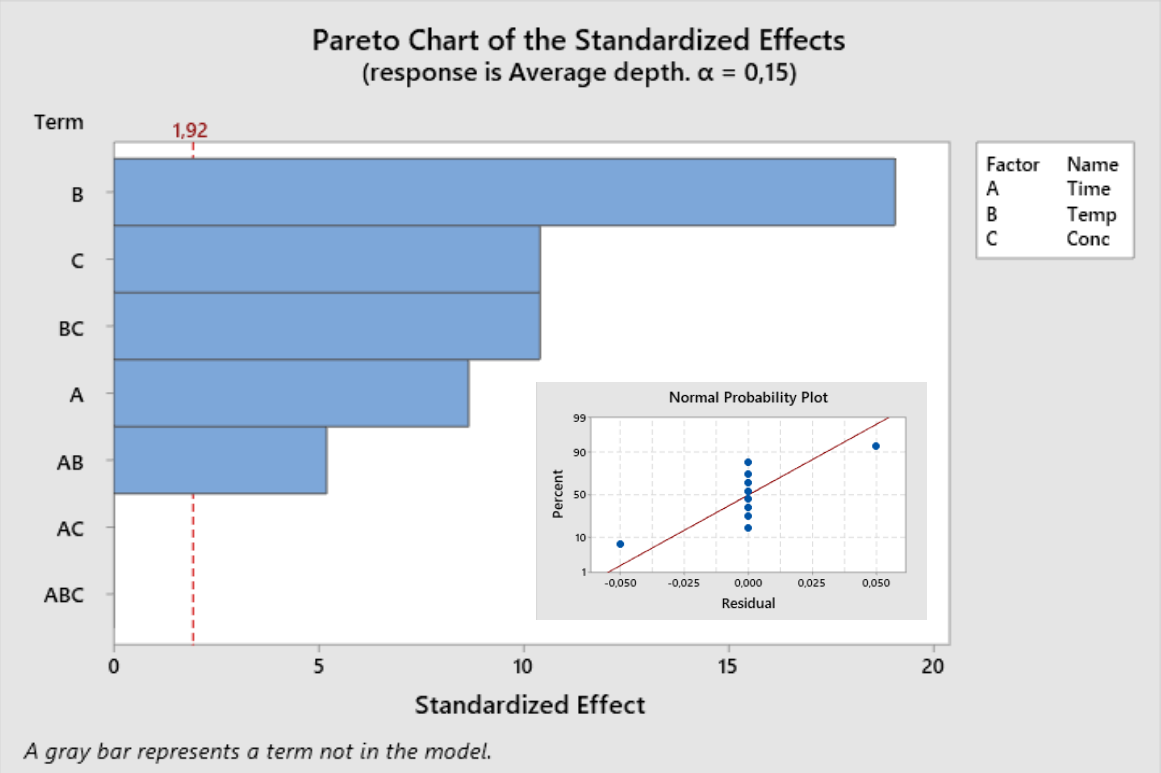

Figure 36 Pareto chart for what factors affect the average depth of the EW for duplex I material. The temperature is the most prominent parameter, and the second most prominent factors are HP concentration as well as the confounding factor of temperature and HP concentration. Also, time is an affecting factor of average EW depth as well as the confounding factor of temperature and time for α=0.15.

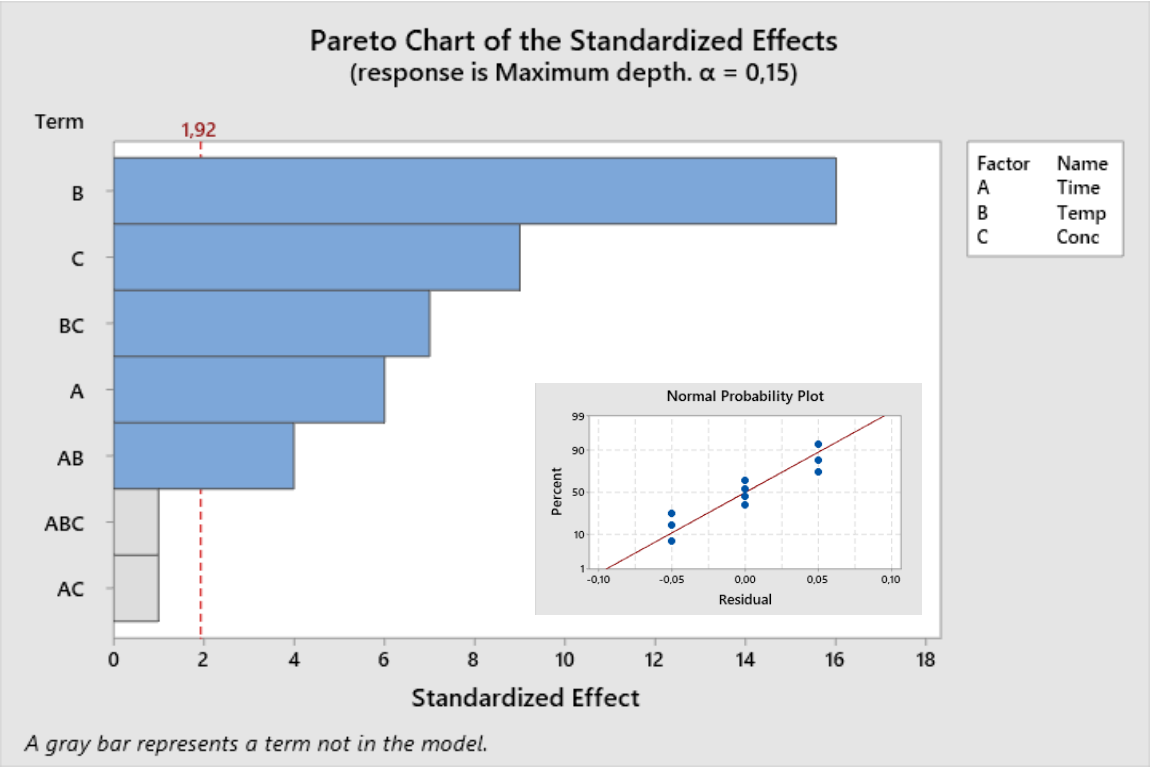

Figure 37 Pareto chart for what factors affect the maximum depth of the EW for duplex I material. The temperature is the most prominent parameter, and the second most prominent factor is HP concentration followed by the confounding factor of temperature and HP concentration. Also, time is an affecting factor of maximum EW depth as well as the confounding factor of temperature and time for α=0.15.

For the following materials only the results for the mass of the absorbed liquid will be discussed, but pareto charts for average and maximum depth as well as the residual plots for all three of the experiments can be found in the appendix, see section 11.1.

6.1.2 Duplex II

For the PM with a 320 mN duplex paperboard, only the temperature has a significant effect on the mass of the absorbed liquid for α=0.15 as can be seen in Figure 38. The values for all the sets of the experiment are compiled in Table 6.

Table 6 The results from the DoE on edge wicking for duplex II material with a thicker paperboard.

Time (s)	Temp. (ºC)	HP Conc. (%)	Mass (g)	Maximum Depth (mm)	Average Depth (mm)
720	30	0	0.1087	0.9	0.6
600	50	16.5	0.1414	1.1	0.8
720	70	33	0.3365	2	1.1
480	30	0	0.0873	0.8	0.6
480	70	33	0.3309	2	1
720	70	0	0.3412	2.5	1.2
480	70	0	0.2302	1.2	1
720	30	33	0.124	0.8	0.6
600	50	16.5	0.1426	1	0.8
480	30	33	0.1035	1	0.5

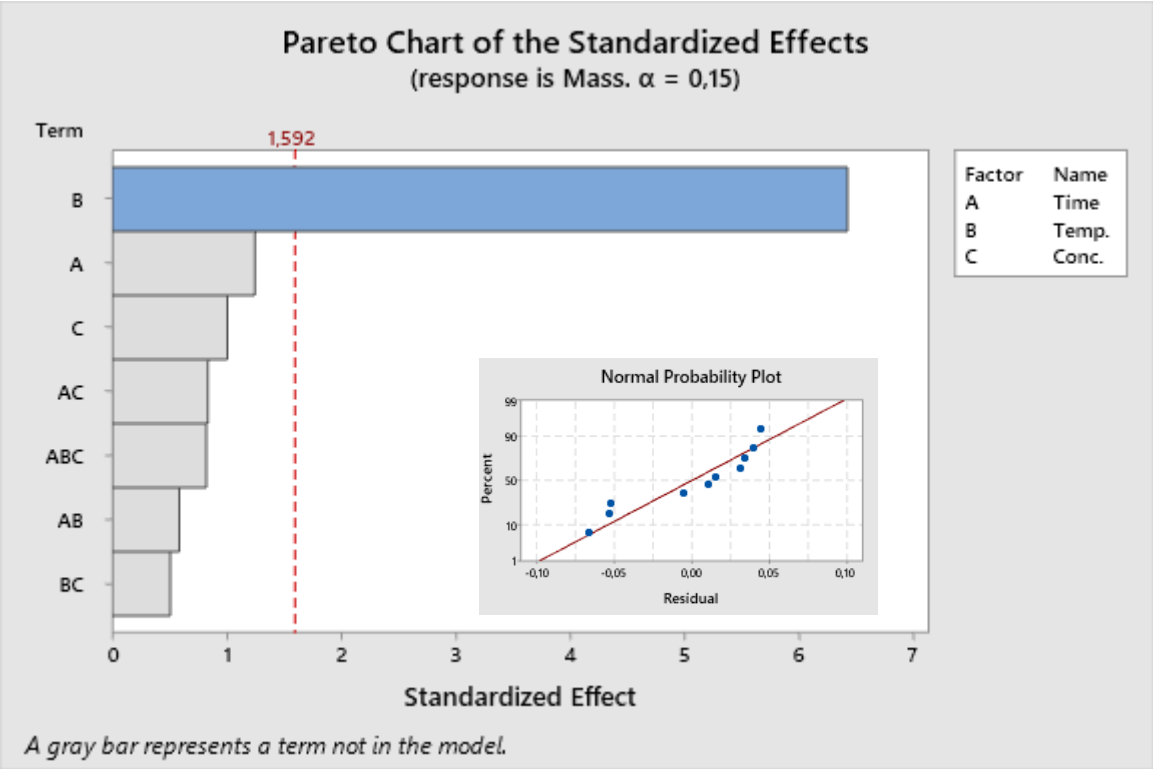

Figure 38 Pareto chart for what factors affect the mass of the absorbed liquid in duplex II material. Temperature is the only significant factor, and time as well as HP concentration do not affect the mass of the liquid absorbed for α=0.15.

6.1.3 Bleached

For the PM with a bleached paperboard both the temperature of the liquid and the time have a significant effect on the mass of the absorbed water for α=0.15 which can be observed in Figure 39. However, the effect of the temperature is much larger than the effect of the time. The resulting values from all the sets in the experiment is compiled in Table 7.

Table 7 The results from the DoE on edge wicking for bleached material.

Time (s)	Temp. (ºC)	HP Conc. (%)	Mass (g)	Maximum Depth (mm)	Average Depth (mm)
720	30	0	0.0779	0.8	0.6
600	50	16.5	0.0911	0.8	0.5
720	70	33	0.1703	1	0.5
480	30	0	0.0655	0.7	0.6
480	70	33	0.1428	1.2	0.6
720	70	0	0.1569	1.9	1
480	70	0	0.1209	1	0.7
720	30	33	0.087	0.6	0.4
600	50	16.5	0.0897	0.9	0.5
480	30	33	0.0724	0.6	0.4

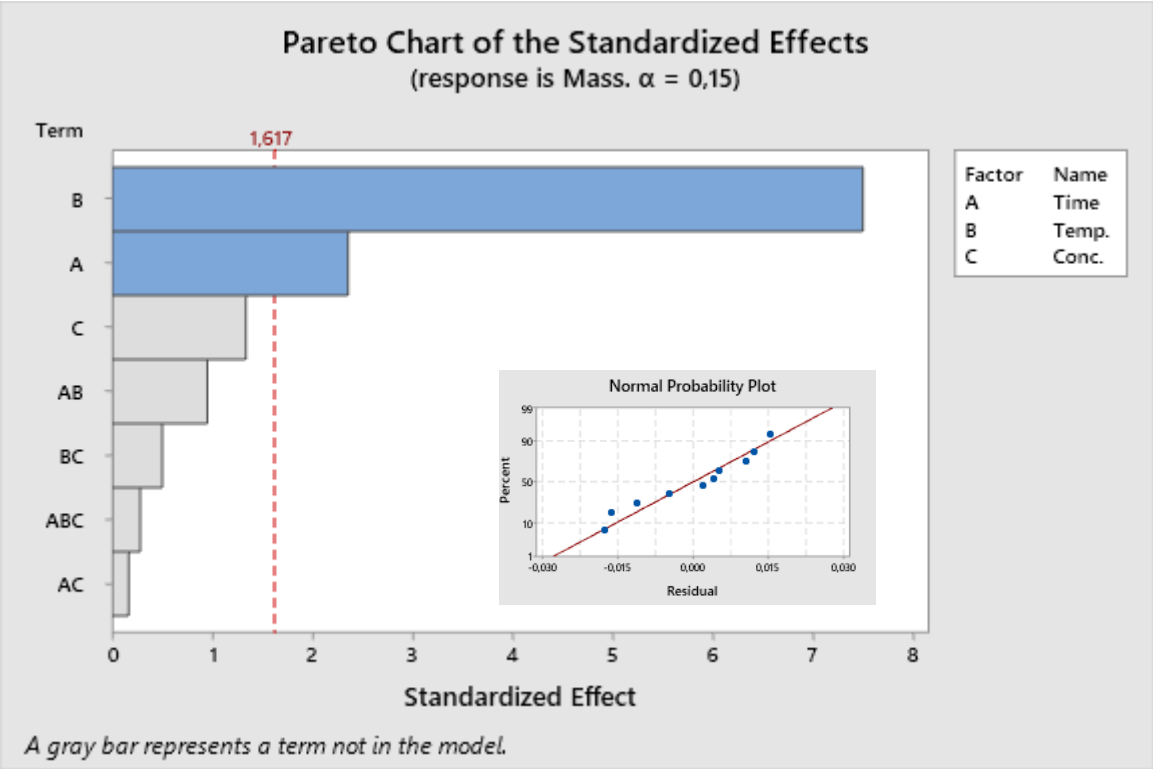

Figure 39 Pareto chart for what factors affect the mass of the absorbed liquid in bleached material. The most prominent parameter is temperature, and substantially less prominent, but still significant, is the time. HP concentration does not affect the mass of the liquid absorbed for α=0.15.

6.2 Edge Soaking

Three different failure modes could be observed when performing cap detachment, and examples of these modes can be seen in Figure 40. The initial delamination either occurs between the hot melt and the frame of the cap, between the décor layer and the clay-coat or within the paperboard.

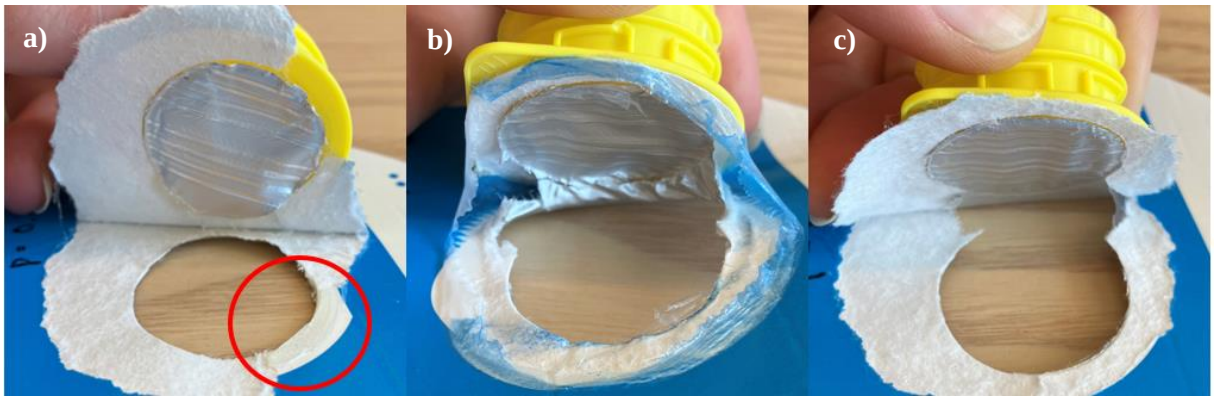

Figure 40 Failure modes for bleached material. a) cap to hot melt (circled) b) décor layer to clay coat c) within paperboard.

In the following sections, the result from each testing scenario for the two different materials studied is presented. Both the cap detachment forces, and the failure modes are discussed. Since

the method of quantifying the amount of ES present in a specimen can be performed in two ways (mass and area), plots for both parameters have been made. However, the area measurements were found to be difficult to perform accurately for small amounts of ES since it dries out quickly which leads to noise for low amounts of absorbed water, see Figure 41. Therefore, only mass plots will be displayed in the following sections. However, the plots using area as ES quantification method show similar results and can be found in the appendix, see section 11.2.

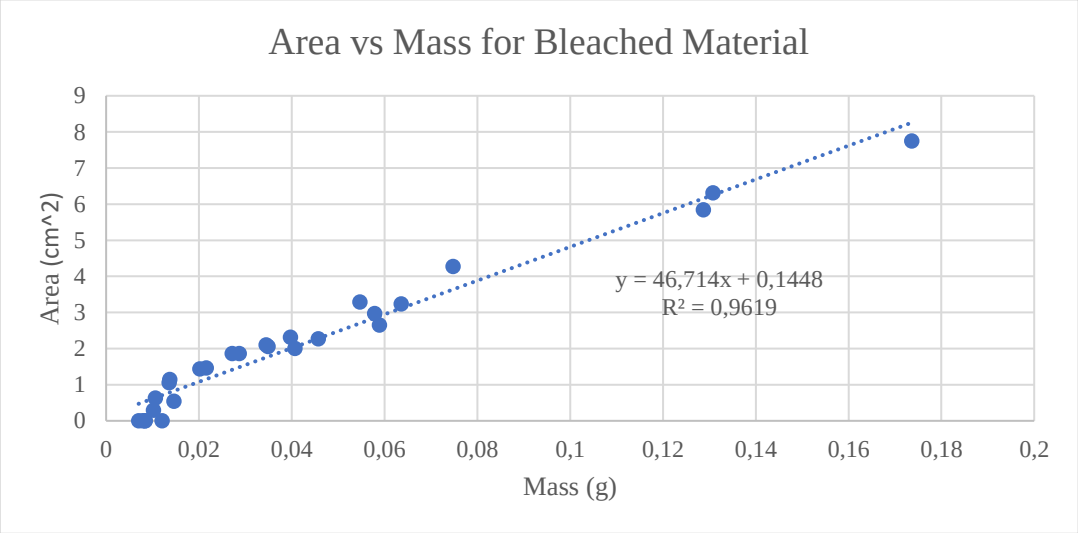

Figure 41 Example of the relationship between mass and area of the specimens of bleached material used for the wet cap attachment. Noise in the lower region is present.

6.2.1 Cap applied on and detached from specimens with wet paperboard

When applying the cap and performing cap detachment on specimens with ES still present, one can observe that the amount of force needed to remove the cap decreases with increasing degree of ES for both materials. Even very small degrees of ES results in a substantial decrease of the needed force, see Figure 42 and Figure 43.

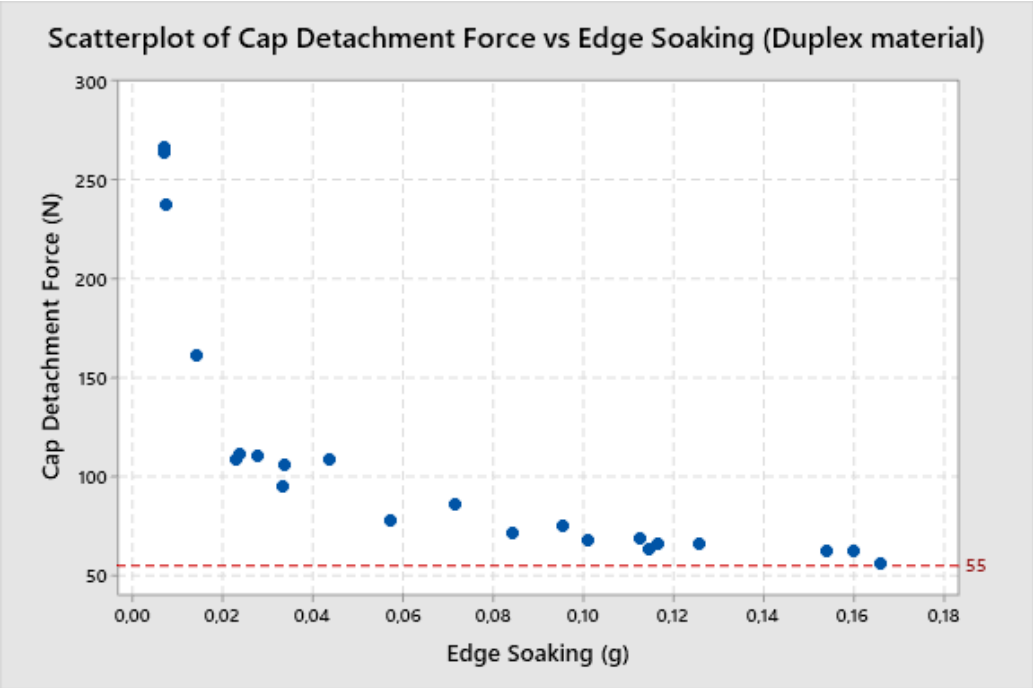

Figure 42 Cap Detachment Force vs Edge Soaking (mass) for duplex material where cap attachment and cap detachment are performed directly after the ES quantification. A rapid decrease of needed force for increasing degree of ES. A plateau of approximately 55 N is reached.

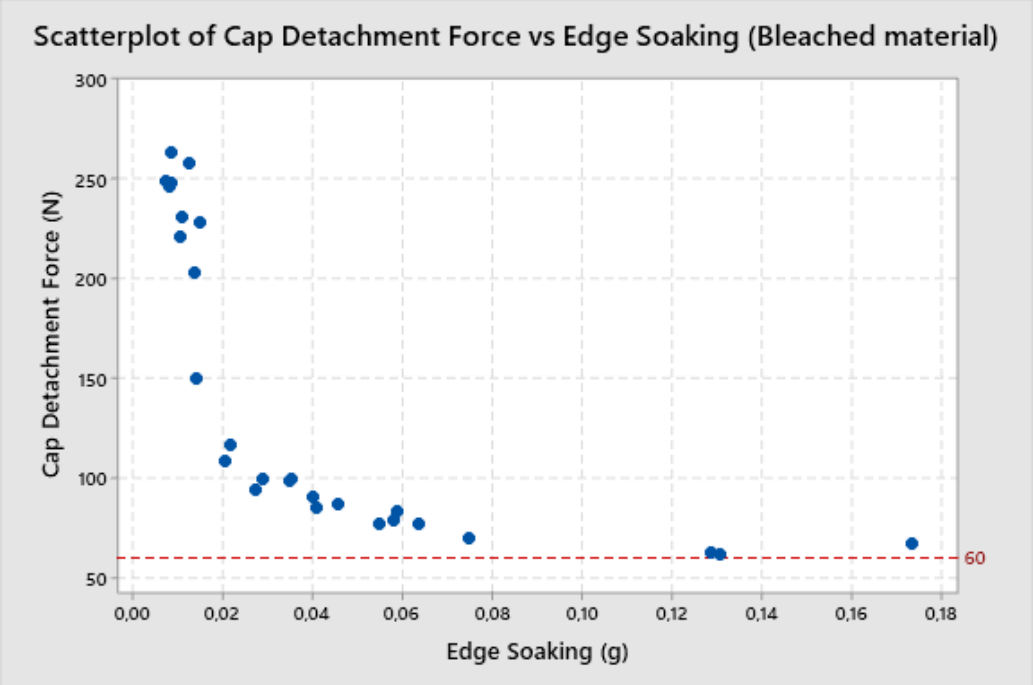

Figure 43 Cap Detachment Force vs Edge Soaking (mass) for bleached material where cap attachment and cap detachment are performed directly after the ES quantification. A rapid decrease of needed force for increasing degree of ES. A plateau of approximately 60 N is reached.

A plateau is quickly reached around 0.1 g of absorbed water with the approximate values of 55 N for duplex and 60 N for bleached material. In Figure 44, a comparison between the size of

0.0228 g and 0.1009 g ES in duplex material can be seen which approximately corresponds to the sizes corresponding to the initial force drop and the plateau.

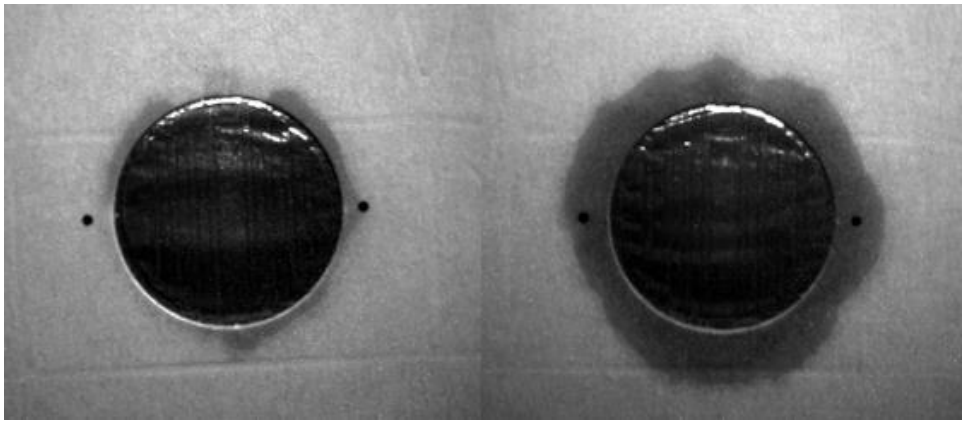

Figure 44 IR images of specimens with different amounts of ES: a) 0.0228 g b) 0.1009 g.

The failure mode for this testing scenario varies depending on the amount of ES. For specimens with large degree of ES and therefore lower cap detachment force, the delamination occurs between the décor layer and the clay coat. However, for specimens with small amount of ES (less than approximately 0.015 g) the breakage occurs between the hot melt and the frame of the cap or within the paperboard. This pattern can be found both for the duplex and the bleached material. In Figure 45 examples of décor to clay coat delamination are shown for both materials. For high amounts of ES, the material close to the PLH bends quite a bit during cap detachment.

Figure 45 Delamination between décor layer and clay-coat for large degree ES.

6.2.2 Cap applied on dry and acclimated specimens

For acclimated specimens no clear trend can be observed from the force measurements for duplex material, see Figure 46. The force needed for cap detachment stays high even for high degrees of ES. The distribution is quite narrow.

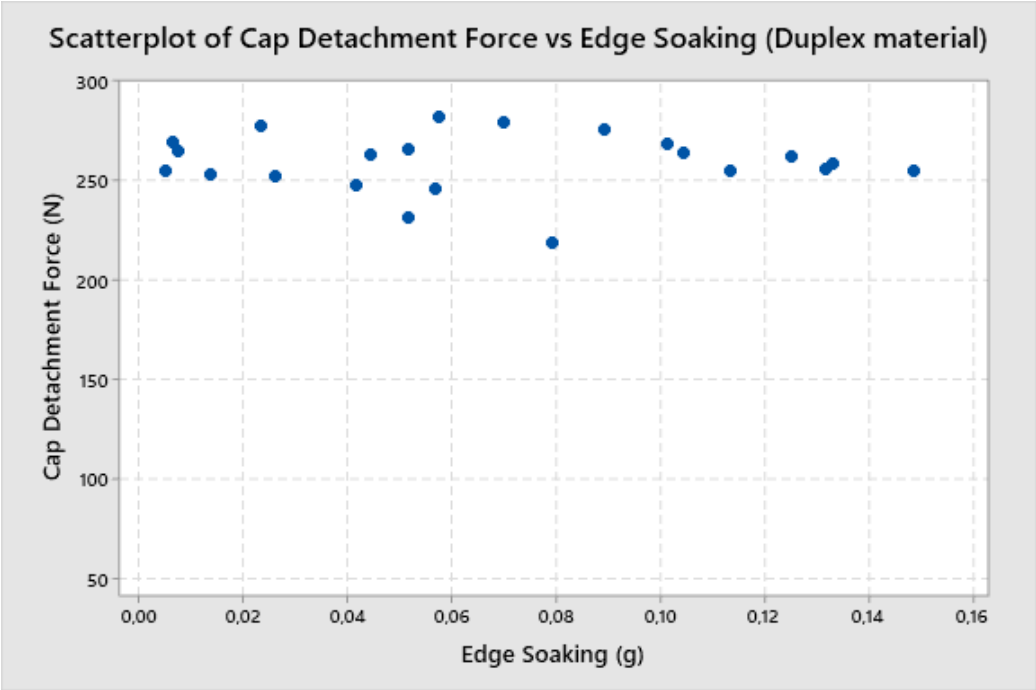

Figure 46 Cap Detachment Force vs Edge Soaking (mass) for duplex material that has been dried in a 60 ºC oven over night, followed by acclimatizing in a climate-controlled environment (23 ºC and RH 50%) for two weeks. No clear trend is observed.

For bleached material however, there are some indications that the cap detachment force decreases with increasing amount of ES. The change in needed force is not as rapid as for the specimens with wet board, and seems to halt around 200 N, see Figure 47.

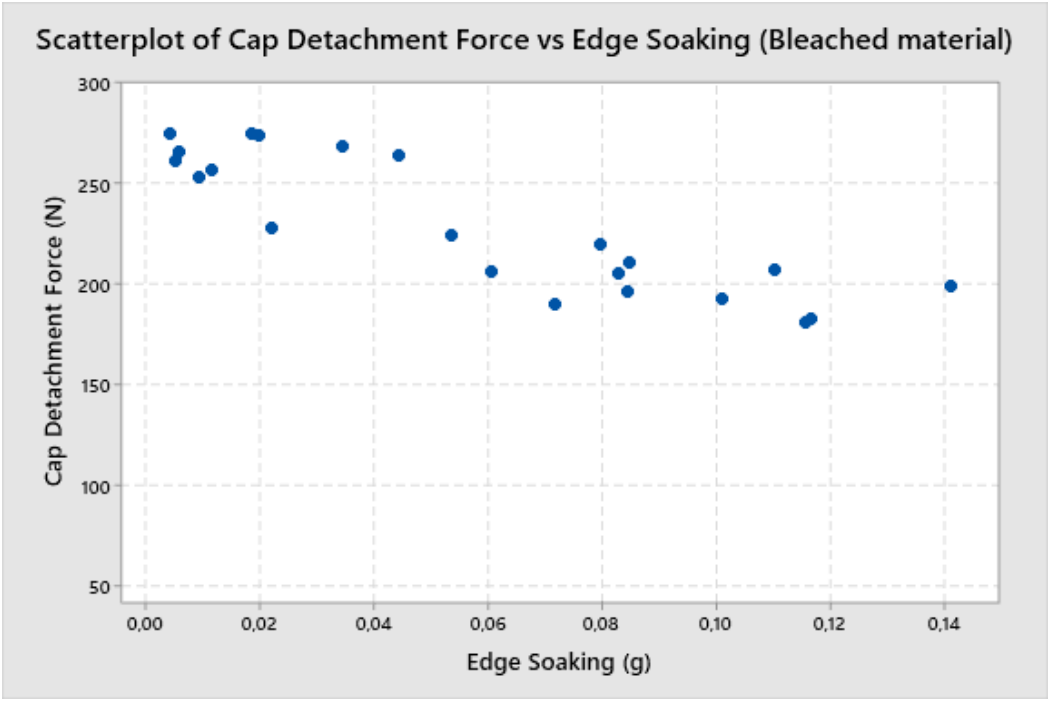

Figure 47 Cap Detachment Force vs Edge Soaking (mass) for bleached material that has been dried in a 60 ºC oven over night, followed by acclimatizing in a climate-controlled environment (23 ºC and RH 50%) for two weeks. A slightly decreasing trend for increasing degree of ES is observed.

The failure modes vary between the hot melt and the cap frame, and within the paperboard. Examples of delamination within the paperboard for both types of material can be seen in Figure 48 below.

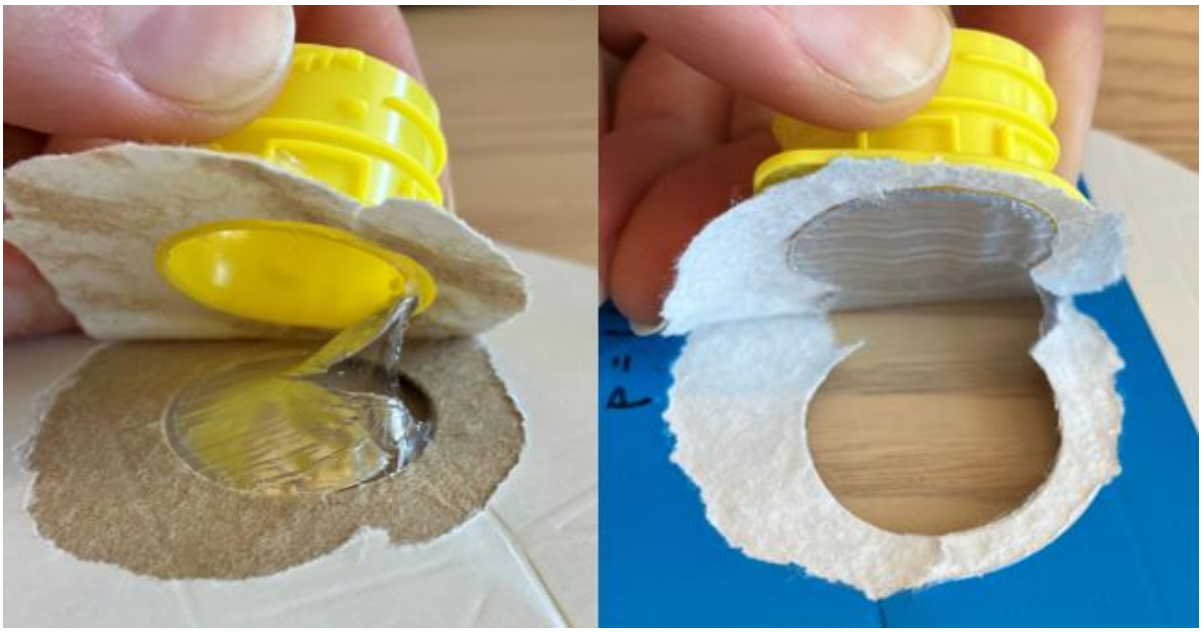

Figure 48 Delamination within the paperboard for dried out specimens.

6.2.3 Cap applied on dry non-acclimated specimens

For cap detachment performed on specimens that have been dried using an oven after ES but not acclimated, no clear trend can be observed. The force needed is stable for the investigated interval. However, the dispersion of the values is quite large for both materials. For duplex, no values under 150 N are recorded, see Figure 49. For bleached however, a few values under 150 N are recorded and one under 100 N (86 N), see Figure 50. No clear pattern can be seen for either of the materials, and the spread is more prominent than for the acclimated specimens in 6.2.2.

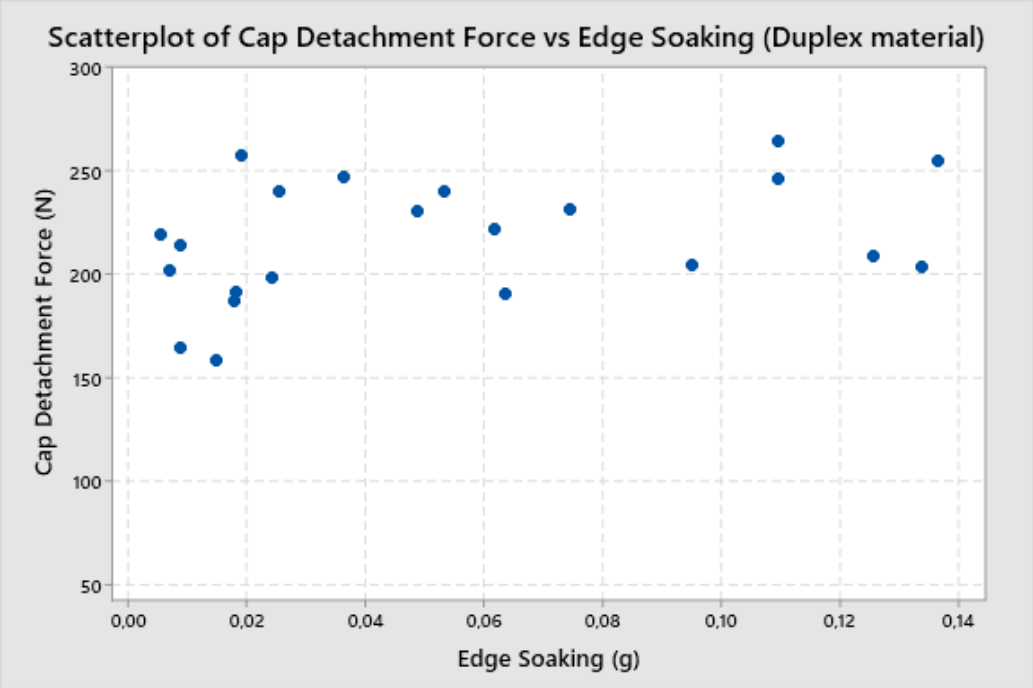

Figure 49 Cap Detachment Force vs Edge Soaking (mass) for duplex material that has been dried in a 60 ºC oven over night without acclimatizing in a climate-controlled environment afterwards. To trend is observed and the dispersion is high.

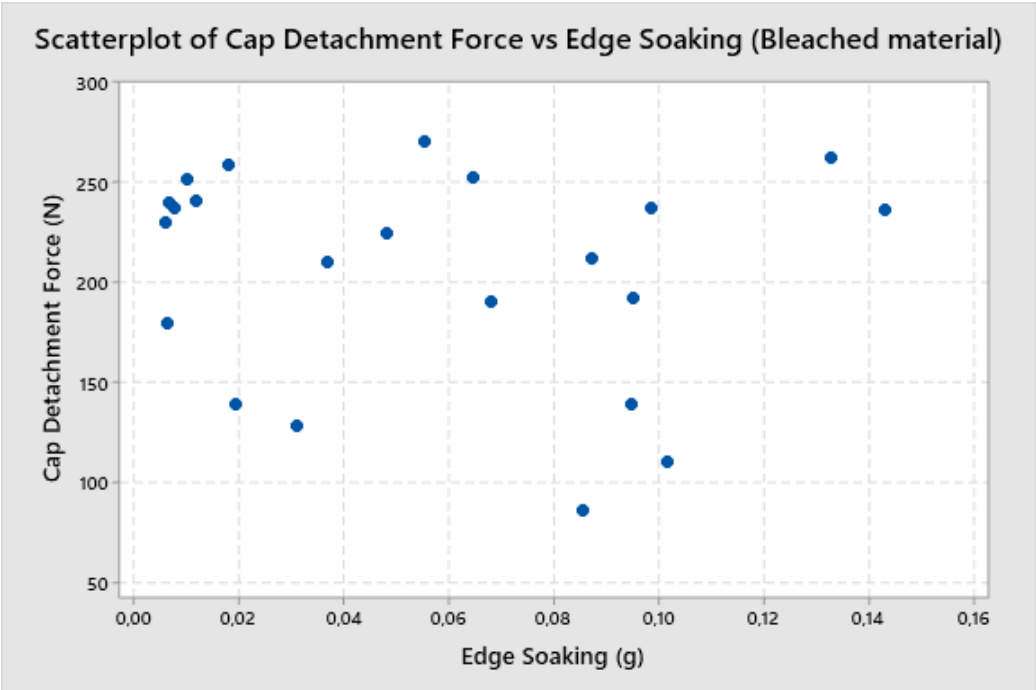

Figure 50 Cap Detachment Force vs Edge Soaking (mass) for bleached material that has been dried in a 60 ºC oven over night without acclimatizing in a climate-controlled environment afterwards. No clear trend is observed, and the dispersion is high.

7. Discussion

7.1 Edge Wicking

For all materials tested the temperature has the biggest effect on both mass of the absorbed liquid, average distance, and maximum distance. With the current test-set up that is being used in the Material Analysis laboratory, and that was used for this study it is quite hard to control the temperature over time. The lid covering the heated bath must be removed when depositing and removing the specimens from the liquid, and this lowers the temperature of the bath. To increase the robustness of the current TM one should therefore start by trying to minimize the temperature fluctuations during testing.

Even though the result indicates that the concentration of HP does have an impact on the EW, for at least some of the materials tested, it cannot be ruled out that water could be a possible replacement for HP in the TM. When using water, the risks associated with higher temperatures are fewer than for HP and it is therefore possible that similar results could be obtained by raising the temperature of water a few degrees above what is used today for the 70 ºC HP bath. It would therefore be suitable to perform additional tests to investigate this possibility.

7.2 Edge Soaking

For both materials tested both the area of the soaked material and the mass of the absorbed water can be used as quantification method for the degree of ES. However, when dealing with very small degrees of ES, mass is the most suitable method. At least when performing the tests with the equipment and resources that were used in this project. The IR-camera is in a laboratory separated from the ESEE, which in this case results in performing the ES a couple of specimens at the time. Since there is a scale in the laboratory where the ESEE equipment is located, the weighing of the specimens can be performed directly after the creation of ES for each specimen. It is however promising that a linear relationship exists between the mass of the absorbed water and the area of the soaked material, because if this method were to be further developed and used during production at some point, weighing the filled packages to obtain the amount of ES is not possible. That it would be possible to measure the area instead and therefore also gain information about the mass of the absorbed liquid is valuable information.

For the cap detachment performed on specimens with wet board a clear trend can be observed as a decrease in the force needed for cap detachment for increasing degree of ES. The force rapidly drops and then quickly reaches a plateau. That a plateau is reached is reasonable since a certain amount of force will always be needed to remove the cap. The breakage also occurs between the décor layer and the clay coat for large degrees of ES which might be a reason for why there is no prominent difference in the shape and value of the plateaus between the materials with different paperboards. It seems reasonable that the clay coat is also affected by the water and that this is the failure mode even if it was not the result expected prior to the measurements. It might be interesting to further investigate this. The bending of the paperboard indicates that the paperboard is also weakened by the water even though the breakage does not occur within it.

The results also indicate that the degree of ES does not affect the cap detachment force to a large extent once the paperboard is dried again. No permanent damage seems to occur to the

fibre network when exposing it to large amounts of water followed by drying. The wide distribution of values for the non-acclimated material strongly suggests that the acclimation of the specimens following the drying in oven is necessary to be able to minimize variations and obtain information about the paperboard. One thing to keep in mind when analyzing the results is that the PM used in the study is not stored in a climate-controlled environment prior to performing the ES. However, it is deemed that the amount of moisture in the specimens prior to ES should not affect how much water that can be introduced using the ESEE tool and therefore not of importance for this study.

Even though many of the uncertainties introduced when performing the cap attachment could be minimized by using the YuMi robot, some variations still exist. The amount of hot melt applied for each cap was improved by switching to more optimal temperatures and by exchanging some filters, but some fluctuations still appear. However, most delaminations do not occur between the hot melt and the cap, which indicates that the amount is sufficient. The placement of the cap is performed in the same location for all specimens but since the specimens have some deviations, even when using the automatic cutting table, there can be small differences on where the cap is applied in relation to the PLH. This should however not have a substantial impact on the results.

When analysing the results obtained in this study it is of importance to remember that the testing is performed on flat PM and cannot be directly compared to cap detachment measurements on packages.

8. Conclusions

8.1 Edge Wicking

The TMs measuring EW are deemed relevant and necessary to perform also in the future, and to improve them, the first step should be to try to minimize the fluctuations in temperature during testing. This since the temperature was found to have the most prominent effect on both the mass of the absorbed liquid and distance for all tested materials. From the limited experiments performed in this master thesis it cannot be ruled out that it would be possible to exchange the chemical used in the TMs from HP to water in combination with a higher temperature. Additional testing should be done, and this activity needs to be performed in collaboration with the Base Material department and preferably also the paperboard producer.

8.2 Edge Soaking

The amount of ES influences the cap detachment when the paperboard is still wet. Even for small degrees of ES the force needed decreases rapidly for both types of paperboards. For specimens where the ES has been dried out, the trend is not as clear. For the bleached material that has been acclimated after drying, there could exist a relationship, but the decrease is not as rapid as for the wet material. For the non-acclimated material as well as the acclimated duplex material, no trend is present. However, the dispersion of values is a lot higher for the non-

acclimated specimens. Therefore, there is no value in evaluating non-acclimated material in the future since the noise obscures the properties we want to examine.

9. Outlook

9.1 Edge Wicking

Short-term, effort should be made to reduce the temperature fluctuations during testing by improving the water bath set-up used.

Eventually, the activity of trying to find a replacement chemical for HP should be performed in collaboration with Base Materials and the paperboard producer. A first step could be to investigate PM that consists of paperboard without sizing to compare with the results from this study. It would also be of interest to compare the results obtained in this study with similar experiments using water and temperatures higher than 70 ºC.

9.2 Edge Soaking

Since the aim of this study was to investigate the relationship between degree of ES and cap detachment, designing the test set-up to mimic real life conditions was no priority. However, since there are indications that a relationship might exist, it would be interesting to create a set-up that more closely resembles the existing test set-up for cap detachment on packages.

Testing was performed on uniform ES defects. This is usually not how the ES appears in production and it could therefore also be of interest to investigate if and how the relationship changes for defects that are less uniform around the PLH.

That the failure mode for high degrees of ES was between the décor layer and the clay coat and not within the paperboard is a somewhat surprising result and it might therefore be of value to perform a study using ES with the clay coat layer in focus. An example of how this could be conducted is to use a material where printing is performed on the décor layer and where clay coat therefore is not used. By comparing the results of that experiment with this thesis could contribute with interesting and important insights.

10. Bibliography

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11. Attachments

11.1 Edge Wicking

11.1.1 Duplex I

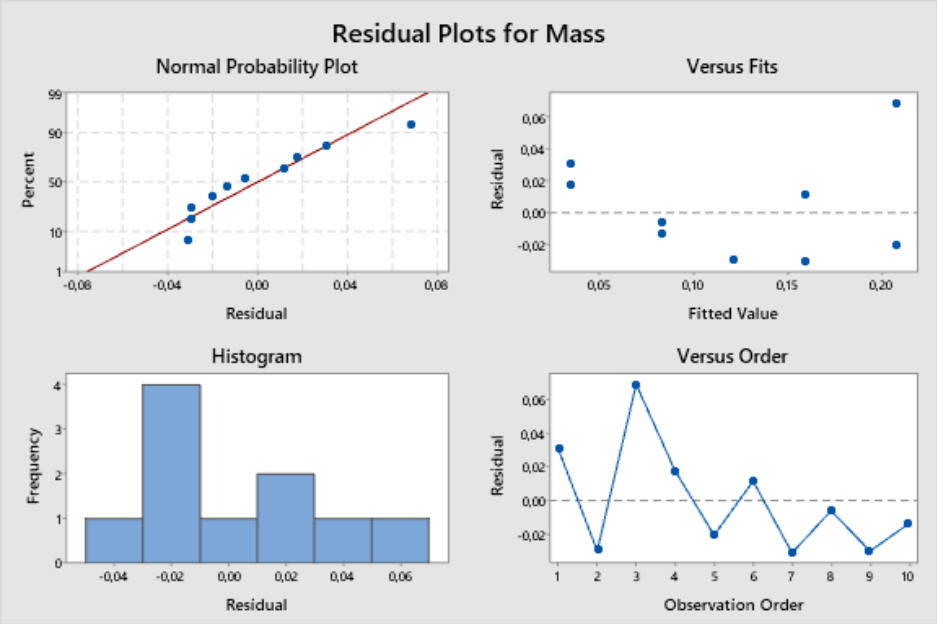

Figure 51 Residual plots for the experiment investigating the mass of the EW for duplex I material. The residual vs order plot in the bottom right corner does not correspond to the actual order that the sets are performed in. The correct residual vs order plot can be found in Figure 52 below.

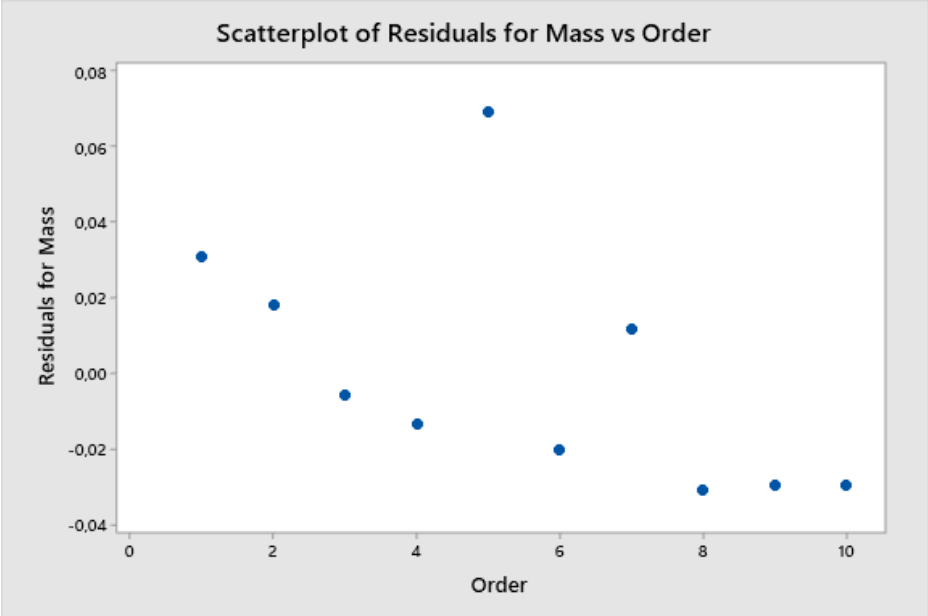

Figure 52 Residual plot for mass vs the actual order that the sets of the duplex I experiments are performed in.

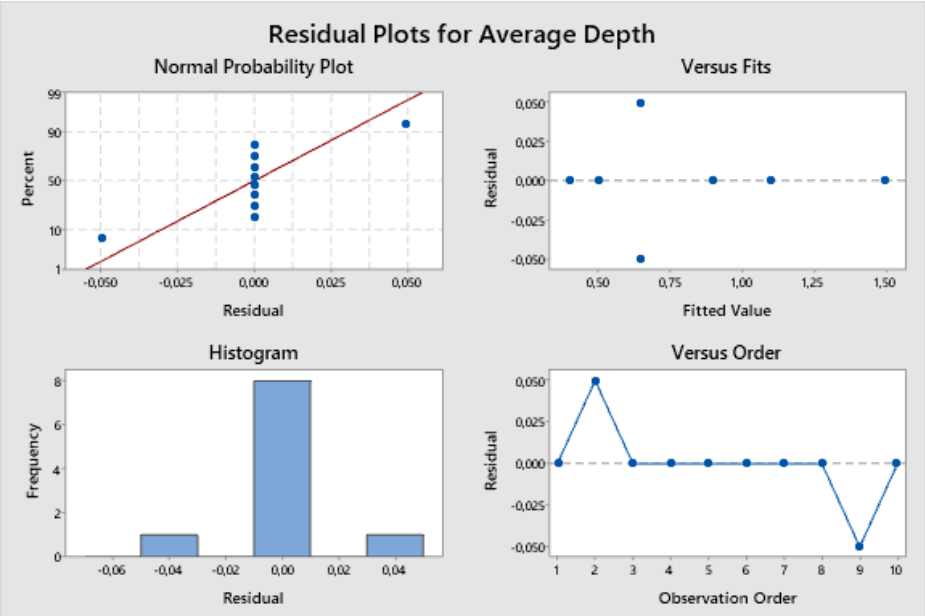

Figure 53 Residual plots for the experiment investigating the average penetration depth of the EW for duplex I material. The residual vs order plot in the bottom right corner does not correspond to the actual order that the sets are performed in. The correct residual vs order plot can be found in Figure 54 below.

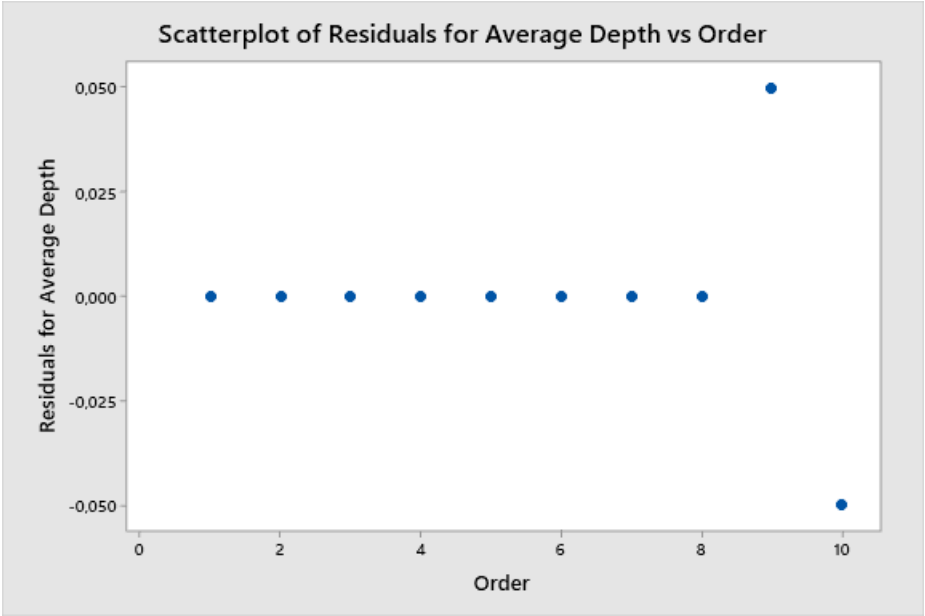

Figure 54 Residual plot for average penetration depth vs the actual order that the sets of the duplex I experiments are performed in.

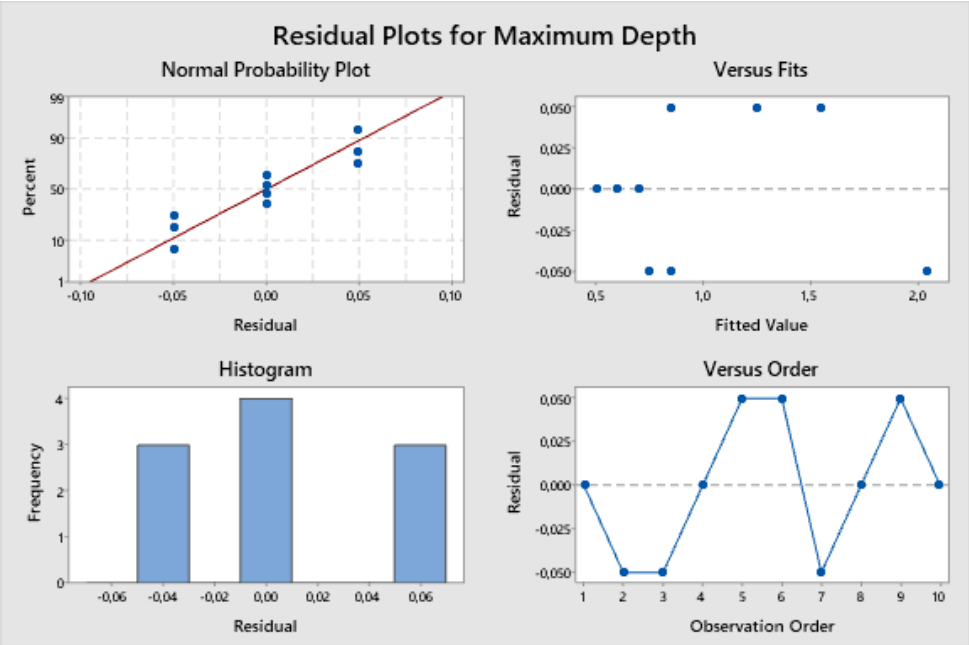

Figure 55 Residual plots for the experiment investigating the maximum penetration depth of the EW for duplex I material. The residual vs order plot in the bottom right corner does not correspond to the actual order that the sets are performed in. The correct residual vs order plot can be found in Figure 56 below.

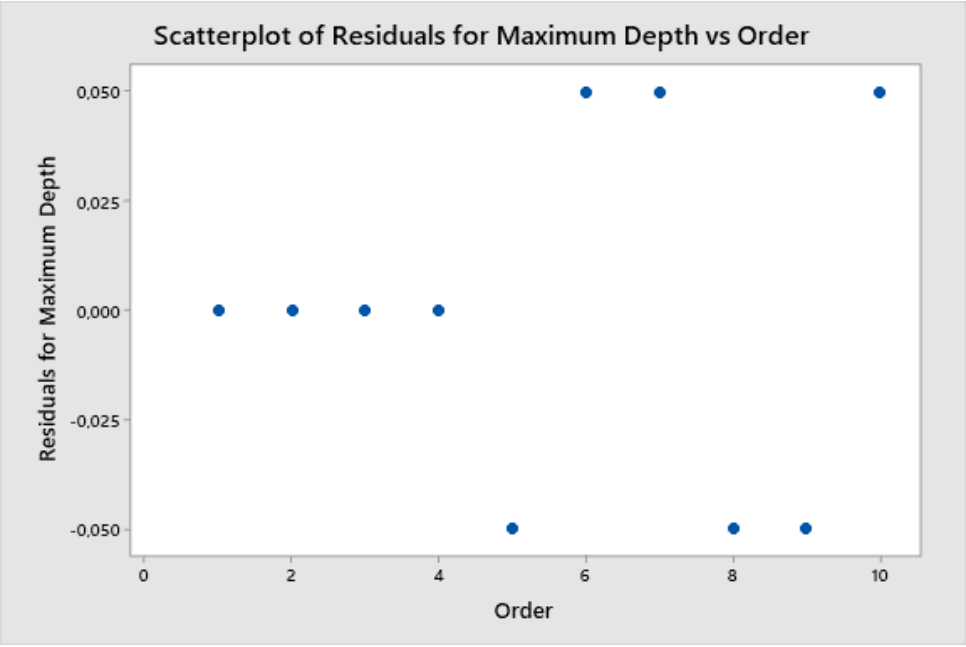

Figure 56 Residual plot for maximum penetration depth vs the actual order that the sets of the duplex I experiments are performed in.

11.1.2 Duplex II

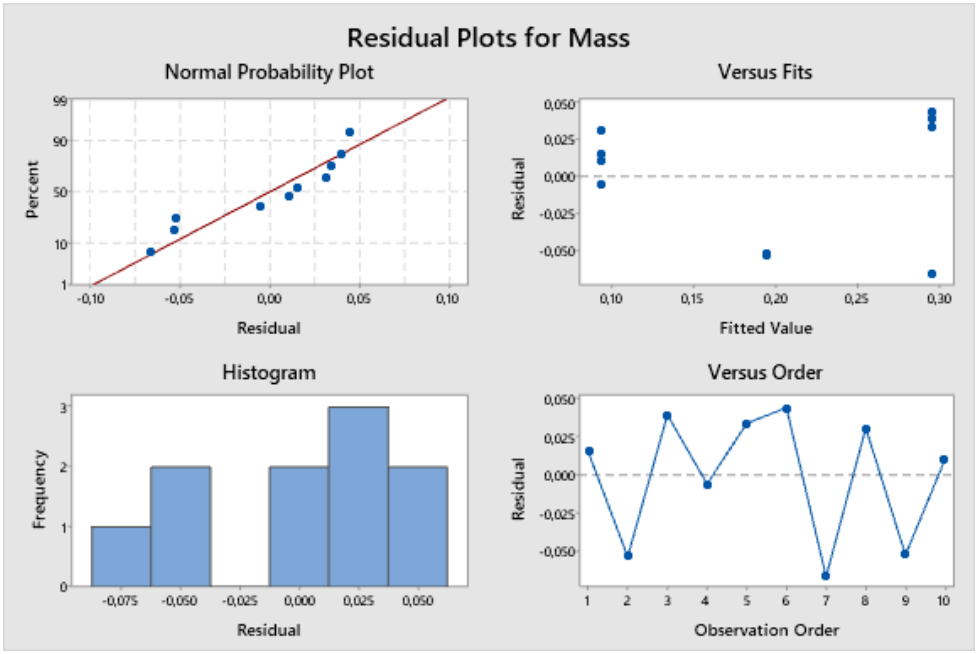

Figure 57 Residual plots for the experiment investigating the mass of the EW for duplex II material. The residual vs order plot in the bottom right corner does not correspond to the actual order that the sets are performed in. The correct residual vs order plot can be found in Figure 58 below.

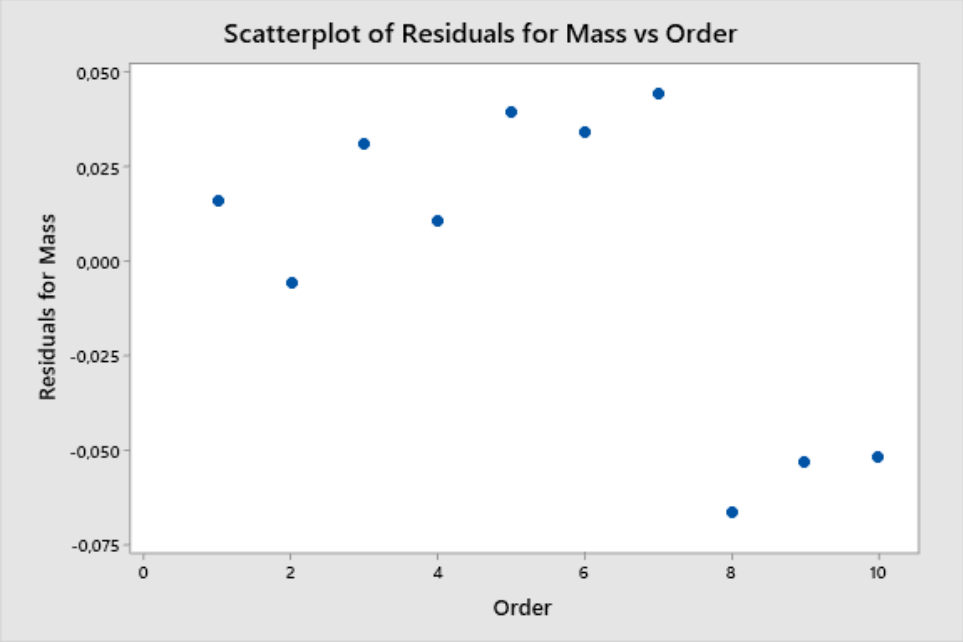

Figure 58 Residual plot for mass vs the actual order that the sets of the duplex II experiments are performed in.

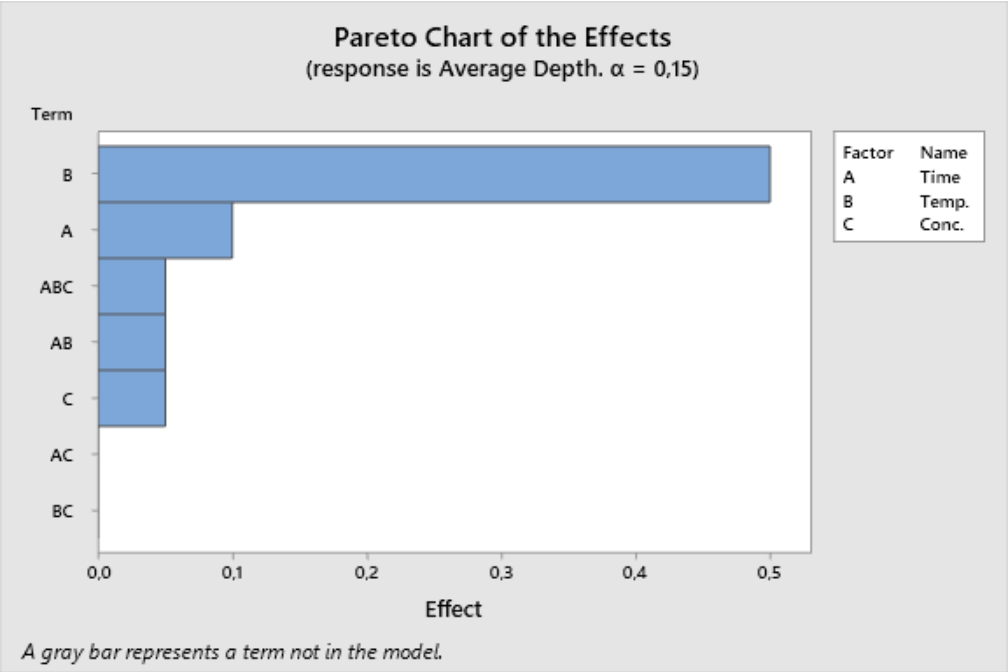

Figure 59 Pareto chart for what factors affect the average penetration depth of the EW for duplex II material. Even though most of the factors have a significant effect on the result for α=0.15, the most prominent factor, by far, is the temperature.

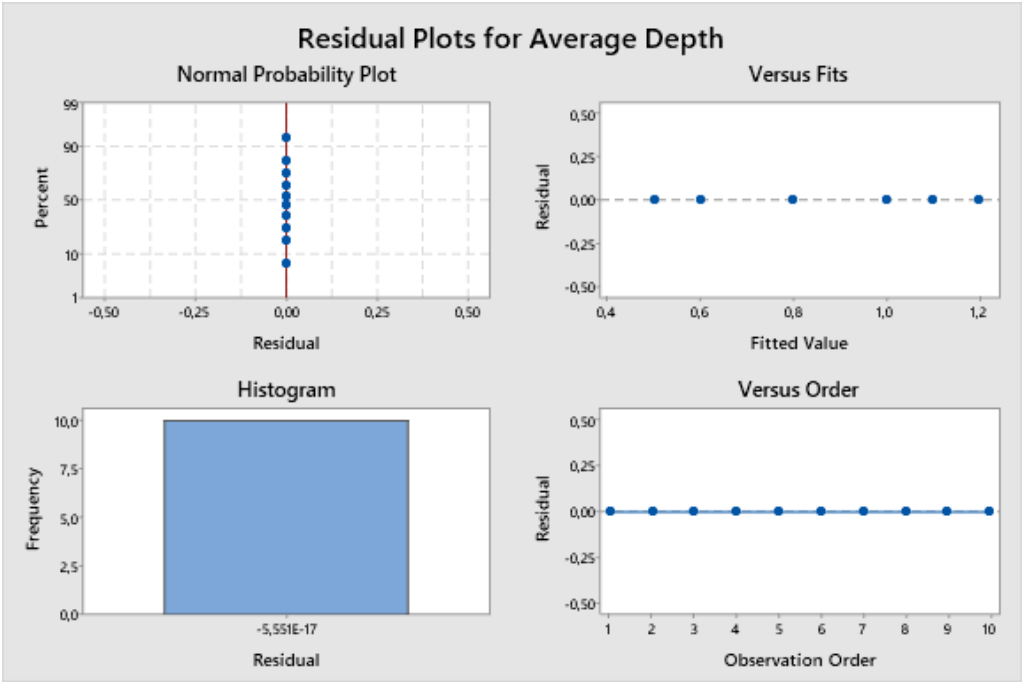

Figure 60 Residual plots for the experiment investigating the average penetration depth of the EW for duplex II material. The residual vs order plot in the bottom right corner does not correspond to the actual order that the sets are performed in. The correct residual vs order plot can be found in Figure 61 below.

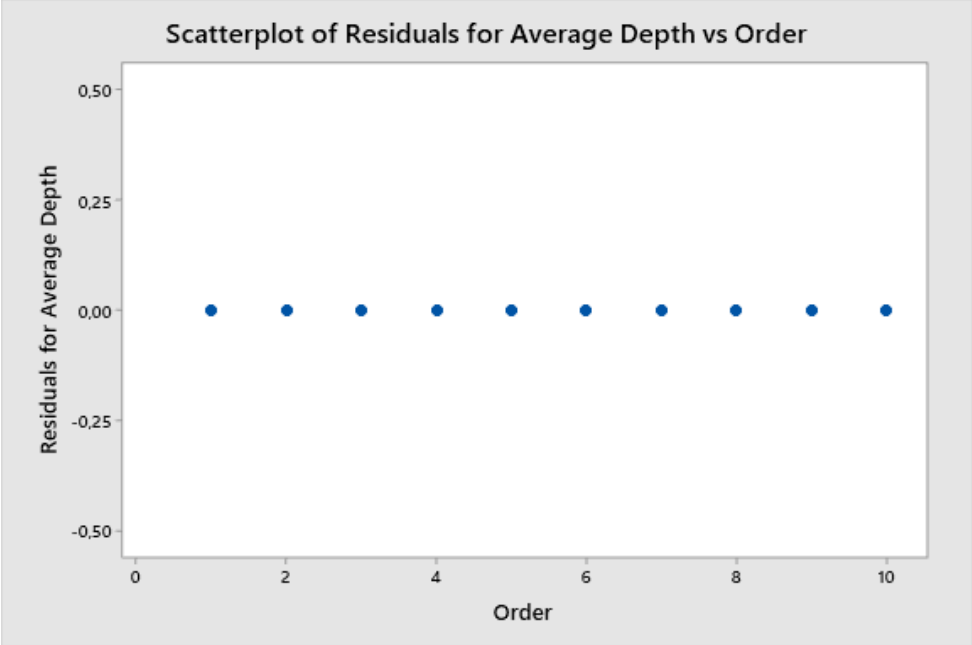

Figure 61 Residual plot for maximum penetration depth vs the actual order that the sets of the duplex II experiments are performed in.

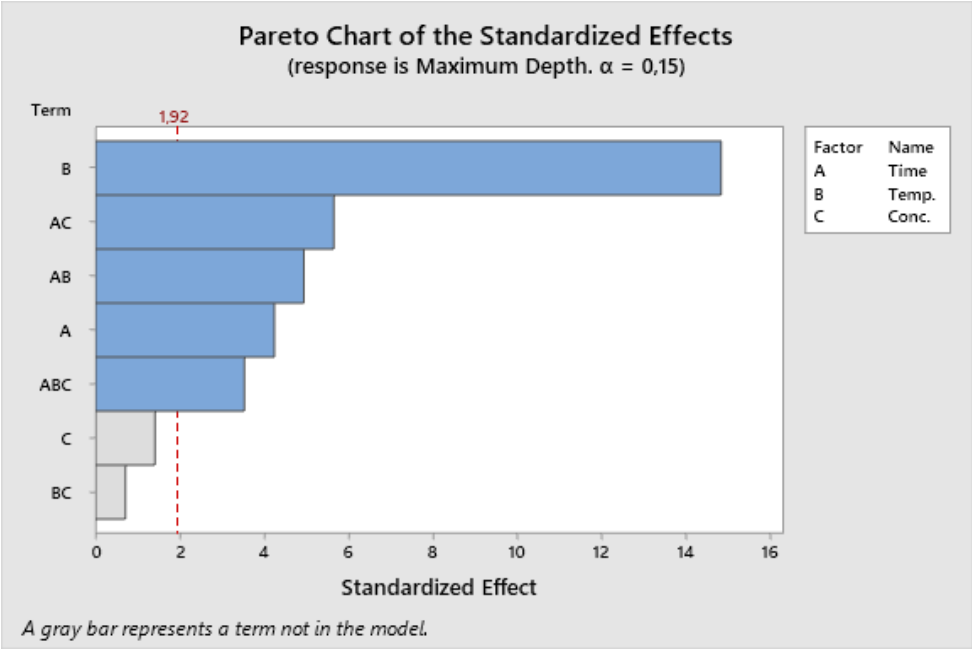

Figure 62 Pareto chart for what factors affect the maximum penetration depth of the EW for duplex II material. Even though most of the factors have a significant effect on the result for α=0.15, the most prominent factor, by far, is the temperature.

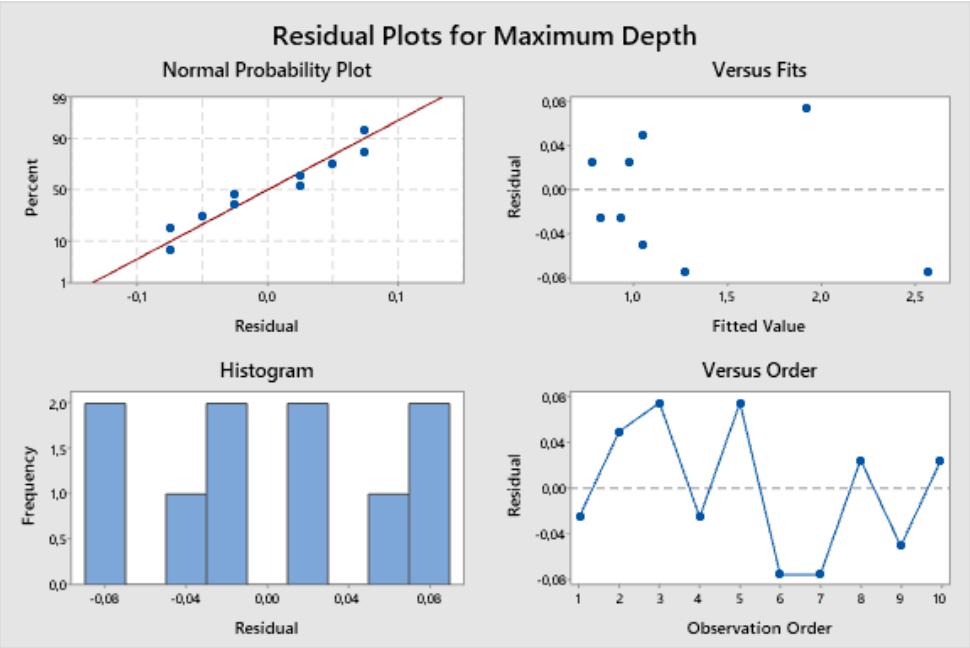

Figure 63 Residual plots for the experiment investigating the maximum depth of the EW for duplex II material. The residual vs order plot in the bottom right corner does not correspond to the actual order that the sets are performed in. The correct residual vs order plot can be found in Figure 64 below.

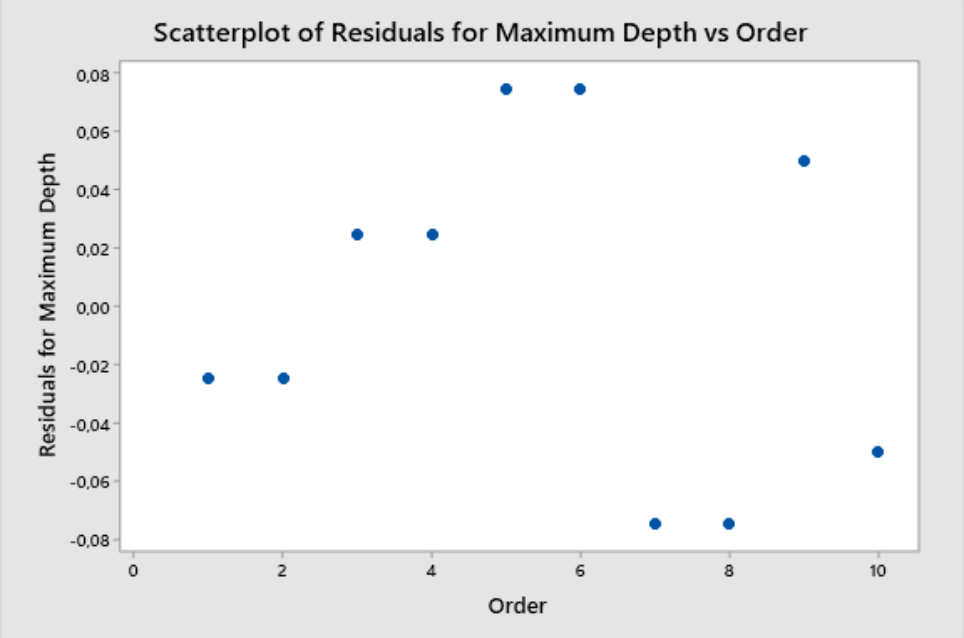

Figure 64 Residual plot for maximum penetration depth vs the actual order that the sets of the duplex II experiments are performed in.

11.1.3 Bleached

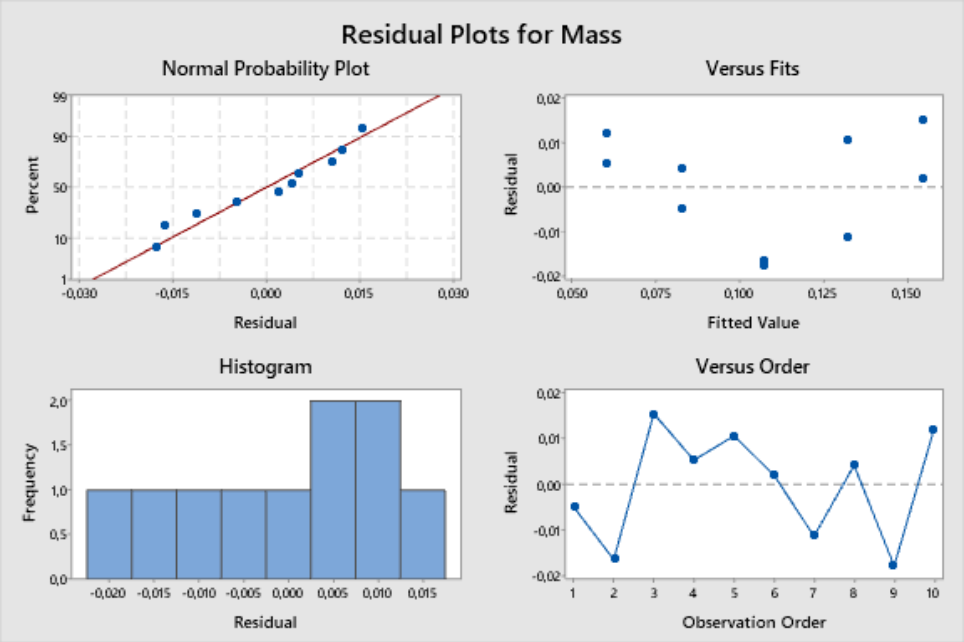

Figure 65 Residual plots for the experiment investigating the mass of the EW for bleached material. The residual vs order plot in the bottom right corner does not correspond to the actual order that the sets are performed in. The correct residual vs order plot can be found in Figure 66 below.

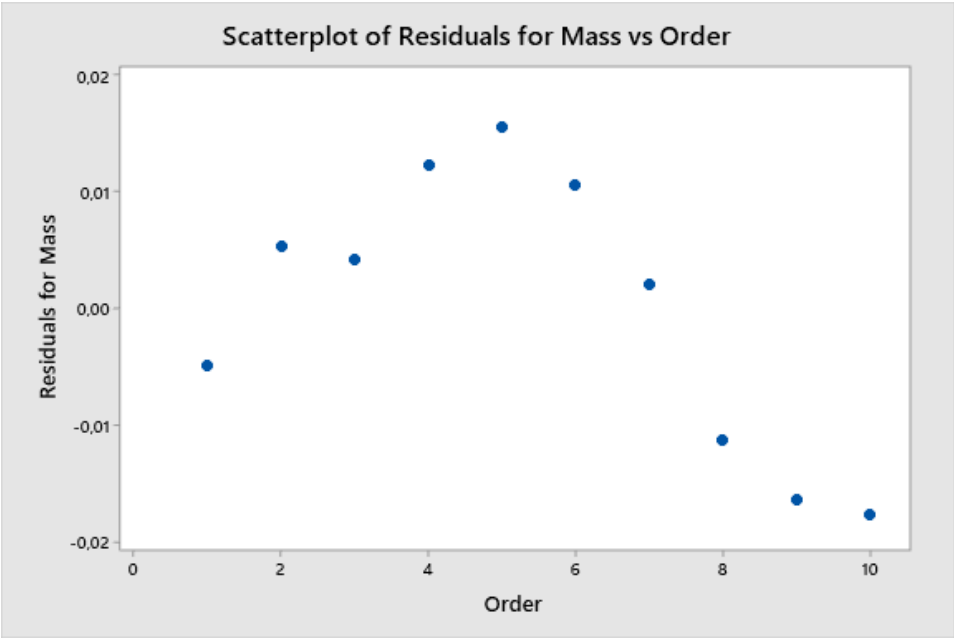

Figure 66 Residual plot for mass vs the actual order that the sets of the bleached experiments are performed in.

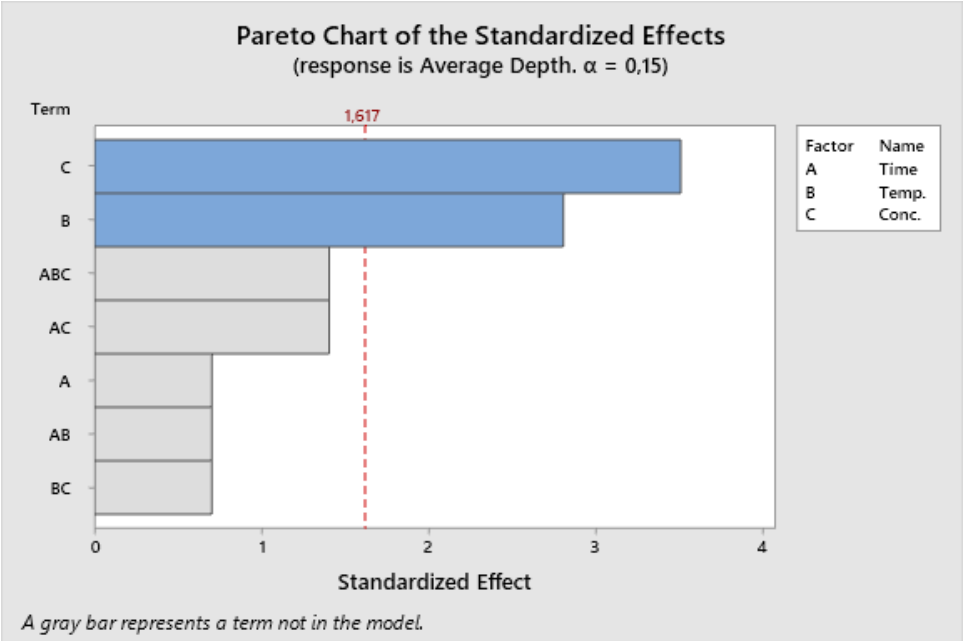

Figure 67 Pareto chart for what factors affect the average penetration depth of the EW for bleached material. For α=0.15, both HP concentration and temperature have a significant effect, but the most prominent factor is the HP concentration which differs from the other results where temperature is the most prominent factor.

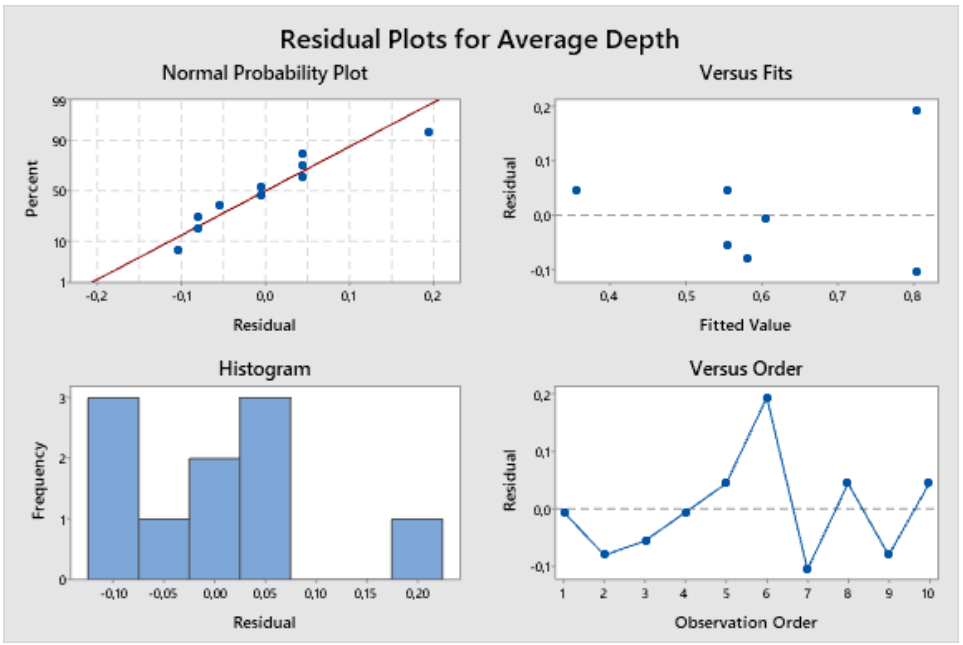

Figure 68 Residual plots for the experiment investigating the average penetration depth of the EW for bleached material. The residual vs order plot in the bottom right corner does not correspond to the actual order that the sets are performed in. The correct residual vs order plot can be found in Figure 69 below.

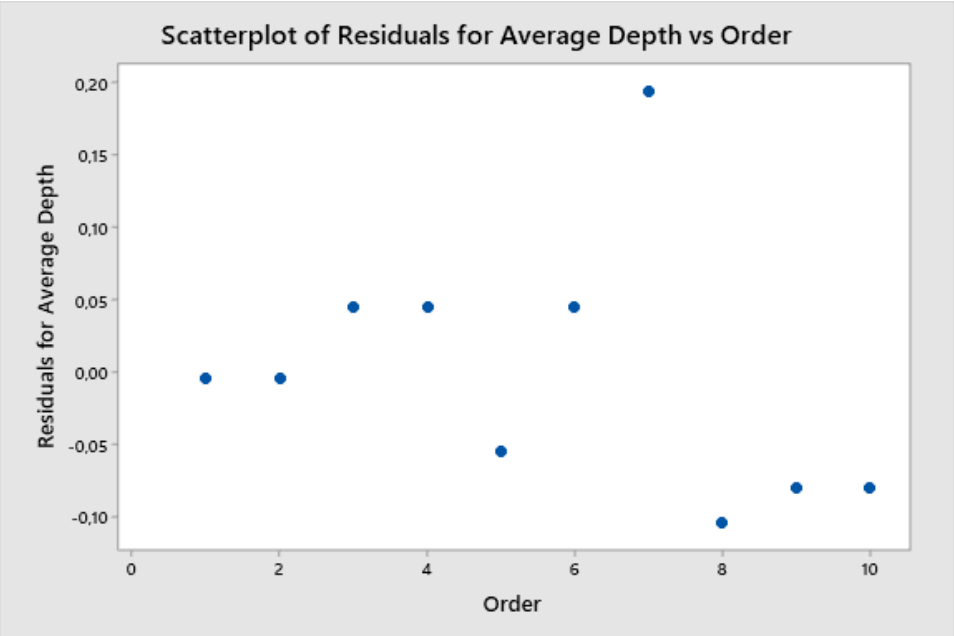

Figure 69 Residual plot for average penetration depth vs the actual order that the sets of the bleached experiments are performed in.

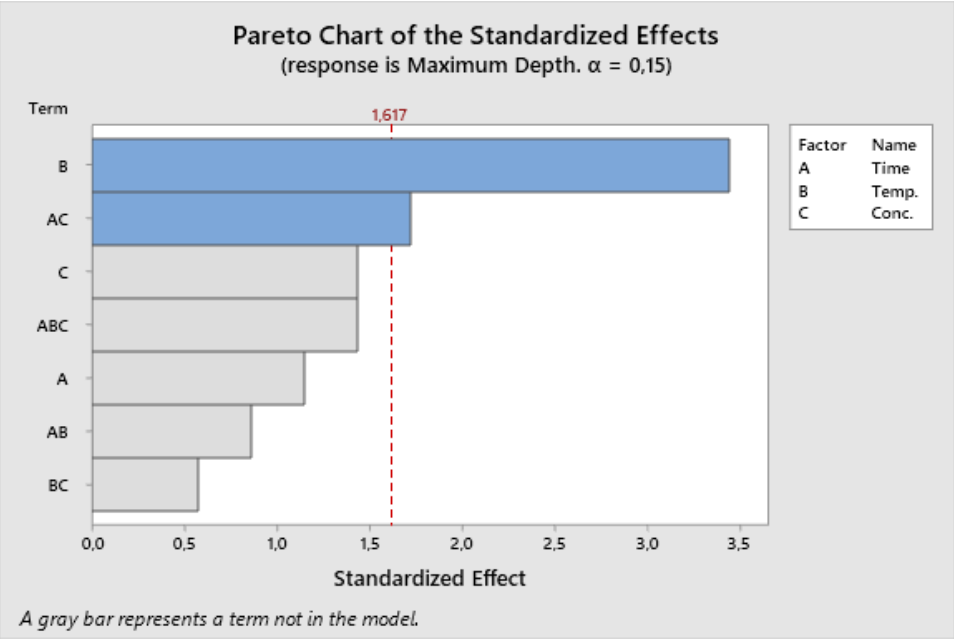

Figure 70 Pareto chart for what factors affect the maximum penetration depth of the EW for bleached material. Both temperature and the confounding factor of time and HP concentration have a significant effect on the result using α=0.15. However, the most prominent factor, by far, is the temperature.

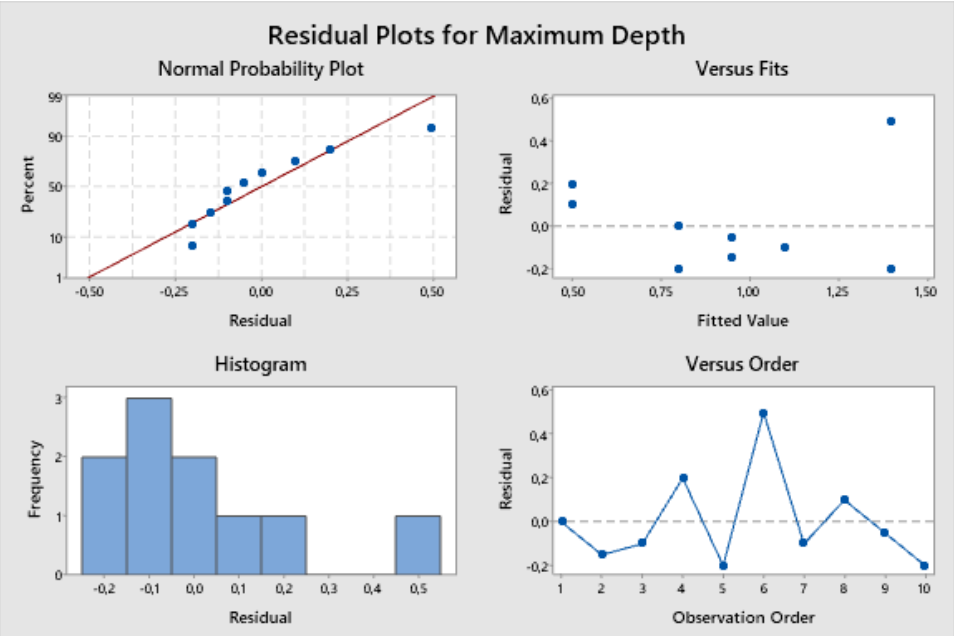

Figure 71 Residual plots for the experiment investigating the maximum penetration depth of the EW for bleached material. The residual vs order plot in the bottom right corner does not correspond to the actual order that the sets are performed in. The correct residual vs order plot can be found in Figure 72 below.

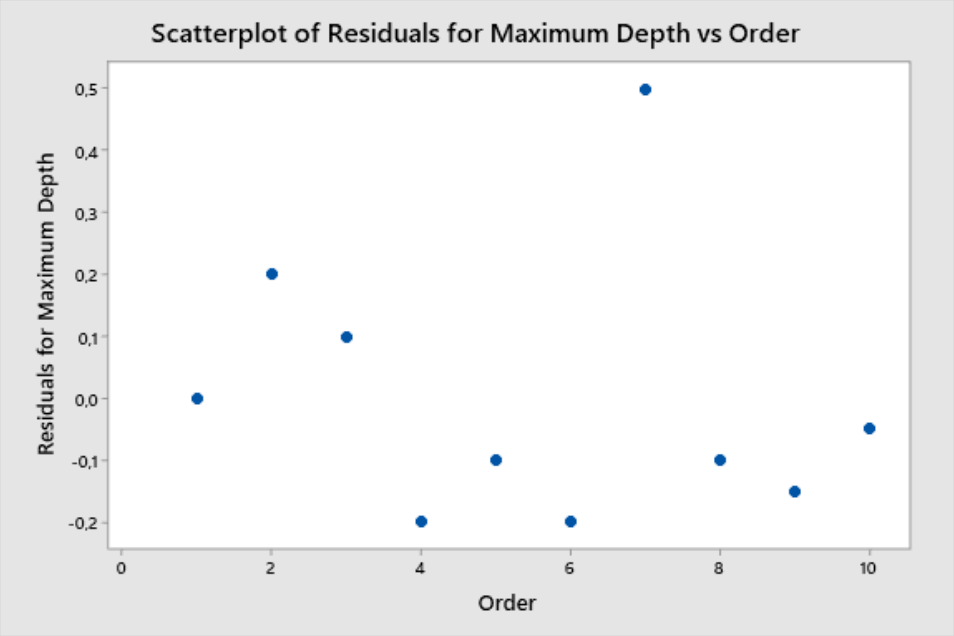

Figure 72 Residual plot for maximum penetration depth vs the actual order that the sets of the bleached experiments are performed in.

11.2 Edge Soaking

11.2.1 Cap applied on and detached from specimens with wet paperboard

Table 8 Compiled parameters for wet duplex specimens.

Wet Cap Detachment – Duplex Material					
	Mass (g)	**Area (cm^2)**	**Time (s)**	**Pressure (bar)**	**Cap detachment force (N)**
1	0.0069	0	0.1	0.5	264
2	0.0138	0.98	0.1	0.75	161
3	0.0275	1.45	0.1	1	110
4	0.0435	2.33	0.5	0.5	108
5	0.0228	1.26	0.3	0.5	108
6	0.0235	1.39	0.2	0.5	111
7	0.0073	0	0.1	0.5	237
8	0.0067	0	0.05	0.5	266
9	0.0333	1.68	0.2	0.75	95.2
10	0.0335	1.68	0.2	1	106
11	0.0842	3.7	0.4	1	71
12	0.1127	5.08	0.6	1	68.5
13	0.1166	5.38	0.8	1	65.4
14	0.1661	7.12	1	1	55.4
15	0.16	6.69	0.9	1	61.7
16	0.0716	3.22	0.3	1	85.9
17	0.0569	2.77	0.25	1	77.6
18	0.0952	4.39	0.5	1	74.9
19	0.1539	6.5	0.55	1	61.7
20	0.1147	5.11	0.52	1	62.7
21	0.1009	4.9	0.53	1	67.7
22	0.1256	5.32	0.54	1	65.8

Table 9 Compiled parameters for wet bleached specimens.

Wet Cap Detachment – Bleached Material					
	Mass (g)	**Area (cm^2)**	**Time (s)**	**Pressure (bar)**	**Cap detachment force (N)**
1	0.0121	0	0.1	0.5	258
2	0.0084	0	0.05	0.5	248
3	0.0079	0	0.03	0.5	246
4	0.0084	0	0.02	1	263
5	0.007	0	0.01	0.5	249
6	0.0102	0.29	0.1	0.75	221
7	0.0146	0.54	0.1	1	228
8	0.0407	2	0.3	1	85
9	0.0636	3.23	0.5	1	76.5
10	0.0579	2.97	0.7	1	78.3
11	0.0216	1.46	0.2	1	116
12	0.0287	1.86	0.25	1	99.2
13	0.0349	2.05	0.27	1	99.6
14	0.0345	2.1	0.28	1	98.4
15	0.0589	2.65	0.4	1	83.2
16	0.0457	2.27	0.35	1	86.9
17	0.0397	2.31	0.37	1	90.5
18	0.0272	1.86	0.15	1	94
19	0.0136	1.05	0.12	1	203
20	0.0202	1.43	0.135	1	108
21	0.0137	1.14	0.128	1	150
22	0.0106	0.63	0.13	1	231
23	0.0547	3.29	0.7	1	76.9
24	0.0748	4.27	1.3	1	69.4
25	0.1287	5.84	2	1	62.7
26	0.1308	6.31	2.5	1	61.2
27	0.1736	7.74	3.2	1	66.8

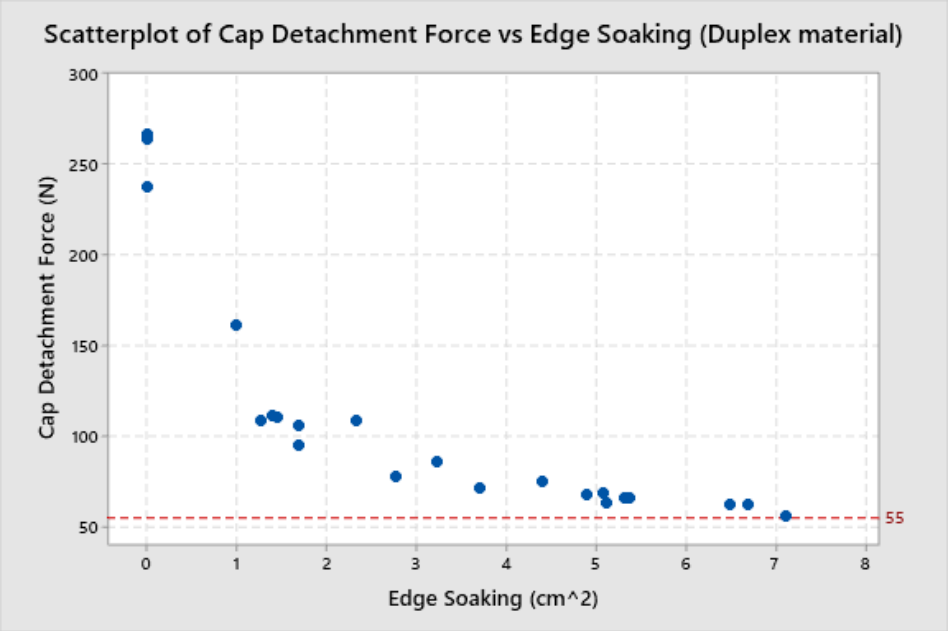

Figure 73 Cap Detachment Force vs Edge Soaking (area) for duplex material that cap attachment and cap detachment are performed directly after the ES quantification.

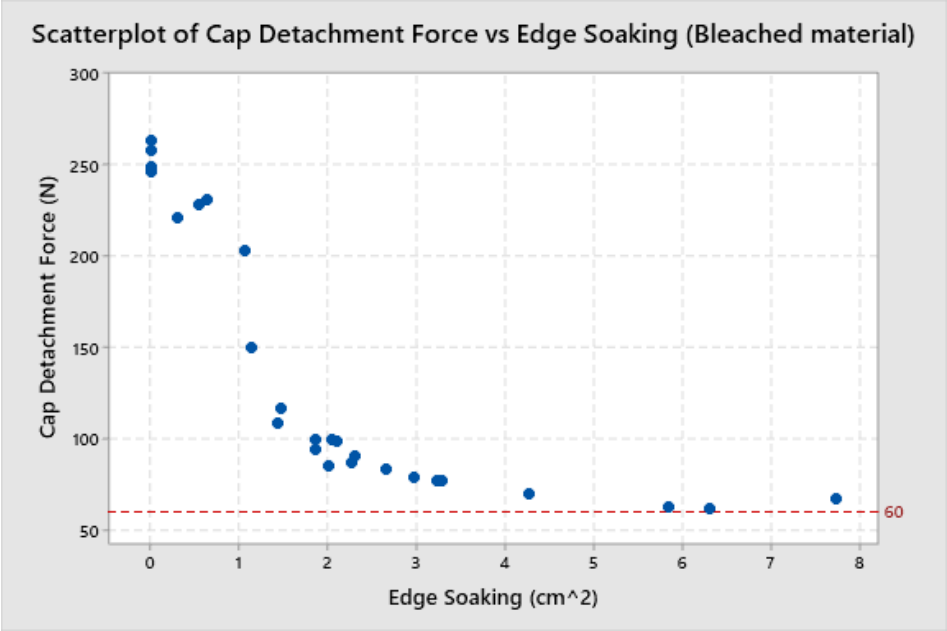

Figure 74 Cap Detachment Force vs Edge Soaking (area) for bleached material that cap attachment and cap detachment are performed directly after the ES quantification.

11.2.2 Cap applied on dry and acclimated specimens

Table 10 Compiled parameters for dried, acclimated duplex specimens.

Acclimated Cap Detachment – Duplex Material					
	Mass (g)	**Area (cm^2)**	**Time (s)**	**Pressure (bar)**	**Cap detachment force (N)**
1	0.0075	0	0.1	0.5	265
2	0.0063	0	0.05	0.75	269
3	0.0261	1.21	0.1	0.75	252
4	0.0049	0	0.05	1	255
5	0.0137	1	0.1	1	253
6	0.1136	4.84	0.5	1	255
7	0.0517	2.25	0.2	1	231
8	0.0517	2.54	0.15	1	266
9	0.0415	1.88	0.1	1	248
10	0.0233	1.31	0.08	1	277
11	0.0444	2.19	0.12	1	263
12	0.1043	4.72	0.4	1	264
13	0.1333	5.56	0.6	1	258
14	0.1318	6.22	0.8	1	256
15	0.1487	6.66	1.2	1	255
16	0.0891	4.11	0.55	1	276
17	0.1252	5.29	0.52	1	262
18	0.1012	4.38	0.5	1	268
19	0.0576	2.86	0.3	1	282
20	0.0568	2.53	0.2	1	246
21	0.0794	3.79	0.4	1	219
22	0.0699	3.3	0.45	1	279

Table 11 Compiled parameters for dried, acclimated bleached specimens.

Acclimated Cap Detachment – Bleached Material					
	Mass (g)	**Area (cm^2)**	**Time (s)**	**Pressure (bar)**	**Cap detachment force (N)**
1	0.0092	0.08	0.1	0.75	253
2	0.004	0	0.05	0.75	275
3	0.0058	0.39	0.075	0.75	266
4	0.0051	0	0.05	1	261
5	0.0114	0.75	0.1	1	257
6	0.0218	1.55	0.15	1	228
7	0.0183	1.47	0.18	1	275
8	0.0197	1.35	0.2	1	274
9	0.0441	2.31	0.3	1	264
10	0.0345	2.31	0.4	1	268
11	0.0534	2.98	0.5	1	224
12	0.0605	3.33	0.8	1	206
13	0.0829	4.05	1	1	205
14	0.0843	4.36	1.2	1	196
15	0.1102	5.13	1.5	1	207
16	0.1167	5.52	1.8	1	183
17	0.1156	5.75	2	1	181
18	0.1413	6.52	2.3	1	199
19	0.0718	3.89	0.9	1	190
20	0.0798	4.29	1.35	1	220
21	0.0848	4.37	1.4	1	211
22	0.1011	4.79	1.5	1	193

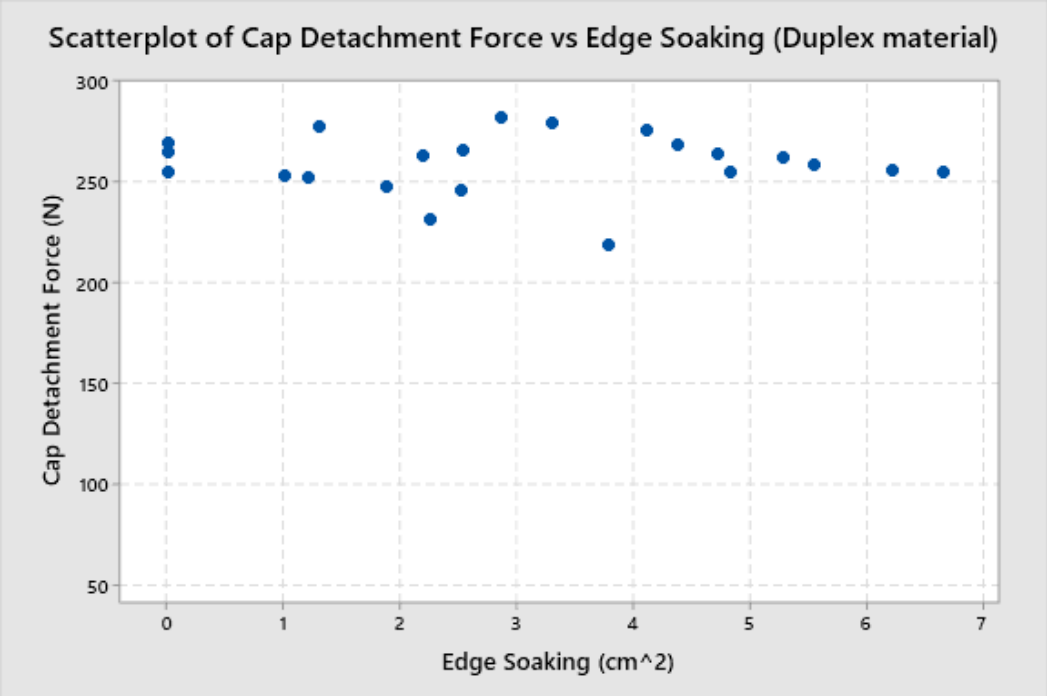

Figure 75 Cap Detachment Force vs Edge Soaking (area) for duplex material that has been dried in a 60 ºC oven over night, followed by acclimatizing in a climate-controlled environment for two weeks.

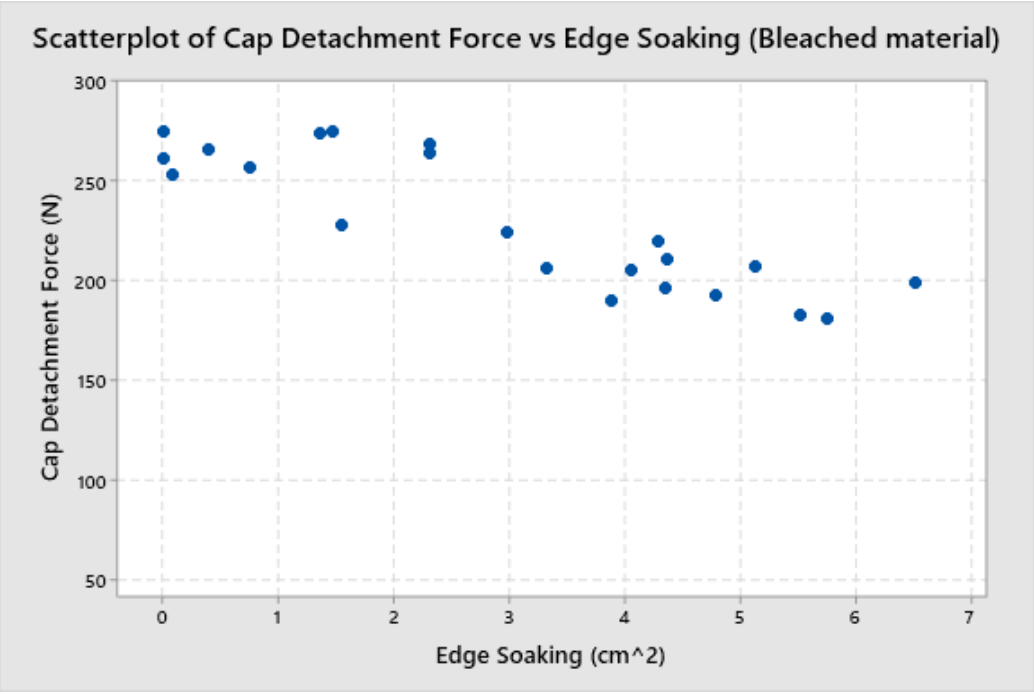

Figure 76 Cap Detachment Force vs Edge Soaking (area) for bleached material that has been dried in a 60 ºC oven over night, followed by acclimatizing in a climate-controlled environment for two weeks.

11.2.3 Cap applied on dry and non-acclimated specimens

Table 12 Compiled parameters for dried, non-acclimated duplex specimens.

Non-Acclimated Cap Detachment – Duplex Material					
	Mass (g)	**Area (cm^2)**	**Time (s)**	**Pressure (bar)**	**Cap detachment force (N)**
1	0.009	0	0.1	0.5	164
2	0.0072	0	0.05	0.5	202
3	0.0057	0.08	0.05	0.5	219
4	0.0193	1.22	0.1	0.75	257
5	0.009	0	0.05	0.75	214
6	0.015	0.92	0.05	1	158
7	0.0179	0.84	0.06	1	187
8	0.0254	1.44	0.08	1	240
9	0.0243	1.31	0.1	1	198
10	0.0183	0.89	0.12	1	191
11	0.0363	1.93	0.14	1	247
12	0.0488	2.41	0.16	1	230
13	0.0534	2.46	0.18	1	240
14	0.0619	2.51	0.2	1	222
15	0.0638	3.08	0.23	1	190
16	0.0747	3.78	0.3	1	231
17	0.1367	5.71	0.5	1	255
18	0.1098	5.17	0.6	1	246
19	0.1096	5.03	0.6	1	264
20	0.0953	4.3	0.5	1	204
21	0.1257	5.57	0.7	1	209
22	0.134	5.87	1	1	203

Table 13 Compiled parameters for dried, non-acclimated bleached specimens.

Non-Acclimated Cap Detachment – Bleached Material					
	Mass (g)	**Area (cm^2)**	**Time (s)**	**Pressure (bar)**	**Cap detachment force (N)**
1	0.0065	0	0.1	0.5	240
2	0.0077	0	0.08	0.5	237
3	0.0059	0	0.05	0.5	230
4	0.0116	0.37	0.1	0.75	241
5	0.0098	0	0.1	1	251
6	0.018	0.64	0.1	1	259
7	0.0552	3	0.5	1	270
8	0.0856	3.83	1	1	85.8
9	0.0872	4.57	1.5	1	212
10	0.1327	5.99	2	1	262
11	0.143	6.58	2.5	1	236
12	0.0063	0	0.02	1	179
13	0.0194	1.37	0.1	1	139
14	0.0481	2.52	0.3	1	224
15	0.0308	1.81	0.2	1	128
16	0.0369	2.14	0.25	1	210
17	0.0681	3.8	0.8	1	190
18	0.0647	3.63	0.9	1	252
19	0.0985	4.32	1	1	237
20	0.095	4.48	1.2	1	192
21	0.0949	4.9	1.5	1	139
22	0.1017	5.18	1.8	1	110

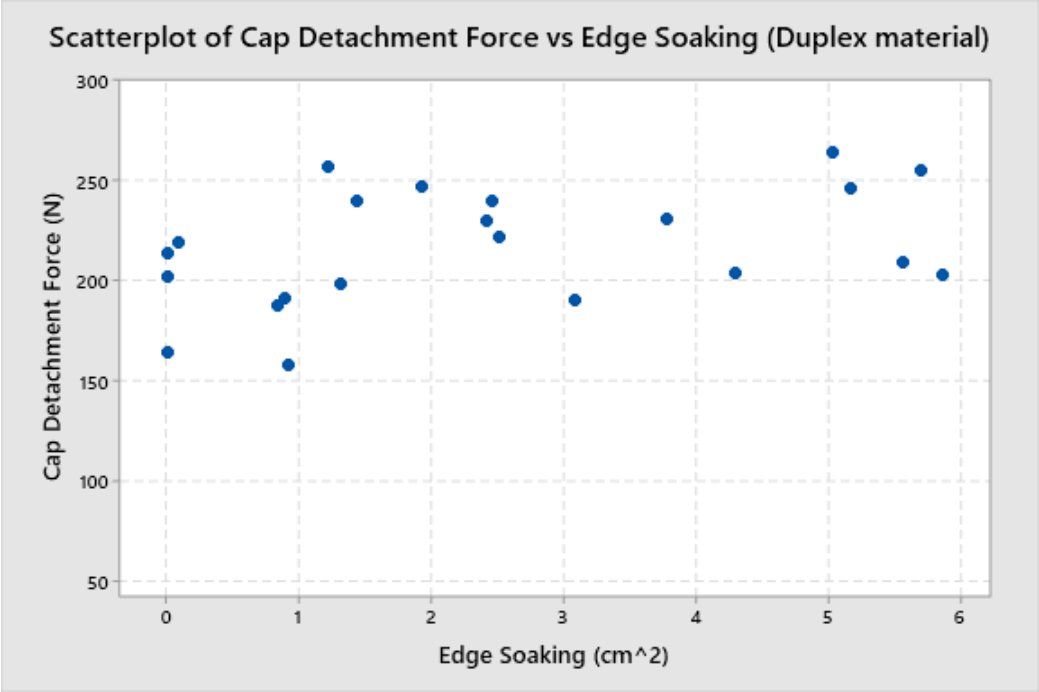

Figure 77 Cap Detachment Force vs Edge Soaking (area) for duplex material that has been dried in a 60 ºC oven over night without acclimatizing in a climate-controlled environment afterwards.

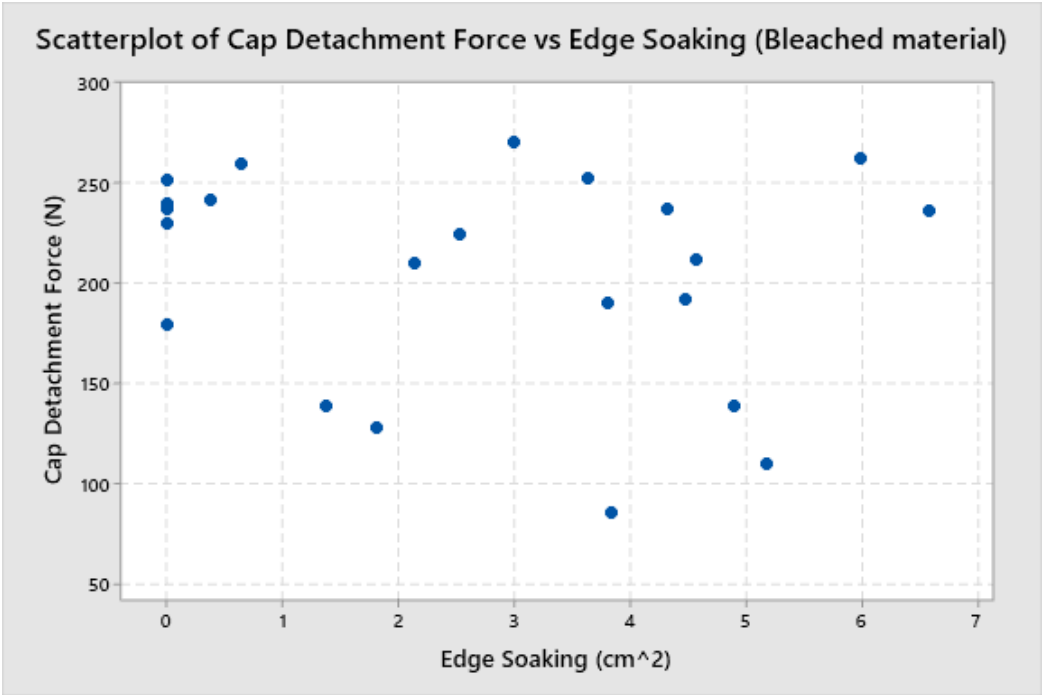

Figure 78 Cap Detachment Force vs Edge Soaking (area) for bleached material that has been dried in a 60 ºC oven over night without acclimatizing in a climate-controlled environment afterwards.