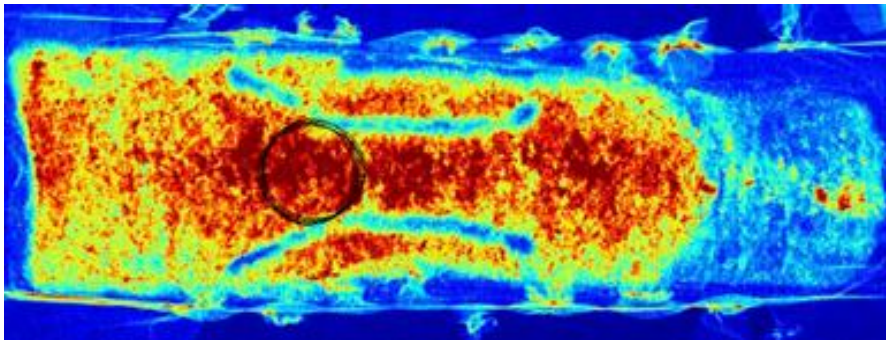


# CHALMERS



## Measuring Retention in a Baby Diaper

Method development for baby diaper testing: From testing principal to an optimized and evaluated method

**Bachelor of Science Thesis in Chemical Engineering**

**HENRIK HAMMARSTRAND**

*Project performed at SCA Hygiene Products, Gothenburg*

Department of chemical and biological engineering  
CHALMERS UNIVERSITY OF TECHNOLOGY  
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## *Abstract*

Laboratory testing is crucial for development and quality testing of baby diapers. The tests consist of everything from feeling the surface diapers dosed with synthetic urine, to measuring core evenness with x-ray images. One of the most important properties to measure in a baby diaper is how wet it is, and how well it can keep the user dry. If the baby's skin is wet it can cause irritation and damage the skin. Since babies wear diapers almost all the time, it is very important that the diapers manage their purpose of protecting the babies from exposure to liquid.

In this project, a new method has been developed. The project has started with modifying testing equipment and evaluating its function. The method properties have been chosen to be able to detect differences, as well to minimize the uncertainties. This was done by different test series with different aims. The results from these tests have led to a suggestion on how to perform the testing and how to evaluate its results. The test is performed by adding liquid to a product in a fixture then squeezing the product in the wetting area. The obtained result gives information on how well the product resists pressure as well as how well the liquid is bound within the product.

## *Sammanfattning*

Laboratorieförsök är en mycket viktig del i utveckling och kvalitetstestning av barnblöjor. Testerna omfattar allt från att känna på ytan av blöjor som blivit doserade med syntetiskt urin, till att mäta jämnhet utifrån röntgenbilder. En av de viktigaste egenskaper att mäta i en barnblöja är hur våt den är, och hur väl den kan hålla användaren torr. Om barnets hud är våt kan detta orsaka hudirritation och skador på huden. Eftersom bebisar använder blöjor största delen av dygnet är det mycket viktigt att blöjorna kan uppnå sitt syfte att skydda bebisar från exponering av vätska.

I detta projekt har en ny testmetod blivit utvecklad. Projektet har startat med modifikation och utvärdering av testutrustning. Testmetodegenskaperna har blivit valda för att kunna upptäcka skillnader, samt minimera osäkerheter. Detta gjordes genom olika testserier med olika syften, och resultaten har lett till ett förslag på en metod samt hur resultatet skall tolkas. Testet utförs genom att dosera en produkt med vätska i en fixtur sedan klämma produkten i vätningsområdet. Det erhållna resultaten ger information om både hur bra produkten står emot tryck, och hur väl vätskan är bunden i produkten.

## *Acknowledgments*

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Lots of gratitude to SCA for the founding, and thanks all the employees at Baby Laboratory.

# 1. Introduction

## 1.1 aim

The aim of this study is to develop a stable and reliable measurement system i.e. test equipment and test method, for measurement of rewet properties in baby diapers. A new analysis principal will be tested, namely gradient pressure. The uncertainty of the measurement system should be low enough to allow proper statistical evaluation of data. This study will focus on the process of developing an operational and evaluated analysis method for laboratory tests.

## 1.2 purpose

Baby laboratory has need of a new and more reliable method for measuring retention than the methods available today. Several different methods have been used to measure retention, but none of these methods have the ability to compare retention in thin diapers that correlate to real use. Product development of baby diapers has led to thinner diapers, and either the measurement systems have to be adopted to handle thin products, or new methods have to be developed.

## 1.3 Demarcation

This project will strive towards the mentioned aim, but the timeframe of 10 weeks will set the boundaries for the project.

One factorial design matrix will be performed and one measurement system evaluation. The project will not include any new equipment, but the needed system development and equipment optimization will be performed with the aid of the Technical laboratory support group (TLS). Three predefined products will be used as test samples, and no new products will be tested.

# 2. Background

## 2.1 SCA

SCA is northern Europe's largest forest company<sup>[1]</sup>. Its business consists of three branches, personal care products, forest products and tissue products, and the company conducts sales in roughly 100 countries<sup>[1]</sup>. The gross revenue year 2011 surpassed 80 billion SEK, which makes it one of the largest companies in Sweden<sup>[1][2]</sup>. The company started in 1927 as a merger of several smaller forest and milling companies<sup>[1]</sup>, and has vigorously grown ever since. The branch of interest for this study is the personal care branch, and the rest of this article will primarily treat that part of the company.

Personal care accounted for 30% of SCA's revenue the year 2012<sup>[3]</sup> and has many world renown brands such as Tena, Libro and Libresse. The Personal care branch of SCA is divided into baby hygiene products, incontinence products, and feminine hygiene products where each division has large amounts of sales worldwide<sup>[3][4]</sup>. The Personal care part of the company started out in 1975 when SCA bought Mölnlycke health care, thereby gaining access to all of Mölnlyckes brands, e.g. Libero and Libresse<sup>[1]</sup>. The single largest category of health care products is incontinence care, which accounts for roughly 50% of sales. Second largest in terms of sales is baby care products, which accounts for

close to 30%. This study is performed on the account of baby laboratory, but the results may influence the other research and development departments as well.

## *2.2 The modern disposable diaper*

Disposable diapers have been available since the 1940s<sup>[4]</sup> and account the major part of sales in today's baby hygiene market. Continuous development has led to the very efficient diapers that are available today. The production companies are constantly working on achieving sustainable products capable of meeting the market's demands.

Three parameters are often discussed when investigating the performance of a diaper: Inlet time, surface dryness and retention capacity. Inlet time is the time it takes for liquid to be absorbed into the product. Failure to rapidly absorb liquid could disrupt the comfort and in worst case lead to leakage. Retention is a measurement of how well the core can retain the absorbed liquid. Failure to keep the absorbed liquid in the core can lead to wet skin for the user. Surface dryness is a measurement of how wet the material in contact with the user's skin is and how much liquid can be extracted from that material. If the surface material is wet, this will affect the user regardless of how well retention and inlet time work. All these measurement parameters intend to give information on how well a product can keep babies dry. Failure in any of these three measurements is enough to require improvement, and so all three have to be thoroughly investigated.

It is important to keep in mind that these three parameters are in direct conflict with each other. Inlet time is shortened by using a more hydrophilic surface material, but that would make it harder to drain the surface material, and to keep liquid from coming back up i.e. worse surface dryness and retention. The reality for baby care products is always a compromise, forcing the products to be optimized with all kinds of properties.

## *2.3 The skin barrier*

Skin is one of the most important barriers for a human being<sup>[5]</sup>, and serves to protect us from many hazards. Human skin mainly consists of three different types of polar lipids namely ceramides, cholesterol and free fatty acids<sup>[5]</sup>. Since humans have very narrow acceptance limits for electrolyte concentration in body fluids, the skin has to precisely control which substances are allowed to pass<sup>[5]</sup>. The skin is rather impermeable to water, but if water of high ion concentration is in contact with the skin, there will be an osmotic pressure forcing water out of the skin<sup>[5]</sup>. A liquid with the ion concentration similar to urine causes a relatively high pressure, and exposure to such a liquid can disrupt the balance and cause skin irritation<sup>[5][6]</sup>. It has been documented that exposure to urine has significant effect on skin irritation<sup>[6]</sup>, thus it is very important to protect the body from long periods of exposure.



### 3. Theory

#### 3.1 The products

The baby diapers in this project consist of four layers as shown in figure 1. Only a brief explanation of all the parts will be given in this report, for more information see reference 4.

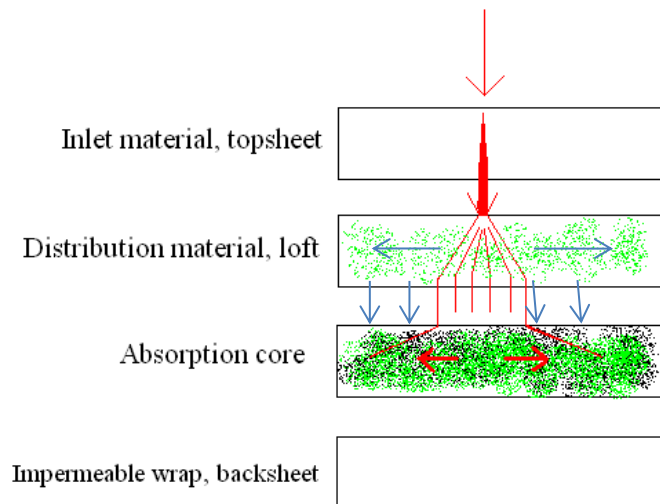


Figure 1: The four layers of a baby diaper and the liquid spread direction.

The first part of the diaper is the layer in contact with the skin. This is called a top sheet and is typically made of hydrophilic nonwoven materials. The reason why the top sheet is hydrophilic is to enable fast absorption of all liquid that is in contact with the wearer's skin, and to keep the skin dry<sup>[4][5][6]</sup>. The second part is called a distribution layer. This layer is what drains the top sheet of moisture<sup>[7]</sup>. The distribution layer also disperses all the liquid from the top sheet as much as possible, to increase the contact area of the absorption core, and keep the core from getting locally saturated<sup>[4]</sup>. The distribution layer in the three tested product has been made of two layers of hydrophilic nonwoven and fiber between them. When the product is subjected to external pressure between the top sheet and the absorption core, the distribution material should work as a protection. A distribution layer with good mechanical properties prevents liquid from easily traveling through. This protects the user from retention<sup>[7]</sup>. The third layer is the absorption core, made of SAP (super absorbent polymer) and pulp<sup>[7]</sup>.

Pulp has excellent absorption properties with strong capillary forces able to absorb and transport liquid, even counter gravitationally<sup>[8][9][10]</sup>. Pulp has high absorption capacity per weight and fast absorption rate, but the drawback is that it cannot sustain liquid under pressure, see section [3.2] for absorption mechanics. To increase absorption capacity further and to improve retention properties, there is often SAP in the diapers as well.

SAP is less expensive than pulp<sup>[11][12][13]</sup> and can absorb tremendous amounts of pure water, up to 1000g liquid/g SAP<sup>[8][9]</sup>. It has the ability to sustain liquid under pressure<sup>[12]</sup>. The drawback with SAP is that it absorbs liquid much slower than pulp, for absorption mechanism see section [3.3]. SAP in combination with pulp is what most baby diaper cores consist of today<sup>[7]</sup>. The two materials in harmony can provide an absorption core with fast absorption, good distribution, extreme absorption capacity and the ability to withstand pressure.

### 3.1.1 Test product Y, size 4 (7-14 kg):

The Product Y is displayed in figure 2. It contains: A top sheet made of a hydrophilic nonwoven material, a distribution layer of similar thickness to product C (section 3.2.3). It has two cores, and a back sheet made of an impermeable, hydrophobic polymer material.

The cores consist of pulp and SAP. There are drainage channels in the core, spaces with no absorption material, for good fit and eased transportation of liquid. The product has two cores to focus the absorption to the front of the product. The first core covers the whole product, and the second part is smaller and focused to the most likely wetting area. At the bottom and the top of the core, are covering layers of only pulp and no SAP, to prevent SAP penetration to the product surface. The core is kept in place with a core wrap.

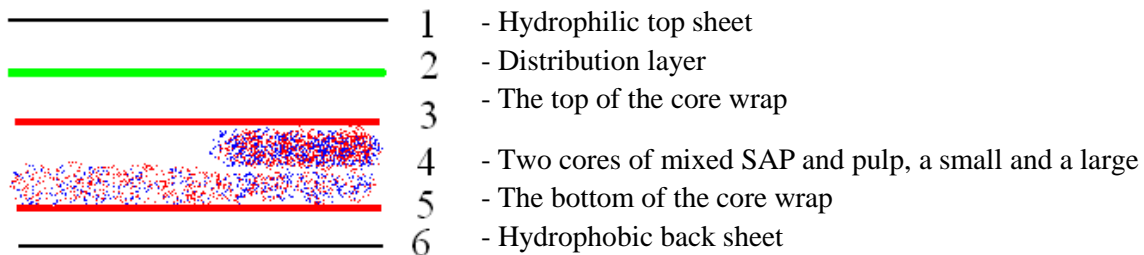


Figure 2: Length wise cross section principal sketch of Product Y.

### 3.1.2 Test product B, size 4(7-18 kg):

Product B (figure 3) contains: A top sheet of a hydrophilic nonwoven material. It has a thicker loft than product Y and C. The core is a laminate of two nonwoven materials with SAP between them. Product B has a core wrap glued to the loft as well as the back sheet, surrounding the core. The core is made of only SAP and no pulp. The top and the bottom of the core are glued in a pattern of squares that trap the SAP. When enough liquid has been absorbed, the squares burst due to the swelling of the SAP

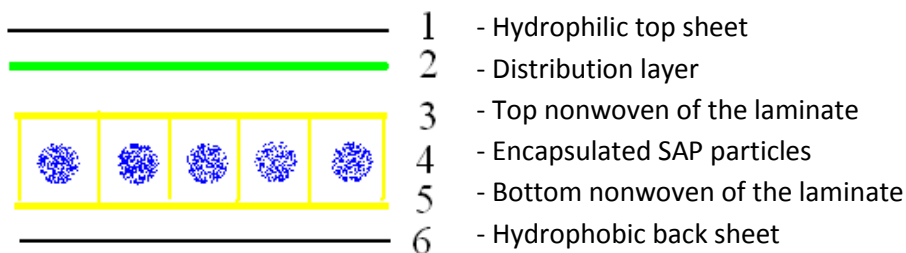


Figure 3: Length wise cross section principal sketch of Product B.

### 3.1.3 Test product C, size 4(7-18 kg)

Product C (figure 4) contains: A top sheet of hydrophilic nonwoven material, and a distribution layer. It has two cores of mixed SAP and pulp one small and one large, covered with a core wrap. The large core covers the whole product, and the smaller core focuses the absorption capacity to the most

likely wetting area. Product C differs quite substantially from product Y, where product C seems thinner and not as broad. The cores are glued to the core wrap, and there are no drainage channels in the core.

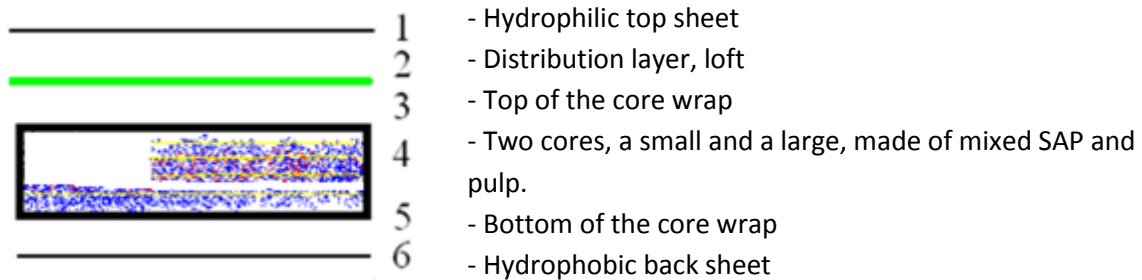


Figure 4: Lengthwise cross section principal sketch of Product C.

### 3.2 Absorption material pulp

Pulp is made up of cellulose fibers. The OH groups of the fibers are capable of forming strong hydrogen bonds with water. Regular wood consist of hemicellulose and lignin as well as the fibers, giving it a dense impermeable structure which keeps most fluids from getting absorbed<sup>[10]</sup>. When the cellulose fibers are structured as they are in pulp however, there is room between the fibers<sup>[10][16]</sup>. These small spaces between fibers work as capillaries, with hydrophilic walls. Water is transported into these small spaces, and can travel between them. The capillary forces allow the water to flow through the pulp, and the permeation can be modeled in many different ways<sup>[15]</sup>.

One way is the Washburn expression of the Laplace equation<sup>[10]</sup>, shown in equation 1. A brief explanation of the phenomenon is given in figure 5.

$$\frac{dl}{dt} = r * \gamma * \cos(\theta) / 4 * \eta * l$$

Equation 1: The Washburn expression

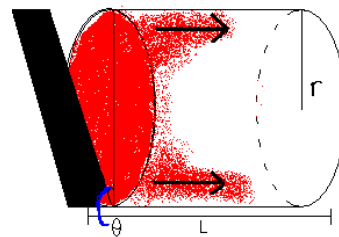


Figure 5: Illustration of liquid flow in one capillary, due to the intermolecular attraction between the liquid and the walls of the capillary

Where:  $\eta$  = viscosity  
 $\gamma$  = surface tension  
 $\theta$  = Contact angle

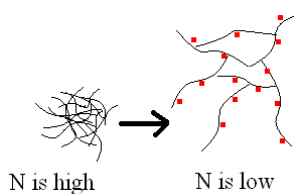
Equation 1 explains how the position of the solvent changes over time in a capillary i.e. the absorption speed. This can be used to model the absorption of liquid in pulp, where millions of small

capillaries are present and the sum of all capillary flows can be seen as the actual flow of liquid through the material.

When applying a force to a pulp system filled with solution, not only does the applied pressure contradict the absorbing force, it reduces the holes where water molecules are stored. Reducing this volume changes the whole pulp system, and lowers the capillary effect thus almost removing the absorption capacity completely<sup>[10]</sup>.

### 3.3 Absorption material SAP

The absorption principal of SAP can be explained by looking at the cross linked polymer network as a unit cell, with an elastic semi permeable membrane. Water can travel in and out of the cell, with osmosis as primary driving force<sup>[12]</sup>. A contrary force arises when the membrane of the cell is strained, due to the inner volume. The driving force of absorption i.e. the difference in ion concentration is reduced the more liquid that the SAP absorbs. The absorption equilibrium will depend on the “membrane pressure”, the liquid properties, and the ion concentration within the unit cell<sup>[12]</sup>.



There is no actual membrane, the membrane pressure is explained by the change in degrees of freedom of the polymers when the solvent is absorbed<sup>[8][9]</sup>. Lowering the degrees of freedom lowers the entropy, which demands the addition of energy to occur. See figure 6

Figure 6: The degrees of freedom in an absorbent polymer.

The liquid properties, such as viscosity and surface tension will remain rather unchanged during absorption and swelling, but the ion concentrations will change due to the absorbed amount of liquid<sup>[17]</sup>.

There are several models for predicting the rate of swelling for a polymer, i.e. in this case the absorption rate. The kinetics can be modeled by the expression in equation 2, based on Fick’s second law of diffusion<sup>[17][12]</sup>.

$$\frac{dQ}{dt} = \frac{Q_{max} - Q}{r^2 / \pi^2 * D}$$

Equation 2: Swelling based on Fick’s second law of diffusion

Where:

Q<sub>max</sub>= The maximum swelling(g/g) the polymer can handle

Q= The level of swelling at a given time.

r= The radius of the unswollen particle

D=The diffusion coefficient, depending of all phenomena affecting diffusion e.g. temperature and ionic strength

This is the reason why SAP can sustain liquid under pressure while pulp cannot<sup>[17]</sup>. If pressure is added to a saturated polymer the degrees of freedom are reduced, but the osmotic pressure remains. If the pressure is added to a saturated pulp instead, driving force for absorption is reduced as well as the degrees of freedom, thus reducing the capacity much more in pulp than in SAP.

The possibility to model the absorption behavior of the material matrix of concern has been developed, and today's models are very sophisticated. However no complete model has yet been developed, that describes the sum of all absorption rates and the net flow of liquid in a whole diaper system. Combining models and theories with laboratory testing make for even deeper understanding and more knowledge about the systems than either one does individually.

### *3.4 Vacuum pressure*

For this test method, the effect from the vacuum on the scale has to be considered. The amount of air inside the cup can be calculated through the ideal gas law, equation 3 where the gas (79%N<sub>2</sub> and 21%O<sub>2</sub>) can be considered far enough from its boiling point at the given pressure to have insignificant influence from the kinetic gas theory. The vacuum can be considered to decrease the weight by approximately 0,06g, thus the vacuum was sustained through the whole test.

$$P * V = n * R * T$$

Equation 3: The ideal gas law

Where:

P=pressure

V=volume

n=amount of particles

R=the gas constant

T=temperature

### *3.5 Design of experiments*

Design of experiment (DOE) is a statistical tool for investigating the effect of several parameters to a process<sup>[18]</sup>. If each parameter was investigated separately, the matrix effect of more than one factor could never be determined. That is why optimizing a process can be done in a specified manner, namely DOE<sup>[18]</sup>. Not only is the effect from each parameter determined in a DOE, but also the effect from parameters in combination.

In this projects center points were used. Center points enable evaluating the linear interpretation of the results, i.e how well the linear models work. Since multiple replicative center points were used, they can be used to quantify random effects.

To establish which effects are true and which are random, all the effects from terms of more than 2 parameters were deemed random. If any other term inclined to be extremely small, it was excluded from the model and seen as a random effect. Then the random term was quantified by looking at all the random effects and the variation in the center points. A minimum level for true effects at 95%

confidence level is used in this project, and the effects have to surpass this level to be considered true. DOE is a commonly used statistical tool at SCA Gothenburg<sup>[9][19]</sup>.

## 4. Methodology

### 4.1 Test setup

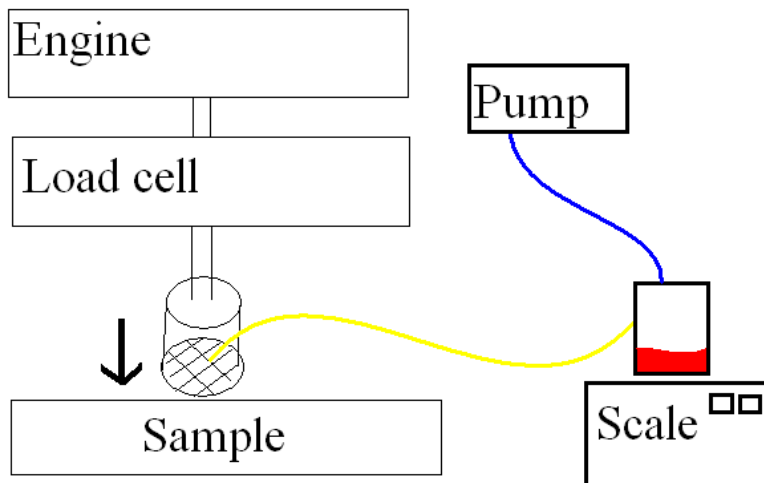


Figure 7: Flow chart of the testing procedure

The testing equipment consists of the following parts, as can be seen in figure 7. One engine controlled by software, that moves a load cell. The load cell registers the pressure the product is exposed to. Connected to the load cell is a pressure cup, constructed by TLS. Connected to this pressure cup, is a small plastic tube that leads to a vacuum cup on a scale. The vacuum cup is connected to a vacuum pressure. Any liquid coming up from the sample will be sucked through the tube and into the cup on the scale.

The load cell is calibrated using a 1 kg weight, henceforth the pressure that the glass fiber cup exposes the diaper to will be quantified in kg instead of the SI unit Pa.

### 4.2 Test liquid

All the tests were carried out using readily mixed and controlled synthetic urine. Synthetic urine (SUB) is a liquid with similar composition, surface tension, pH, ionic strength, density, conductivity to urine. Since these properties greatly affect absorption, see section [3.2-3.3], synthetic urine is preferable to other test liquids such as water or NaCl solution.

- For the early stage tests, batch nr [130313AN12] and [210313AN07] were used.
- For the DOE, batch nr [210313AN07] was used.
- For the verification test, batch nr [240413AN08] was used.

### 4.3 Sample preparation, sampling

For all products used in the DOE and the verification test, the following procedure was followed: 200 products from the same manufacturing batch of each type were weighed one by one. The 120 products closest to the average weight were chosen for the testing, the rest were discarded. The products were mixed and chosen at random for the DOE test series as well as the verification test series. The dry weights were noted for each sample, and used as covariates for statistical tests in the DOE and the verification test.

All the chosen products were conditioned for 24 hours in a constant climate room of 23°C and 50% air humidity.

The products for all the other tests were similar to product Y, though not from the same batch. They were not weighed or conditioned. They were allowed to get decompressed inside minigrip bags for at least one hour before testing.

#### *4.4 Dosing*

The samples were put up in a fairly user like position, using metal fixtures. Velcro (burdock) patches in combination with clips held the products in place.

The addition of liquid was executed automatically. The dosing was performed using a dosing equipment with the liquid flow 10ml/s for all samples. To add different volumes, the dosing time was changed. The dosage equipment was calibrated before each run, with acceptance criteria of  $\pm 2\%$ . The time after the last dose was measured with a quartz controlled timer.

#### *4.5 Test procedure*

After pre-determined time, a sample with complete dosing was put under the load cell according to figure 7. The engine lowers the glass fiber cup with high speed until the pressure of the load cell reaches a starting value. The program continuously reads 2 values of load per second, as well as 2 values of weight per second. The computer then regulates the speed of the engine to achieve a linear pressure increase. When the maximum pressure is reached, the pressure is kept constant during a period of time. After the time for constant pressure, the engine is reset to starting position and the data collection stops.

The program saves the weight data, as well as the pressure data. Each value of liquid weight corresponds to a specific time, and at that specific time, the pressure is also registered. The program plots the liquid weight, the actual pressure and the target pressure as responses of time for each sample. One of these plots can be seen in Appendix 1, figure 23. After one test, the sample is disposed and the next sample can be tested.

#### *4.6 Method development procedure*

Developing a test method from the very start required a well-structured procedure for setting up all the tests and interpreting their result. This project aimed at following method development procedure commonly used at SCA with regard to planning and execution. A brief description of the main test series and their purposes is given in this section, for an overview see figure 8.

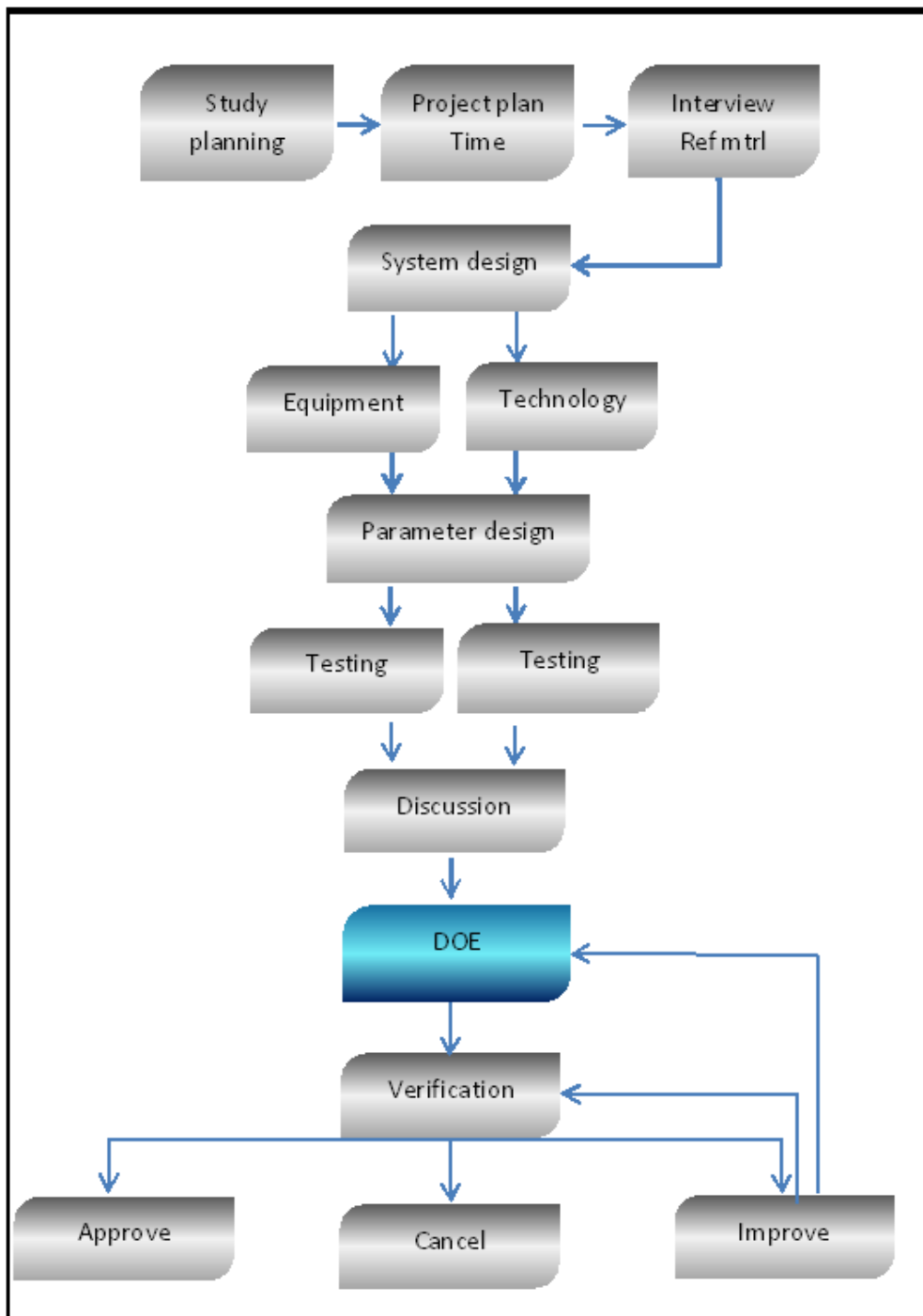


Figure 8: Flow chart of method development procedure.

To identify and optimize the important input parameters for the method, a DOE was performed. Since there was enough time for 20 test runs in this DOE, it was decided to use a full factorial design of 4 parameters and 4 center points. This demanded for all the parameters with effect on the result to be identified. The four most important had to be chosen and used as factors. All the other parameters had to be set to an appropriate constant level. For the parameters that were to be



factors in the DOE, suitable values for high and low level had to be found through testing and discussion.

The test setup had to be evaluated, and the results from this can be seen in section [5.1]. After concluding that the method could be run according to plan, and that the technical demands were met, the vacuum pressure, the time for constant pressure, and the max pressure were tested to see if they could be kept constant.

Then appropriate values for the high and the low levels of the factors in the DOE had to be found, more about this in section [5.2.3]

The optimization test was run according to the schedule shown in appendix 1 table 5.

When the DOE was finished and the results interpreted, the outcomes together with a visual evaluation were used to determine the final settings. A verification test was then performed, with the three different products described in section [3.1]. The verification test was performed to analyze how well the method performs regarding uncertainty and ability to detect differences between products.

#### *4.7 X-ray*

Quantification of true uncertainty in the test method would require completely homogeny samples. Since all manufacturing processes have some variation and because baby diapers are quite complex material systems, this is practically impossible. For the possibility to analyze uncertainty regardless of product unevenness, an attempt was made to connect core properties to test results.

To investigate the correlation between specific sample properties and results, products were analyzed with x-ray images, which show the density of the core area. Since SAP has higher density than pulp, a certain density would indicate a certain composition and distribution of SAP and pulp. The products tested as center points in the DOE were chosen to be as similar as possible with regard to distribution in the wetting area. For the verification test samples with much absorption material, as well as samples with little absorption material were chosen.

X-ray images never show the whole picture, only the density of the core horizontally. Thus no correlation to e.g. loft thickness or glue distribution can be investigated with this procedure. The x-ray images do not either show how the absorption material is distributed vertically, whether the material is close to the top sheet or the back sheet. However the images can be used to pick out similar or very different products in means of SAP distribution.

In this work, the products tested as center points in the DOE were chosen to be as similar as possible with regard to distribution in the wetting area. To collect more data on the effect that material distribution has on the results, all the products in the verification test were analyzed in x-ray as well. The results from these comparisons are discussed in section [5.4], examples of images can be seen in appendix 4, figure 26-31

#### 4.8 The verification test

After suggesting an optimal set of parameters for the testing procedure, a verification test was performed. The test series was performed on the three products described in section [3.1] and 10 sample of each product was tested.

To understand the effects of the inhomogeneity of the core in the measuring area, all the products were analyzed with x-ray. By visual evaluation of the images, 5 samples with high absorption material density, and 5 samples with the contrary were chosen for each product type.

For the method to accurately handle many different types of products is absolutely crucial. There are many types of products that will need to be tested, and this method should be able to analyze them all, thus a third product is tested in this verification test. The tests are run in the order shown in Table 1.

run order	product	absorption mrtl(+/-)
1	b	-
2	b	+
3	c	+
4	c	-
5	y	-
6	y	+

Table 1: Run order for the verification test, where (+) means more absorption material in the pressure area, and (-) means less.

#### 4.9 Analyzing the results

All statistical calculations were carried out using Minitab statistical software version 16. Simple calculations were performed using Microsoft excel, and some graphs were made using Matlab.

Each sample gave approximately 200 data points, describing how liquid flows from the product under pressure over a period of time. Having this amount of data poses the question of what is important to analyze, from a development or an end user point of view. The two primary responses obtained from the purposed method are:

- At which pressure does liquid start flowing out of the product? (firstP)
- How much liquid can be extracted from of the product? (totW)

These two responses provide important information on how products perform, both the aspect of pressure resistance, and how well free liquid is retained. There are other possible ways of analyzing the data, for example the flow rate at the start of the curve, or the decrease in flow rate over time but firstP and totW were chosen for this project.

One of the most important aspects of the method is to have stable results, as can be seen in the aim of the project. To achieve stability, the standard deviation (stdev.) was calculated for firstP as well as totW, and both were used as responses in the DOE. Using these responses enabled optimization of parameters to minimize the deviations in the method. The relative within group stdev. was used to quantify repeatability in the verification test.

Another way of measuring uncertainty is to quantify the total area between the weight curves and comparing them to the average of the weight curves. This way, not only the starting point or the end point of the weight curves was analyzed. For more information about this method, see appendix 3

#### *4.10 Information sources*

To reach deeper understanding about the development division, two scientists at SCA were formally interviewed, Ted Guidotti and Helena Corneliussen. Ted works as a senior scientist, and specializes in absorption. The full protocol of the interview can be seen as reference 9. The second interviewed employee is Helena Corneliussen, who is a lead product developer. Helena's role is to assist development projects with product expertise, and to take part in development projects. They both have a great deal of experience of corporate development work, and take essential parts in lots of different projects for baby hygiene product development. Helena's interview is referred to as reference 19.

The rest of the sources used were chosen by studying lots of scientific literature. All fields from medical journals to fluid metrics were studied, and a few of the most reliable articles were chosen to support the claims made in this article.

## 5. Results and discussion

### 5.1 System design

#### 5.5.1 Equipment improvements

The test setup was evaluated by using a calibration weight of 10g, tarring the scale and registering weight when the load cell was moving, see results in figure 9. This showed that optimization was needed. After improving the test setup, the test was performed again and there was only acceptable noise i.e detection limit below 0,05g was in the weight data.

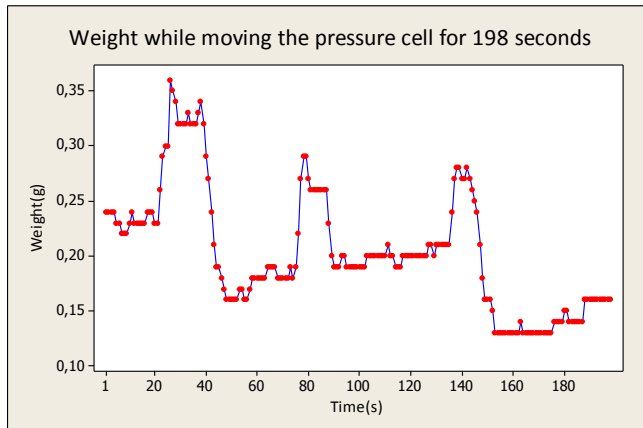


Figure 9: Weight values as a response of time for the first equipment set up

The results indicate that with further development, method for measuring retention could be fashioned using this measurement principal and the development will continue. The following equipment optimizations were made:

A manometer was installed to measure the vacuum pressure. The tubing was fixated to minimize its mechanical influence on the scale. A weight of 500g was added to the liquid containment cup to increase inertia and lessen the mechanical influence from the tubing.

### 5.2 Parameter design

#### 5.2.1 The time for constant pressure

Finding the optimal time for constant pressure was done by testing 5 products at different times for constant pressure. The result graphs can be seen in figure 10 and the results indicate that to reach the point where the flow rate has started to decrease, 50 sec of constant pressure time is sufficient.

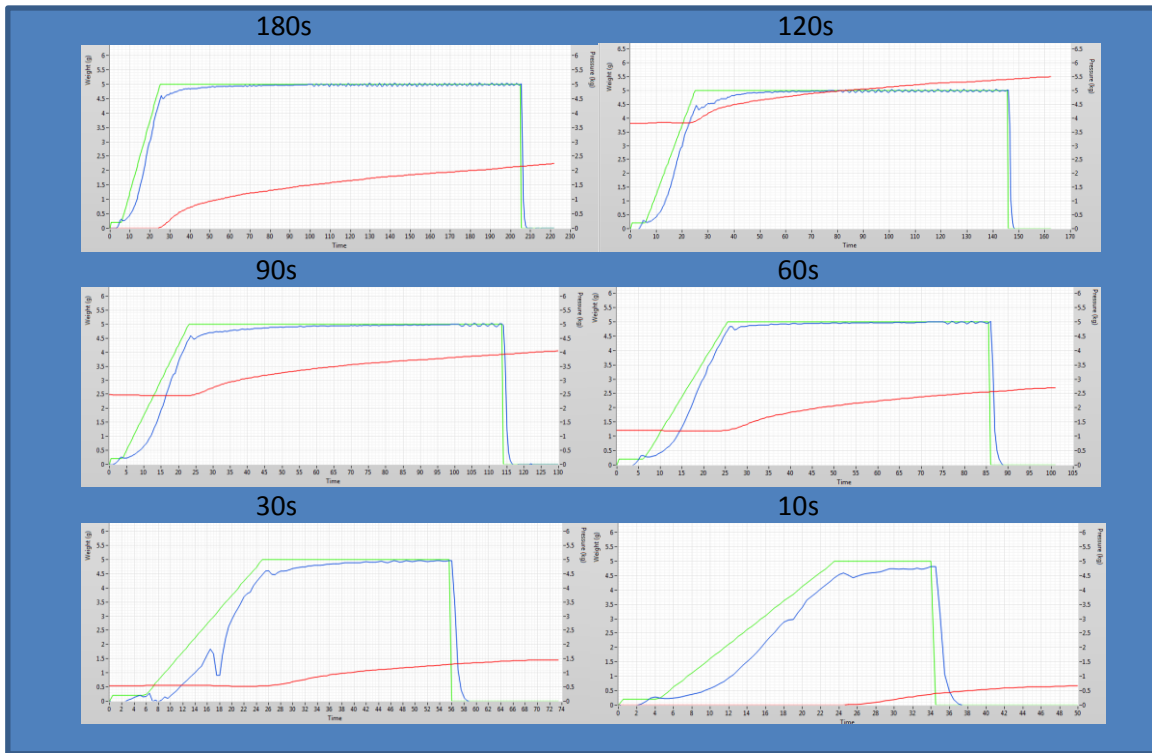


Figure 10: The result graphs for the different times of constant pressure

### 5.2.2 The vacuum pressure.

To find the appropriate vacuum pressure, a test series was performed where two levels were evaluated. In the results, no significant difference can be detected. There is, however an inclination that a lower vacuum pressure results in lower stdevW, as can be seen in table 2.

Property	Repeat 1	Repeat 2	Repeat 3	Repeat 4	Repeat 5	Repeat 6	Average	stdev
totW,lowp	0,59	0,58	0,69	0,75	0,63	0,71	0,6583333	<b>0,068823</b>
totP,lowp	4,772	4,655	4,531	4,486	4,27	4,566	4,5466667	0,169325
totW,highp	0,93	1,24	0,82	0,8	0,85	0,68	0,8866667	<b>0,191172</b>
totP,highp	4,616	4,615	4,691	4,365	4,589	4,506	4,5636667	0,114038

Table2: Results from testing the vacuum pressure. Lowp=-0,2 bar, highp=-0,38 bar

#### Hypothesis test (paired t-test)

To evaluate whether or not there is a statistically true difference at 95% confidence level, the results are compared in a paired t-test.  $H_0$  is the null hypothesis, and the test investigates if there is enough evidence to discard the null hypothesis. If  $H_0$  is discarded it means that  $H_1$ , the contrary is true.

Response is totW

$H_0$ : totW,lowp=totW,highp

$H_1: \text{totW,lowp} \neq \text{totW,highp}$

$P=0,074$ , there is not enough evidence to say there is a true difference between the high level of vacuum and the low level. It can be said that the p-value being as low as it is suggests that there may be a difference, only it can't be detected in this test.

---

This test resulted in the vacuum pressure not being evaluated in the DOE, and instead being set to -0,2 Bar.

### 5.2.3 Choosing the DOE parameters

To decide an appropriate dose, data from several different projects was studied, discussion was held and several company sources were consulted. The dose was then set to vary between 50 and 70 ml. The product is dosed thrice, which means a total addition of liquid between 150 and 210 ml.

The waiting time between doses was set to a constant value while the waiting time after the last dose varied. Due to the limitation of the equipment, the measurement can only be performed on one sample at a time. This forces the waiting time between doses to be balanced between time consumption and sufficient time for the measurement itself. The time evaluation led to the waiting time, between two doses, being set to 15 min and the waiting time after the last dose between 5 and 10 minutes.

To decide which max pressure and which slope of the pressure increase to use, careful consideration had to be taken. There was not enough time to investigate both pressure increase time and max pressure as separate parameters in the DOE. By evaluation of early tests as well as through discussion, the time for pressure increase was varied between 10s and 40s. The pressure was then chosen to be as low as possible, yet able to extract liquid in all parameter combinations. For this purpose, the max pressure was set to 5 kg. Two center points for each product were used, for more information about center points in a 2 level factorial DOE, see section [3.5]. Summarized, the parameters in the DOE with high, low and center levels are summarized below:

product	B	-	B&Y	-	Y
Waiting time	5	-	7,5	-	10
Dose	50	-	60	-	70
Pressure inc	10	-	25	-	40

### 5.2.4 The DOE results

All the terms included in the models are displayed in Appendix 2 table 4 with their p-values and coefficients. These were the guidelines for choosing optimal parameters. The stdev. values are analyzed using the least square regression estimation method.

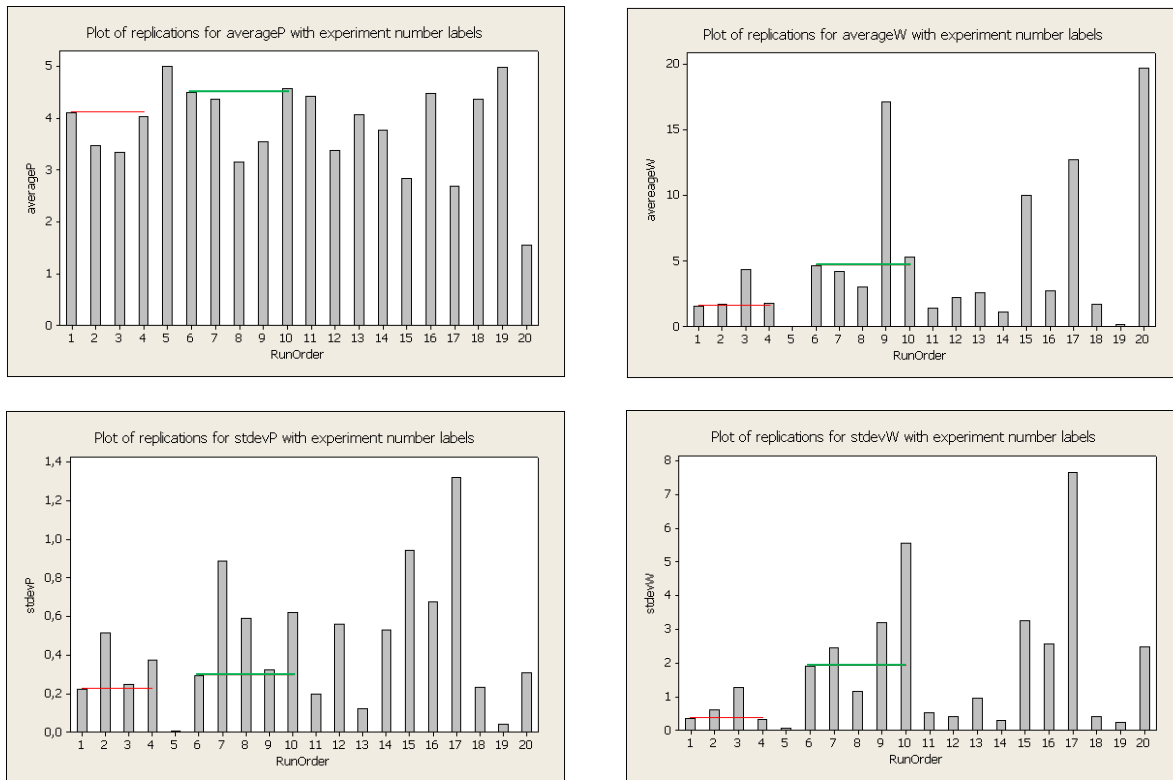


Figure 11: Illustration of the magnitude of the 20 test series, center points for product Y marked with red and for product B with green.

As seen in figure 11 regarding Pstart, the center points show very similar results for both products. For rewet (totW) on the other hand, the product B center points differ both in stdev. and totW. By looking at the x-ray images of the products, this can be explained to some extent see section [5.7]. For totW, the product B was excluded from the model and the results analyzed based on only the product Y part of the DOE.

When the results of the DOE are analyzed, it has to be established which effects are true and which are random. Pareto charts follow, in figure 12-16, which show all the effects from the factors in the Doe result models. There is a red line in the Pareto charts, resembling the level that the effects have to surpass to be considered true effects. All bars to the right of the red line are significant (true) effects.

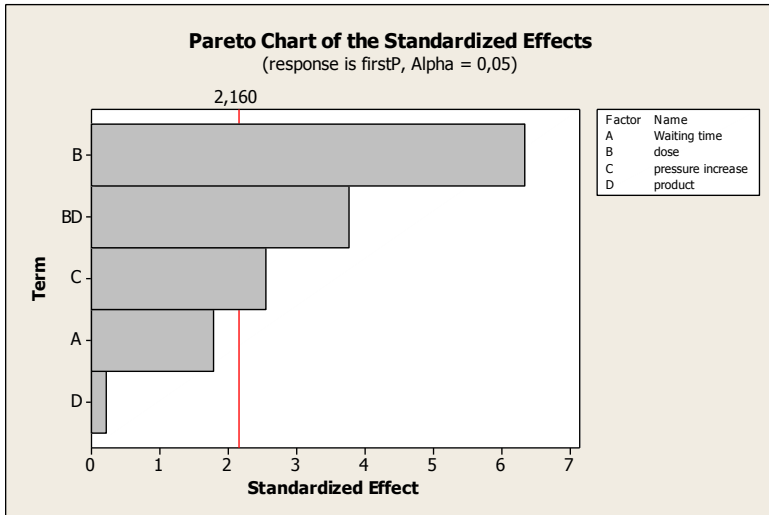


Figure 12: Pareto chart of effects for average firstP

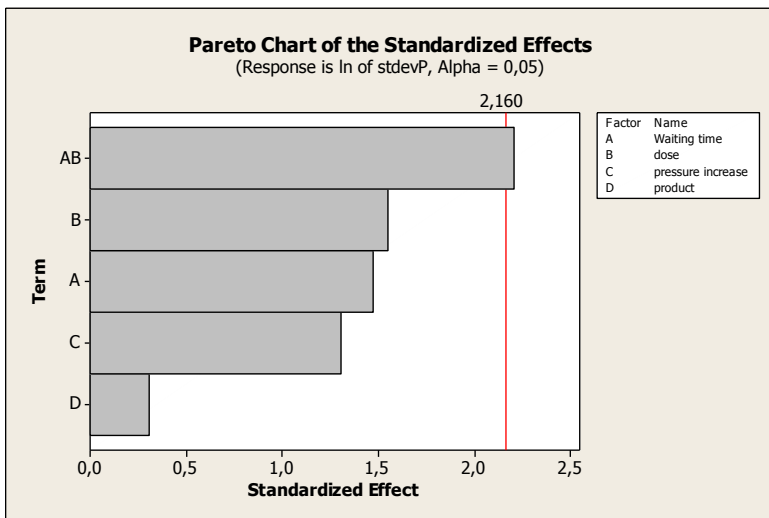


Figure 13: Pareto chart of effects for stdev. firstP



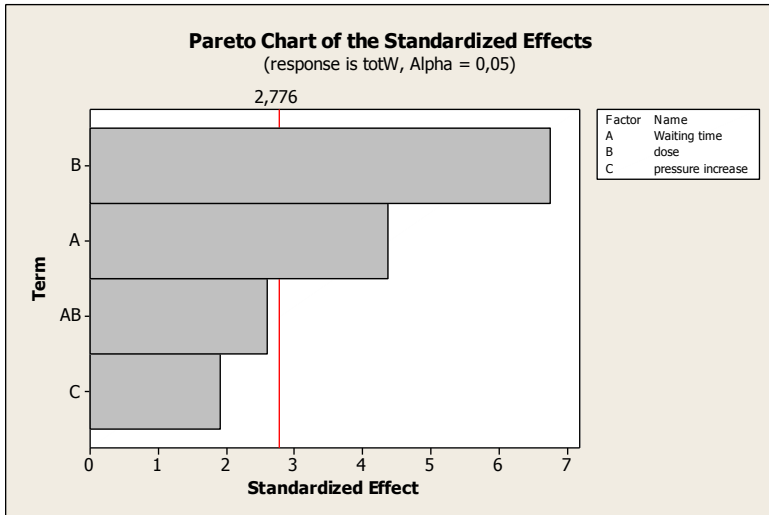


Figure 14: Pareto chart of effects for totW

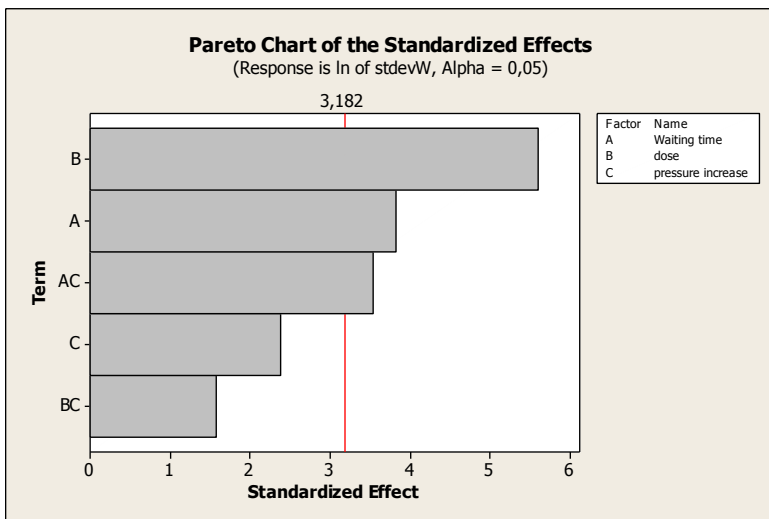


Figure 15: Pareto chart of effects for stdev. totW

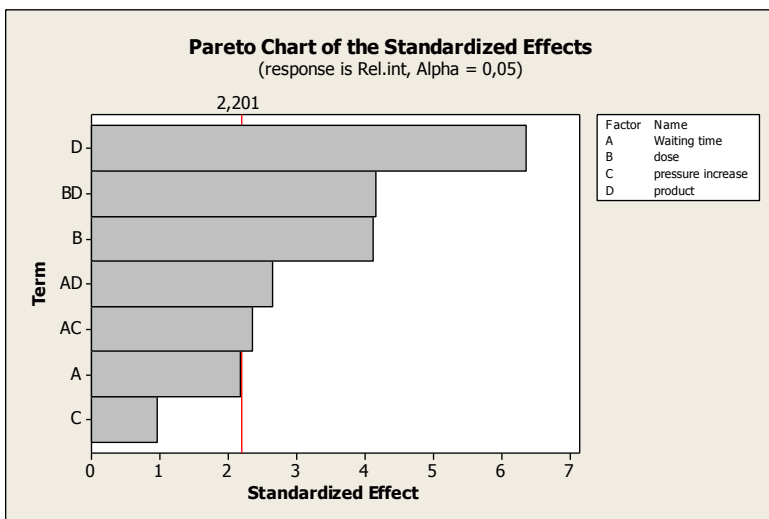


Figure 16: Pareto chart of effects for Rel.int

The effect of each parameter of every aspect on the different result is shown in table 5 (Appendix 1).

Parameter	Rel.int	First P	StdevP	StdevW
waiting time	+	-	+	-Du
Dose	+	+	+	-
pressure increase	+	+	+	-

Table 3: Summary of how the parameters should be optimized according to the DOE.

The results in table 3 combined with discussion and evaluation led to the parameter combination displayed in table 4. Some effects had to be weighed against each other but holistically, the visual judgments agreed with the statistical results.

Parameter	Optimum
Dose	70 ml x 3
Waiting time between doses	15 min
Waiting time after third dose	10 min
Pressure increase	30 s
Time for constant pressure	50 s
Max pressure	3 kg
Negative vacuum pressure	0,2 Bar

Table 4: The optimal combination of parameters for the method.

### 5.3 The verification test

The result from the verification test are summarized in appendix 2, table 7. The weight graphs for each products (+) and (-) are shown in figure 17-19 and the statistical tests are shown in appendix 2.

Since the variances of all different products are not equal and all results are not normally distributed, nonparametric tests are used to statistically evaluate the results when it was not possible to use parametric tests<sup>[20]</sup>.

A difference can be detected between the different products regarding firstP, using Kruskal-Wallis test<sup>[20]</sup>. By analyzing all products with Kruskal-Wallis tests, the products can be grouped<sup>[20]</sup>:

B	1
Y	1
C	2

Where higher group means better score.

A difference can be detected between the different products regarding totW, using Kruskal-Wallis test<sup>[20]</sup>. By analyzing all products with Kruskal-Wallis tests, the products can be grouped<sup>[20]</sup>:

B	1
Y	2
C	3

Where higher group means better score.

The results of the verification test indicate that product C has better resistance to pressure than Y and B. The results also show that the products receive the following score rank based on how much liquid they release: C>Y=B

To summarize the results:

- No significant difference can be detected between high amount absorption material(+) and low amount absorption material (-) for any of the samples regarding firstP
- No significant difference can be detected between (+) and (-) and any of the samples regarding totW
- There is however a trend that the (+) products perform better than the (-) products, which supports the claim that some of the result variations depend on the products.
- The most even of the products resulted in a within repeatable relative stdev.W of between 0,3 and 0,4. The highest relative stdev. was for the product B- run, which resulted in 0,69.

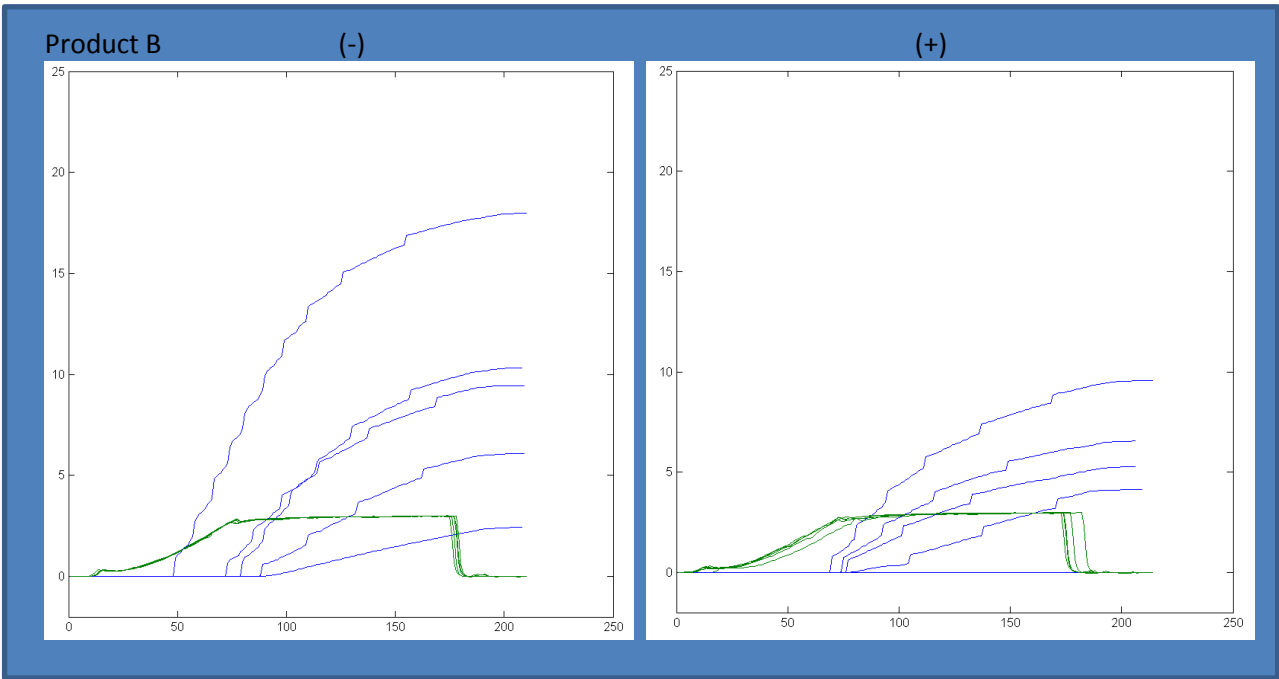


Figure 17 The results for all (+) samples and all (-) samples, product B

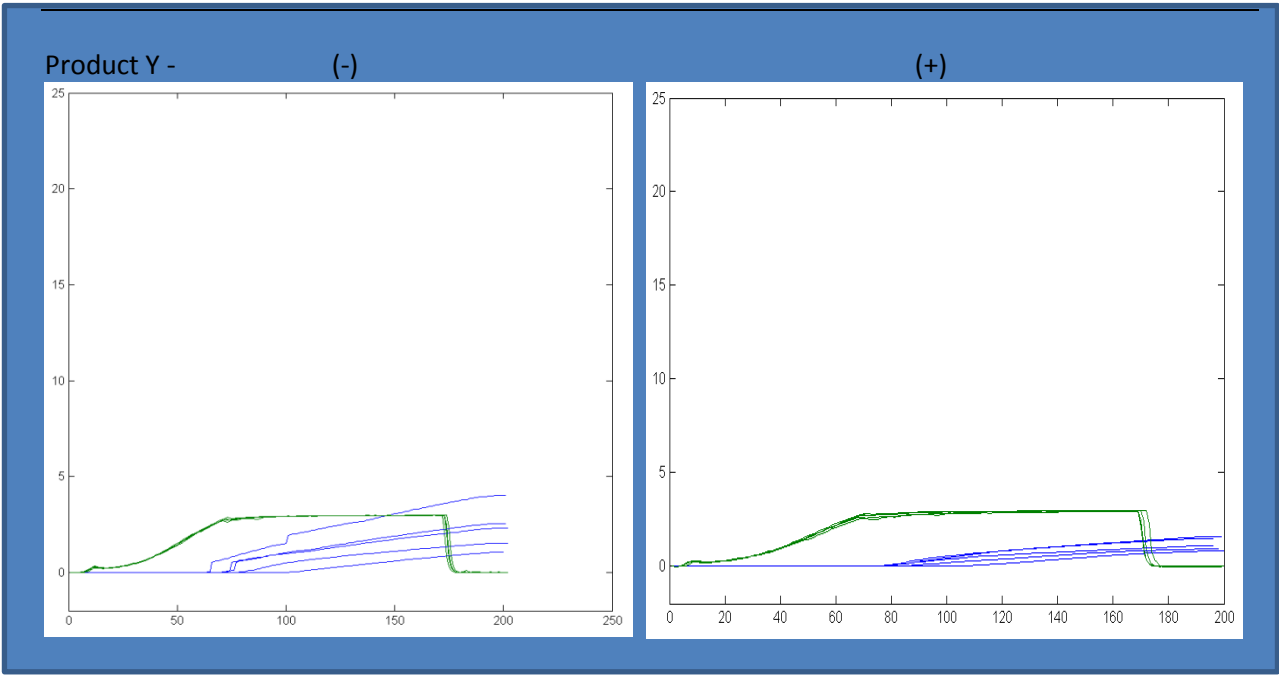


Figure 18: The results for all (+) samples and all (-) samples, product Y

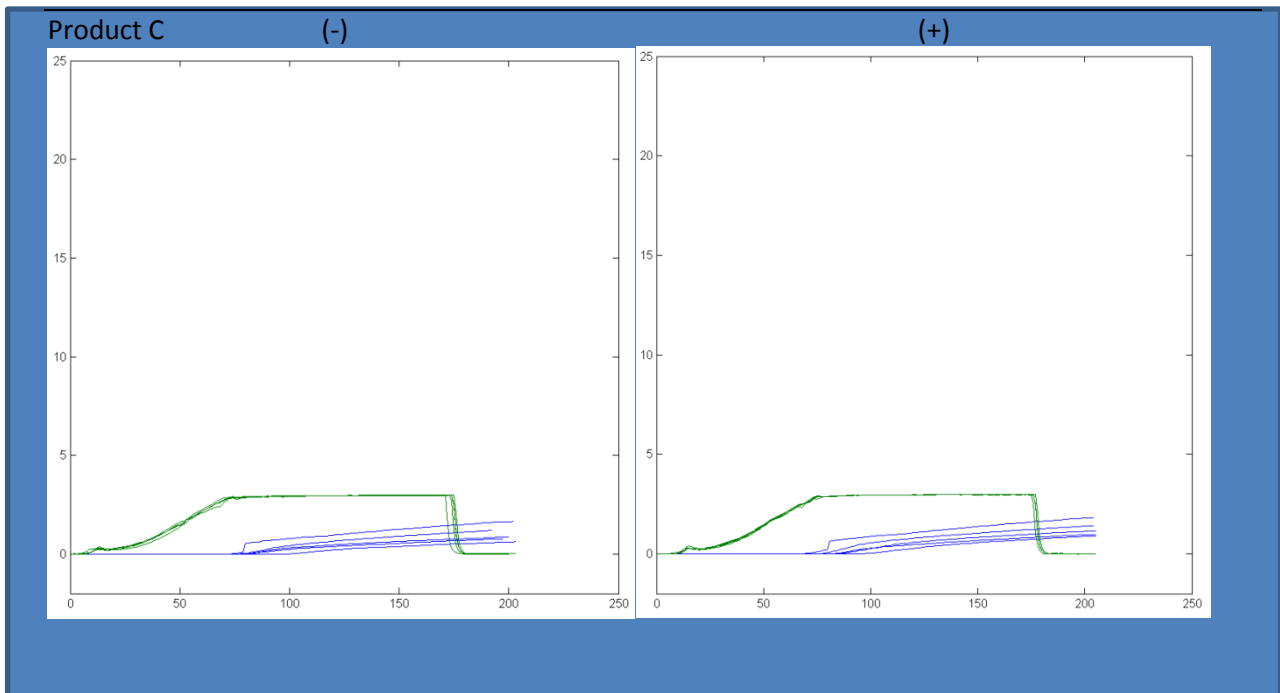


Figure 19: The results for all (+) samples and all (-) samples, product Y

#### 5.4 The product variation

When looking at an overview of the x-ray pictures, a trend can be observed. The results indicate that products with more absorption material in the pressure area, are harder to extract liquid from than the ones with less, see figures 17-19. This is most significant for the product B. An example of a good product B and a bad one are shown in figure 22. In this figure, the product to the left has an area of almost no absorption material right next to the pressure point, i.e. the free liquid can be stored there. The product to the right has an area of almost no absorption material within the pressure area, greatly easing the extraction of liquid.

For product Y the differences between the products are not as distinct as for product B. The Y+ products performed slightly better than the Y- products. The C products were very homogeny, thus the (+) and (-) samples were almost identical. Average values for comparison are displayed in figures 20, 21.

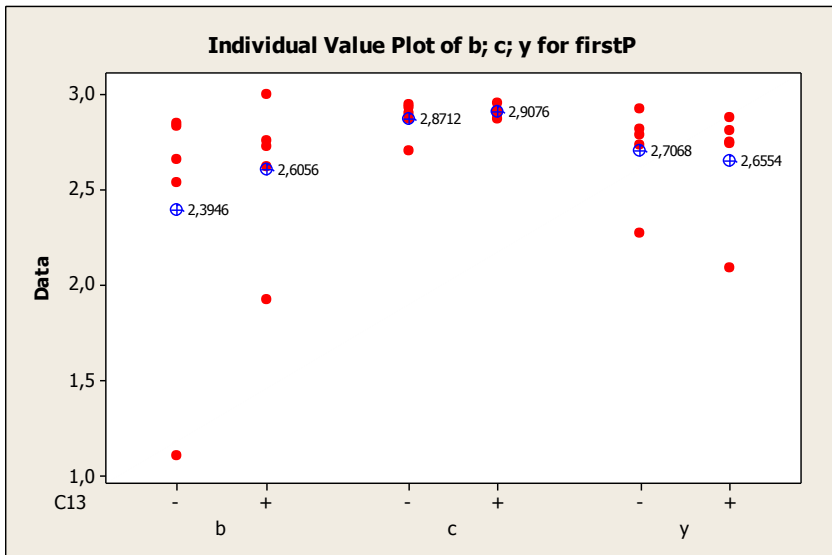


Figure 20: Individual value plot showing the difference between (+) and (-) regarding firstP.

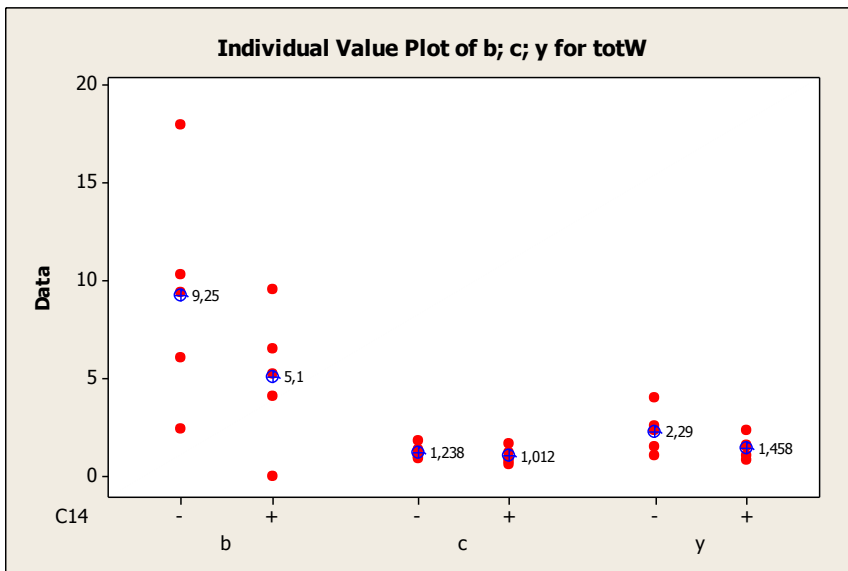


Figure 21: Individual value plot showing the difference between (+) and (-) regarding totW.

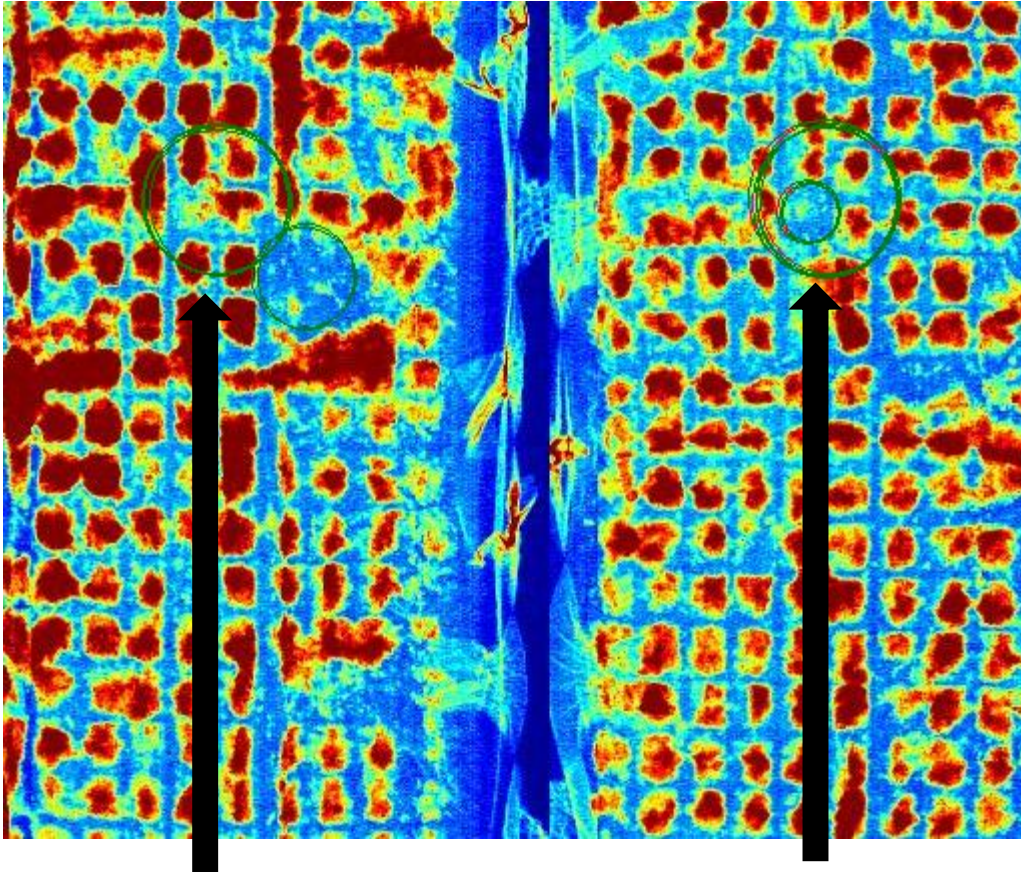


Figure 22: A (+) product B to the left, a (-) product B to the right

### *5.5 Sources of uncertainty in the test setup.*

One source of uncertainty that could explain the low repeatability is the size of the pressure cup. The retention measurement should represent the whole product, yet the pressure in this test is only applied in a small area. For a result that is more relevant from a user point of view, the pressure might have to be applied over a larger area.

Another important source of error is the delay from the extraction of liquid until it can be registered by the scale. It was observed that there is a slight delay before the liquid that is sucked out can be registered by the scale. No attempt to quantify this delay was made and no attempt to minimize it. For suggestion on how to proceed see section [7]. By quantifying the delay, the first pressure could be easily corrected through the software.

Error in marking the wetting point will always occur, since the wetting point measurement is performed manually. The base for measuring the middle of the product was in this project the placement of the distribution material. The distribution material is not always centered in the product, which could cause uncertainty. This could cause problems if the X-ray image is aimed to be used as explanatory factor for retention measurement.

## 6. Conclusion

The purpose with this project was to develop a more reliable and stable test method for measuring retention in baby diapers than the methods used today. A complete measuring system has been suggested and evaluated see table 7, but the method and equipment still needs further improvement before it can be put into use. For suggestions on further development, see section [7].

Testing retention in a way that correlates to real use was one of the targets of this project. The results acquired in the verification testing have come to the same conclusion as a consumer test i.e. product B<Y. This correlation could be worth evaluating by continuously comparing test method results to consumer test results.

The comparison between x-ray images and their test results indicate that there is a connection. It seems that the core distribution of absorption material can explain the results to some extent, as seen in section [5.3]. Concluded, it might be possible to explain some of the product behavior using x-ray images, yet not all of it. If possible the measurement system should be made to provide more even values, instead of adding x-ray analysis to the standard procedure.

Product B, C, and Y start releasing liquid rather similarly even though product B releases much more liquid than either C or Y. This indicates that the first pressure does not necessarily correlate to the total amount liquid that can be extracted. When changing the max pressure from 5 kg, used in the DOE, to 3 kg, used in the verification test, the results were not very affected. This indicated that as soon as the needed pressure to squeeze out liquid is met, the flow rate will depend mainly on how much free liquid exists in the pressure area.

The verification in this work was conducted with rather few tests; two test series per product type because it was probable that the measurement system would need improvement. The quantification of uncertainties would have to be performed according to measurement system analysis.

To draw any accurate conclusion regarding the first pressure, the delay before the liquid reaches the scale must be revised. For suggestion on how to perform this see section [7].

The data interpretation could be changed, because regardless what points of the weight curves are important, the proposed method is a good choice. Because of the evaluation using Rel.int in the DOE, the obtained curves are as relatively similar as possible when looking at the total weight curve. Changing how the data is analyzed does not necessarily mean changing the method. Other aspects such as the flow rate just after the product has started releasing liquid.



## 7. Recommendation

The methodology is recommended to be used for measurement of retention in baby diapers

Further development of this method should start with designing a new pressure cup. The new pressure cup should be larger and preferably ductile, because this would make the pressure more evenly distributed over the product, and not sensitive to core unevenness. The size of the cup must not exceed the area of any products that are to be tested.

The second most important technical optimization is the tubing, which might be possible to shorten to reduce the delay before the liquid is detected. Reducing the tube diameter would also decrease the delay, as implied by the Bernoulli equation see appendix 5 equation 6.

For better stability, the scale should be placed on a more stable surface.

If another way of analyzing data is desired, the recommendation is to study the flow rate at the beginning of the weight curve.

After optimization, the results should be further compared to the results from consumer tests, to see if the connection can be expressed better.

After optimizing the equipment, the software has to be upgraded to achieve a more user friendly program.

The method has to be verified by performing a proper measurement system analysis.

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## Appendix 1: The DOE optimization test

The DOE run order can be seen in table 5.

StdOrder	RunOrder	CenterPt	Blocks	Waiting time	dose	pressure increase	product
18	1	0	1	7,5	60	25	y
13	2	1	1	5	50	40	y
11	3	1	1	5	70	10	y
20	4	0	1	7,5	60	25	y
2	5	1	1	10	50	10	b
19	6	0	1	7,5	60	25	b
1	7	1	1	5	50	10	b
15	8	1	1	5	70	40	y
3	9	1	1	5	70	10	b
17	10	0	1	7,5	60	25	b
10	11	1	1	10	50	10	y
16	12	1	1	10	70	40	y
12	13	1	1	10	70	10	y
14	14	1	1	10	50	40	y
8	15	1	1	10	70	40	b
5	16	1	1	5	50	40	b
4	17	1	1	10	70	10	b
9	18	1	1	5	50	10	y
6	19	1	1	10	50	40	b
7	20	1	1	5	70	40	b

Table 5: The run order and all the parameters for the DOE test

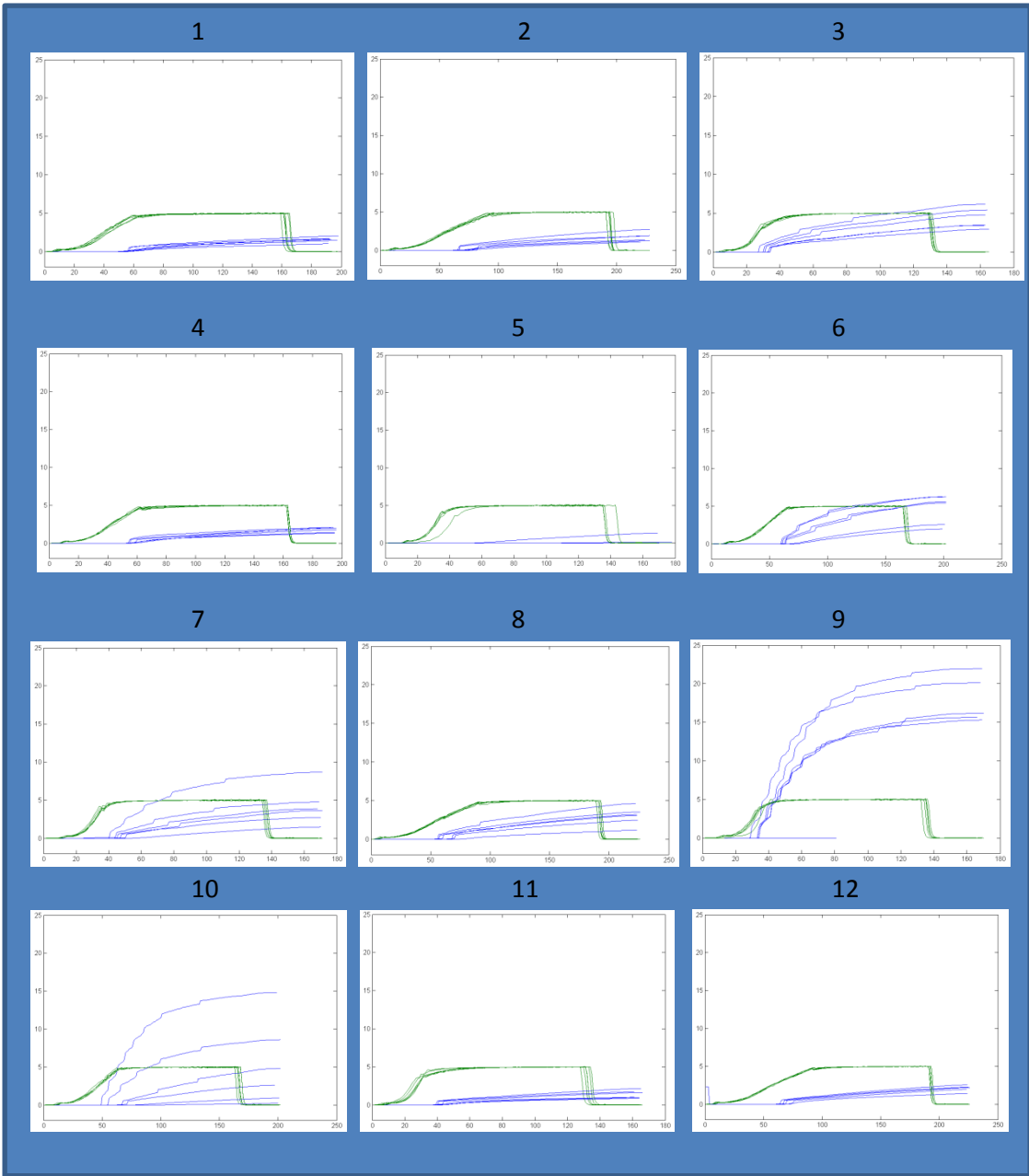


Figure 23: Collection of graphs showing weight of liquid(blue line) as response of time for run 1-12 in the DOE. The green lines represent the actual pressure

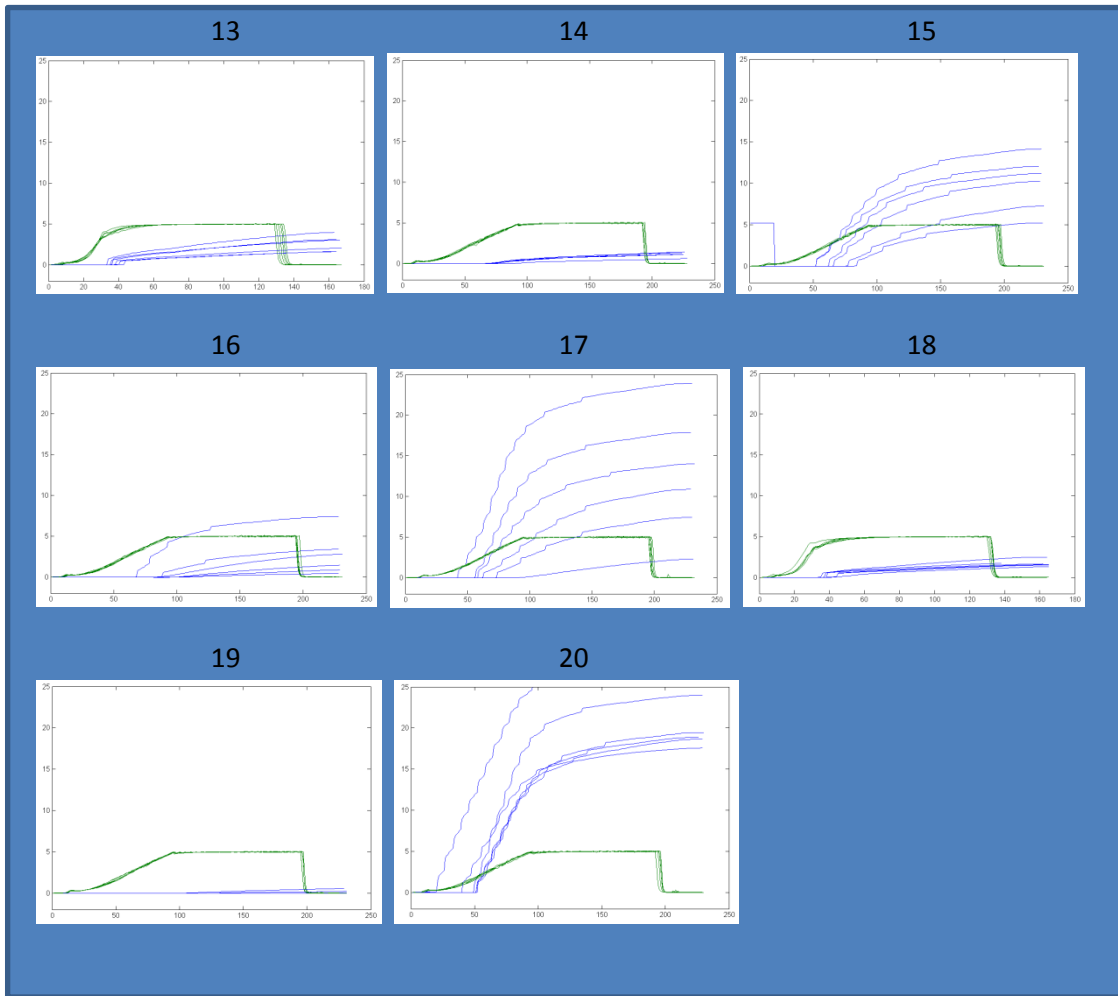


Figure 24: Collection of graphs showing weight of liquid(blue line) as response of time for run 13-20 in the DOE. The green lines represent the actual pressure

Rel.int		
Term	Coeff	P-value
Constant	3,75449	0
Waiting time	0,375117	0,051
dose	-0,063218	0,002
pressure increase	0,0623226	0,355
product	3,49599	0
Waiting time*pressure increase	-0,009628	0,038
Waiting time*product	0,16296	0,023
dose*product	-0,064075	0,002
First P		
Term	Coeff	P-value
Constant	7,45942	0
Waiting time	0,0723842	0,098
dose	-0,064310	0
pressure increase	-0,017339	0,024
product	2,31056	0,839
dose*product	-0,038196	0,002
StdevP		
Term	Coeff	P-value
Constant	8,00384	0,0001
Waiting time	-1,67035	0,163
dose	-0,143867	0,145
pressure increase	0,027275	0,214
product	-0,0793	0,761
Waiting time*dose	0,0250438	0,046
StdevW		
Term	Coeff	P-value
Constant	-3,64888	0,004
Waiting time	0,0512023	0,032
dose	0,0503342	0,011
Pressure increase	0,0721675	0,097
Waiting time*pressure increase	-0,005791	0,038
dose*pressure increase	-0,000641	0,214
Tot W		
Term	Coeff	P-value
Constant	-4,59993	0
Dose	0,143083	0,004
Waiting time	0,35025	0,028
Pressure increase	-0,016736	0,127
Waiting time*dose	-0,008758	0,169

Table 6: All parameters included in the models, on all the different results for the DOE optimization

## Appendix 2: The verification test

product	averageP	stdevP	averageW	stdevW	rel.stdevP	rel.stdevW	rel.int	int
B+	2,60	0,41	5,1	3,50	0,16	0,69	2,49	6,39
C+	2,91	0,03	1,012	0,41	0,01	0,41	1,92	0,78
Y+	2,66	0,32	1,458	0,58	0,12	0,39	1,36	0,64
B-	2,39	0,73	9,25	5,79	0,31	0,63	2,98	12,07
C-	2,87	0,10	1,238	0,37	0,03	0,30	1,65	0,76
Y-	2,71	0,25	2,29	1,14	0,09	0,50	1,93	1,97

Table 7: Descriptive statistics of the verification test results

Is there a difference between the (+) and the (-) regarding total amount extracted liquid?

---

Hypothesis test (Mann-Whitney test) for difference between (+) and (-), **product B**

Response is totW

$H_0: B+ = B-$

$H_1: B+ \neq B-$

	N	Median
-	5	9,43
+	5	5,26

Test of  $ETA1 = ETA2$  vs  $ETA1 \neq ETA2$  is significant at 0,2963

There is not enough proof to say that (+) and (-) are different for product B

---

Hypothesis test (Mann-Whitney test) for difference between (+) and (-), **product C**

Response is totW

$H_0: C+ = C-$

$H_1: C+ \neq C-$

Test of  $ETA1 = ETA2$  vs  $ETA1 \neq ETA2$  is significant at 0,2963

There is not enough proof to say that (+) and (-) are different for product C

---

Hypothesis test (Mann-Whitney test) for difference between (+) and (-), **product Y**

Response is totW

$H_0: Y+ = Y-$

$H_1: Y+ \neq Y-$

The test is significant at 0,2492 (adjusted for ties)

There is not enough proof to say that (+) and (-) are different for product Y



## Is there a difference between the (+) and the (-) regarding first P

---

Hypothesis test (Mann-Whitney test) for difference between (+) and (-), **product B**

Response is firstP

$H_0: B+ = B-$

$H_1: B+ \neq B-$

Test of  $ETA1 = ETA2$  vs  $ETA1 \neq ETA2$  is significant at 0,8345

There is not enough proof to say that (+) and (-) are different for product B at 95% confidence level

Hypothesis test for difference between (+) and (-), **product C**

Response is firstP

$H_0: C+ = C-$

$H_1: C+ \neq C-$

Test of  $ETA1 = ETA2$  vs  $ETA1 \neq ETA2$  is significant at 0,8345

There is not enough proof to say that (+) and (-) are different for product Y at 95% confidence level

Hypothesis test (Mann-Whitney test) for difference between (+) and (-), **product Y**

Response is firstP

$H_0: Y+ = Y-$

$H_1: Y+ \neq Y-$

Test of  $ETA1 = ETA2$  vs  $ETA1 \neq ETA2$  is significant at 0,8345

There is not enough proof to say that (+) and (-) are different for product Y at 95% confidence level

## Is there a difference between the different products regarding totW?

---

### Kruskal-Wallis test for differences between product B, C and Y

---

Response is totW

$H_0: B=C=Y$

$H_1: \text{All not equal}$

$P = 0,001$

$H_0$  can be discarded. There is a difference at 95% confidence level.

### Hypothesis test (Mann-Whitney test) for difference between product B and product Y

---

Response is totW

$H_0: B < Y$

$H_1: B \geq Y$

Test of  $ETA1 = ETA2$  vs  $ETA1 > ETA2$  is significant at 0,0702

$H_0$  can be discarded. There is a difference at 95% confidence level,  $Y < B$

### Hypothesis test (Mann-Whitney test) for difference between product C and product Y

---

Response is totW

$H_0: Y < C$

$H_1: Y \geq C$

Test of  $ETA1 = ETA2$  vs  $ETA1 > ETA2$  is significant at 0,0702

There is not enough proof to say that product  $Y >$  product C regarding totW at 95% confidence level

## Is there a difference between the different products regarding firstP?

---

### Kruskal-Wallis test

---

Response is firstP

$H_0: B=C=Y$   
 $H_1: \text{All not equal}$

$P = 0,007$   
 $H_0$  can be discarded. There is a difference at 95% confidence level.

---

Hypothesis test (Mann-Whitney test) for difference between product Y and product B  
Response is totW

$H_0: Y > B$   
 $H_1: Y \leq B$

Test of  $ETA1 = ETA2$  vs  $ETA1 < ETA2$  is significant at 0,2137  
There is not enough proof to say that (+) and (-) are different for product Y

---

Hypothesis test (Mann-Whitney test) for difference between product C and product Y

Response is firstP

$H_0: C > Y$   
 $H_1: C \leq Y$

Test of  $ETA1 = ETA2$  vs  $ETA1 < ETA2$  is significant at 0,0057  
 $H_0$  can be discarded. There is a difference at 95% confidence level,  $C < Y$

## Appendix 3: Result Interpretation

For this project, only the starting point and the end point were analyzed but to maintain the possibility of analyzing another aspect it was preferable if the weight curves were as similar as possible over all. To quantify the total area the following methodology was used.

$$\text{int}(\text{sample}A) = (|A(1) - \text{avg}(1)| + |A(2) - \text{avg}(2)| + \dots + |A(n) - \text{avg}(n)|) * \frac{\Delta t}{n}$$

Equation 4: Calculation of Int for one sample

$$\text{Rel.int}(\text{sample}A) = \left( \frac{|A(1) - \text{AVGsample}A(1)|}{\text{AVGsample}A(1)} + \frac{|A(2) - \text{AVGsample}A(2)|}{\text{AVGsample}A(2)} + \dots + \frac{|A(n) - \text{AVGsample}A(n)|}{\text{AVGsample}A(n)} \right) * \frac{\Delta t}{n}$$

Equation 5: Calculation of Rel.int for one sample

A(i)=The weight value of sample nr A in the point of time i

n is the number of measurements

$\Delta t$  is the time between the measurements

The deviation area i.e int(samplei) and Rel.int(samplei) was then summed for the whole run, to give a value of total deviation area. This sum is what was compared between the runs.

The error of these approximations is actually 0, because the  $\Delta t$  is as small as the time difference between the measurements in the methods. Instead, the error lies in the measurements due to the 0,5 sec between data points.

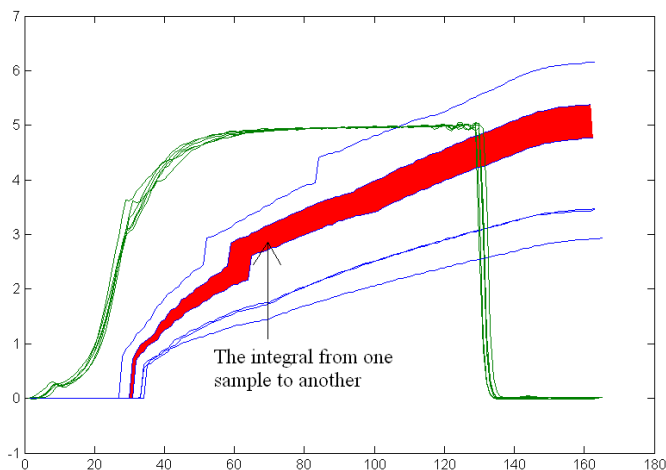


Figure 25: Visualization of the Int. calculations for sample 3 in the DOE

**Appendix 4: X ray images**

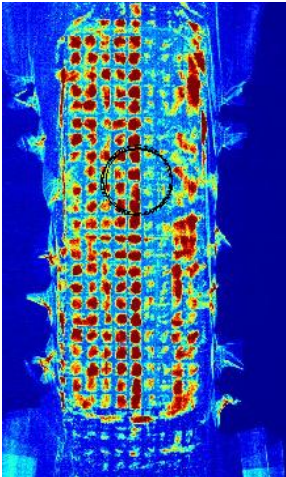


Figure 26: example of a b- sample

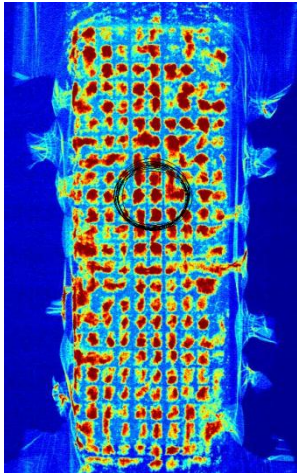


Figure 27: example of a b+ sample

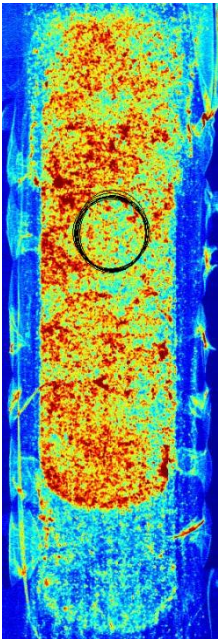


Figure 28: Example of c- sample

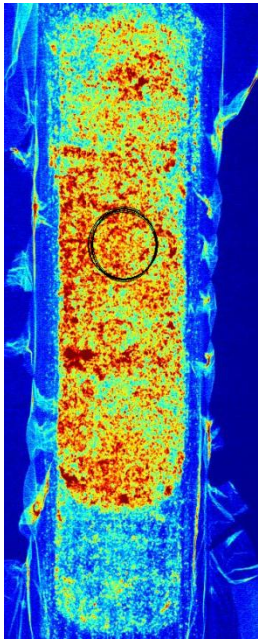


Figure 29: Example of c+ sample

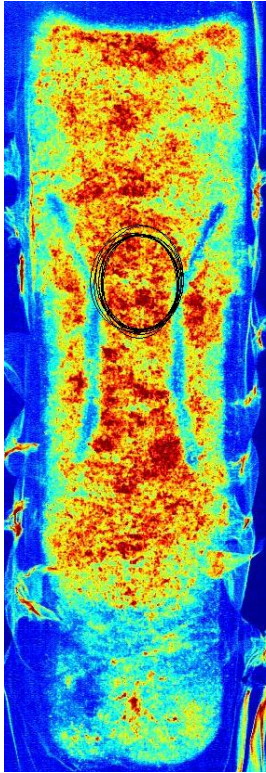


Figure 30: Example of a  $y^-$  sample

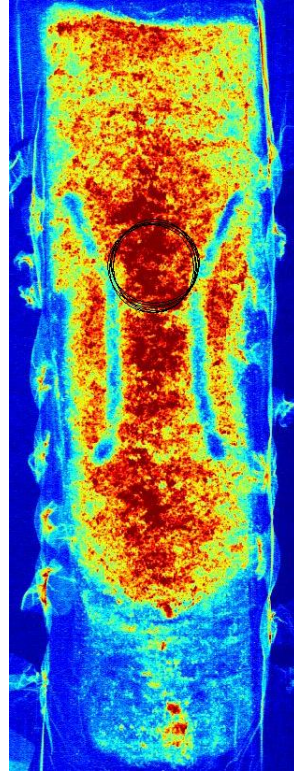


Figure 31: Example of a  $y^+$  sample

## Appendix 5: Additional equations

$$\frac{p_1}{\rho \cdot g} + \frac{u_1^2}{2 \cdot g} + z_1 = \frac{p_2}{\rho \cdot g} + \frac{u_2^2}{2 \cdot g} + z_2$$

Equation 6: Bernoulli's equation for an ideal system

Where:

- The first term=the static pressure
- The second term=the dynamic pressure
- The constant=the atmospheric pressure

Equation 6 states that if the inner diameter is reduced, the driving force will not be reduced which means the time for the liquid to travel through a tube would decrease in an ideal system.