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Optimal peelable seals in packaging concepts undergoing thermal processing

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Introduction

Having an easy-to-open yet safe closed seal is a crucial aspect, both for convenience and for the quality of the food product. Thermal processing of packaging is a widely used step in the food and medical sector, but to the best of our knowledge, research into the post-seal quality during and after (thermal) processing especially for peelable seals has not been investigated thoroughly.

The objective of this best practice guide is to increase process stability when peelable packages are thermally processed. This is achieved by seal strength tests of film samples at several temperatures. Additionally, whole package tests are proposed to determine the influence of the packaging design and the peel performance of the whole package. By using a numerical simulation model for investigations on the packaging level, the design of the packaging geometry and seal contour will be considered in an optimization for the first time. The guide brings these concepts to the work floor of interested companies.

This guide allows food producers to increase the stability of their processes and design more convenient packaging concepts, while reducing the effort to optimize their sealing parameters. This, in turn, will lead to a decrease in waste generation. Because of the demographic change, the market for convenient food and ready meal products is expected to increase steadily according to multiple reports, and this project aims at filling the knowledge gap in industry to pack such products in an optimal way. Companies of the whole process chain (packaging machine producers, material suppliers, packers) can benefit from the guide by offering a set of well-chosen and easy-to-use methods to innovate their business.

The first chapter gives a literature study and generic project results. In the second chapter best practices can be found to assist the quality assessment in your company. The application of these methods, along with additional project results are given in chapter 3. In the end of that chapter a summarizing figure shows how the methods can be combined to evaluate and optimize peel performance of packaging concepts undergoing thermal processing. In the last chapter contact information is given of the research partners of the ThermoPeel consortium.

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1 Literature study and generic results ThermoPeel

In this first chapter a brief overview is given of previous studies on the peel performance of packaging concepts undergoing thermal processing along with generic results of the ThermoPeel-project to act as a first-line assistance.

1.1 Literature study

The consumer demand for convenient food packaging has increased steadily during the last years. Packaging material producers developed peelable heat seal materials to meet this demand. Morris[i] describes three main methods to control the seal strength within the peel seal range: adhesive, cohesive and delamination peel failure. Depending the desired properties (seal temperature window, aging effects, tamper evidence by stress whitening, angel hair presence, combination of peel and lock-seal in one material, tailorable seal strength, etc.) material producers can propose suitable heat seal materials.

The packed product undergoes thermal processing after sealing to extend the shelf life. At the food company, during transportation and storage, at the store and finally at the consumers place it can be cooled and/or heated. Cool processing can be differentiated in chilling at temperatures from 0 to 5°C and freezing at temperatures from -24 to -18°C. Chill processing extend the shelf life by decreasing microbial activity and biochemical reactions. Retorting is a term that is widely used in the food industry to describe the process where the food is heated in the package. Temperatures around 121°C are maintained for a certain time to decrease the amount of microbial and enzymatic activity. At lower temperatures, generally below 105°C, the heating regime is referred to as pasteurization.[ii]

Polyethylene (=PE) and polypropylene (=PP) are most widely used as seal materials in thermal processed food packages. These materials are compared in this comparison of cool and heat processing. Cool processing demands materials that are stable at low temperatures. In general, PE grades are preferred over PP as seal material because of the low glass transition temperature range (LDPE and LLDPE: -125 → -90 °C) leading to plastic behaviour in freezing and cooling temperatures. The glass transition temperature range of many PP-grades (homopolymer and random copolymer: -24 → -6 °C) is at typical freezing temperatures for the food industry, leading to brittle behaviour at these temperatures. Heat processing demands materials that are stable at high temperatures and for this reason PP-grades are preferred over PE. PP, especially the homopolymer, has a high melting temperature range (homopolymer: 161-170 °C) and will be in a solid state during sterilization. Because of the low melting temperature range of many PE-grades (LDPE and LLDPE: 98 → 124) this material will weaken during sterilization and safe heat processing cannot be ensured.[iii]

Insight in the post seal quality during and after thermal processing, however, **is rather limited**. Dixon[iv] proposed a method to study the peel properties of medical packaging materials at elevated temperatures. The peel strengths of coated Tyvek® sealed with PET/PE is measured with a modified tensile testing machine at 20-65 °C. A 50 % reduction in peel strength is observed at 43°C compared to room temperature. Iwasaki[v] tested the influence of a post package sterilisation process (30 min. – 110°C) on the peel strength of a multilayer PE film (LLDPE and HDPE) in relation with the shape of seal bars. The peel strength decreases and the effect of the shape of the seal bar becomes less significant after sterilisation. In a newer paper[vi] the composition of the inner seal layer is varied. With HDPE seal material a smaller reduction of seal strength is achieved after sterilisation at heat sealing temperatures well below the melting peak temperature. The stability of LDPE seal material after sterilization can only be maintained when seal temperatures equivalent to the melting peak temperature are used. From a commercial point, the DIC-group[vii] is developing a new FDA-approved topfilm with improved peel strength at elevated temperatures to withstand the sterilization process when sealed against a PP container.

In a technical paper[viii] Elleithy and Zhang demonstrate the effect of autoclaving (60 min. – 121 °C) on the peel strength of two types of peelable PP topfilm sealed against a PP sheet or cup. The peel strength is decreased after thermal processing. During thermal processing the molecules can rearrange themselves into a more stable state. This rearrangement can lead to a decrease in molecular interaction at the interface with a lower peel strength as a result.

The **knowledge about the internal pressure build-up of flexible packages during thermal processing is also rather limited**. Ghai[ix] proposed a method to measure the internal pressure profile of food systems with an aluminium pressure tight module. Due to chemical reactions of components from the food with aluminium it is suggested to use an inert material such as stainless steel to produce the module. Mathematical models for each food system are suggested to predict the internal pressure profile. The accuracy of the predictions of these models varied from 4 to 13% error. These models are inadequate to predict internal pressure profiles of more complex food systems because of their simplicity (Perfect Gas and Raoult’s law). It is suggested to include water activity to improve these models. The company “Stress Engineering Services[x]“ developed a proprietary algorithm “Bi-Path” to calculate the state of the gas in the head space of flexible pouches during sterilization. The bonding failure model is a critical element of the sterilization simulation. The influence of elevated time and temperature are taken into account to simulate the entire sterilization process. besides this information, there is no further literature available which describes the simulation of the stresses to the packaging due to thermal processing.

Concluding, as of today the public knowledge about the balance between peelability and stability of seal materials during thermal processing of flexible packages is rather limited. There currently are no wide-spread simulation models to optimize this compromise. One outcome of the ThermoPeel project will be the publication of this best practice guide to fill this gap.

1.2 Generic results of ThermoPeel project

Cool processing with peelable PE seal layer:

Seal strength increased during cool processing with peelable topfilms, sealed against bottomwebs. There was no influence of processing temperature on seal strength after processing, when the sealed sample is in standard condition (23 °C, 50 % RH). With peelable pouches, sealed to itself (two peelable surfaces), there was no influence of processing temperature during and after processing.

Hot processing with peelable PP seal layer:

95 and 121 °C are considered as processing temperatures and compared with 23 °C. Seal strength decreased during hot processing with peelable pouches and topfilm-bottomweb concepts. The strongest decrease is observed at 121 °C. The rate of decrease is material dependent. In some cases a decrease in seal strength is observed after thermal processing as well. The decrease in strength during heat processing should be taken into account when designing peelable pouches undergoing thermal processing.

Case studies:

In the case studies the maximization of seal strength was considered as optimal seal performance for non-peelable packaging concepts. Tropical conditions (38, 50 and 60 °C) had no impact on the peel performance of coated paper during and after thermal processing. A decrease in seal strength is observed with coated paper, during processing at 95 °C.

2 Best practices to evaluate peel performance during and after thermal processing

In this second chapter methods are listed and explained to assist the quality assessment of peelable packaging concepts undergoing thermal processing.

2.1 Seal characterization

The ASTM F88[xi] standard describes a method to test seals of flexible barrier materials. This method can be applied on **film samples**. The following steps summarize the method described in the standard and deviate, if necessary, for applying it with thermal processing of peelable packaging films.

- Sample dimensions: The length of the samples must be high enough to ensure clamping of the peel arms. A very wide seal is avoided to prevent heterogeneity of the sealed sample because of local differences in temperature. Sample widths of 30 or 50 mm can be used. Samples are prepared by sealing materials at specified seal temperature, seal time and seal pressure. Prior to characterization, samples are cut in the centre to a width of 15, 25 or 25.4 mm.
- Conditioning time: This is the time between sealing and testing. From a practical point of view it should be low enough to allow quick testing and high enough to reach stability of the sealed sample. This will be explained in section 2.2.
- Sealed samples are tested with a universal testing machine, in tensile direction, with a load cell with an appropriate work range. When a flexible film is sealed on itself an unsupported T-peel test can be done to characterize the seal. When a topfilm-bottomweb structure is used the rigid bottomweb is clamped at one side while the more flexible topfilm makes a 180° bend. A rigid alignment plate can be used to maintain the angle during the test by preventing the bottomweb from bending. The distance between the clamps should be specified, typical values of 10 mm for extendible materials and 25 mm for less extendible materials are used. The speed of the test is between 200 and 300 mm/min.
- Responses: Figure 1 shows typical peel performance responses. Seal strengths are calculated by dividing force with sample width. Maximum and average strength are used for peelable materials. The centre 30% of the raw curve can be used to calculate the average value. The area under the raw curves represents the peel energy.

Seal separation modes that are suggested in the standard such as adhesive, cohesive and delamination peel are visually determined. It is common that a combination of modes apply to the failure of one sealed sample. These modes should be reported.

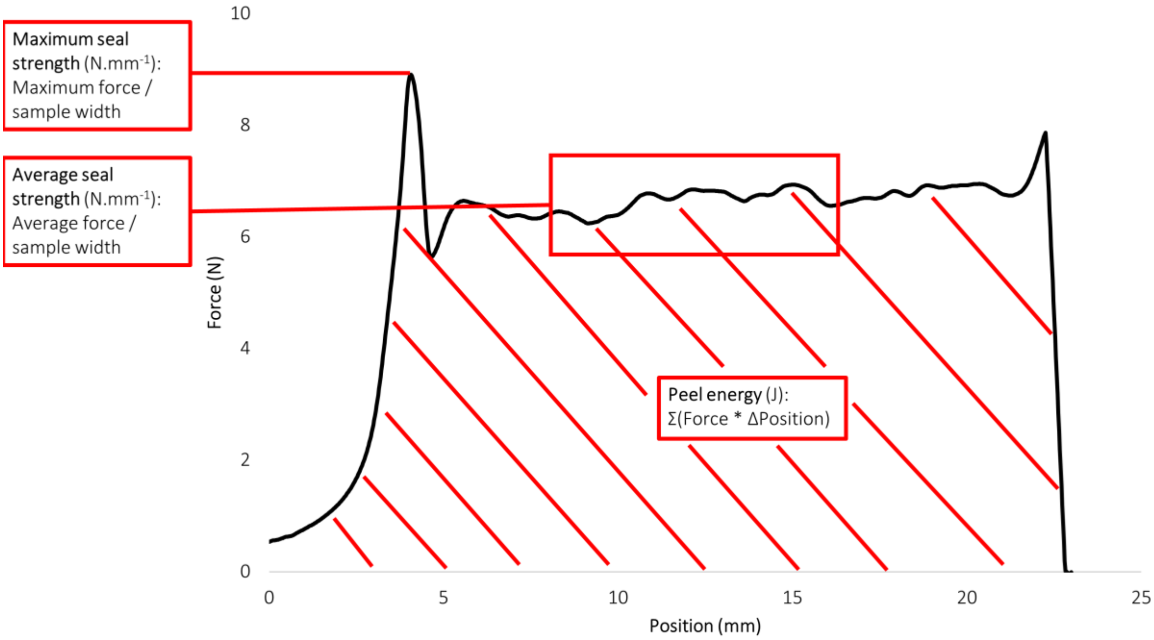

Figure 1: Peel performance responses

2.2 Processing time

The **relevant time range** differs for cool and heat processing. Very long processing times of several weeks and even months are widely used in cool processing. With hot applications such as pasteurization and sterilization, processing time is mostly limited to several hours. To check the influence of processing time on peel performance two commercial material types are considered for cooling (PE) and heating (PP). Peel performance is evaluated with a seal strength test. As previously explained in the literature study PE and PP are considered as well performing conventional seal materials for respectively cool and hot processing. The tests were done on commercially available materials.

i. Cooling

To check the influence of cooling time on maximum seal strength, two concepts were considered: a peelable PET/PE-EVOH-PE 12/45 film sealed as **pouch** and a **topfilm-bottomweb** application, where this film is sealed to a PET/PE 250/35 non-peelable bottomweb. To simulate industrial processing sealed samples with a width of 15 mm were transferred immediately after heat sealing to temperature chambers of -18, 4 and 23 °C. Prior to seal characterization seals are conditioned for 5 minutes at 50 % relative humidity and 23 °C.

Figure 2 and Figure 3 show the influence of cooling time, relevant for food packages, on the maximum seal strength of the two considered concepts. The red line indicates the seal strength directly after sealing, without thermal processing. The pouch slightly decreased and the topfilm-bottomweb slightly increased seal strength at high cooling time at all considered temperatures. Stabilization occurred after 1 day. The impact of cooling time on average seal strength and peel energy is similar.

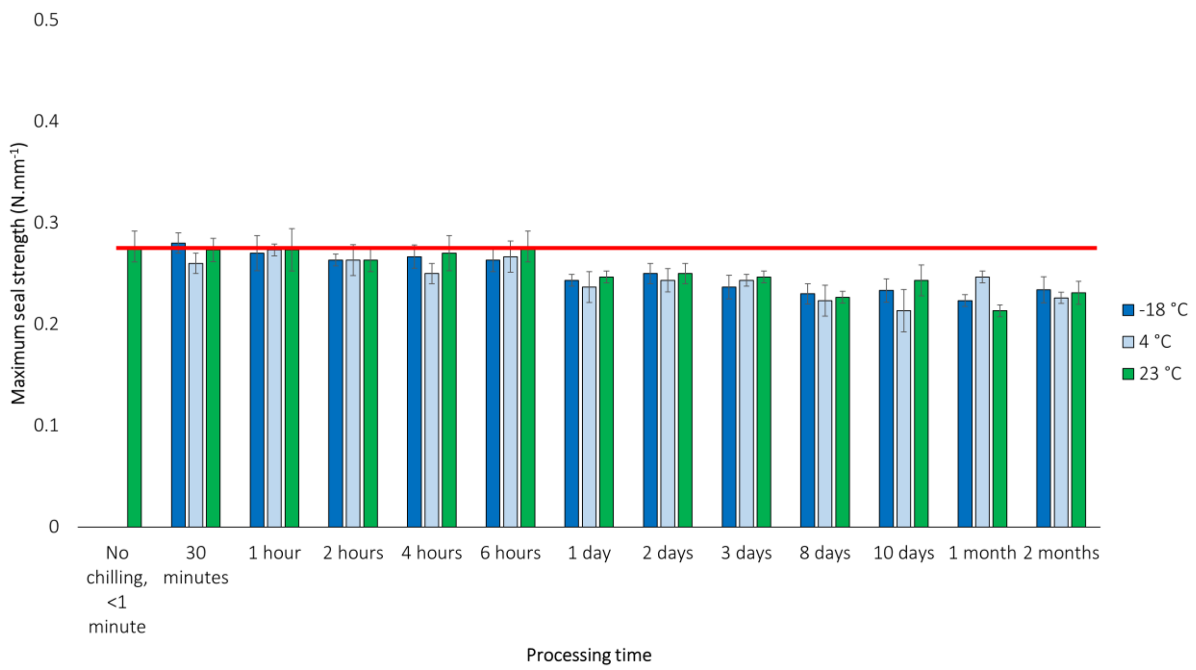

Figure 2: Influence of processing time on maximum seal strength of PET/PE-EVOH-PE peel 12/45 pouch, n=3: average value and standard deviation

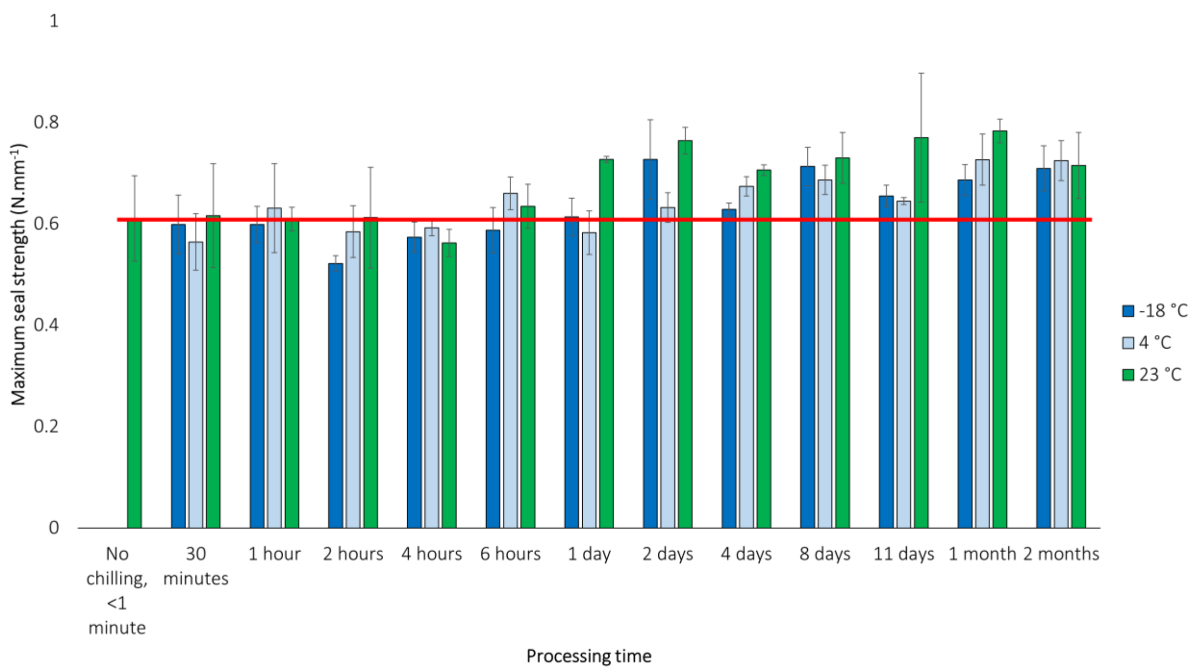

Figure 3: Influence of processing time on maximum seal strength of PET/PE-EVOH-PE peel 12/45 topfilm, sealed to PET/PE 250/35 non-peel bottomweb, n=3: average value and standard deviation

ii. Heating

To check the influence of heating time on the maximum seal strength, two concepts were considered: a peelable PET/PP 12/45 film sealed as **pouch** and a **topfilm-bottomweb** application, where this film is sealed to a PA/PP 60/100 non-peelable bottomweb. To simulate industrial processing sealed samples with a width of 15 mm were transferred immediately after sealing to temperature chambers of 23, 95 and 121 °C. Prior to seal characterization seals are conditioned for 5 minutes at 50 % relative humidity and 23 °C.

Figure 4 and Figure 5 show the influence of heating time, relevant for food packages (5 minutes → 240 minutes) on the maximum seal strength of a pouch and a topfilm-bottomweb concept. There is no influence of heating time with the pouch concept. With the topfilm-bottomweb concept, a slight increase of seal strength can be seen with the heated samples after 120 minutes processing, but these differences are rather limited. The impact of heating time on average seal strength and peel energy is similar.

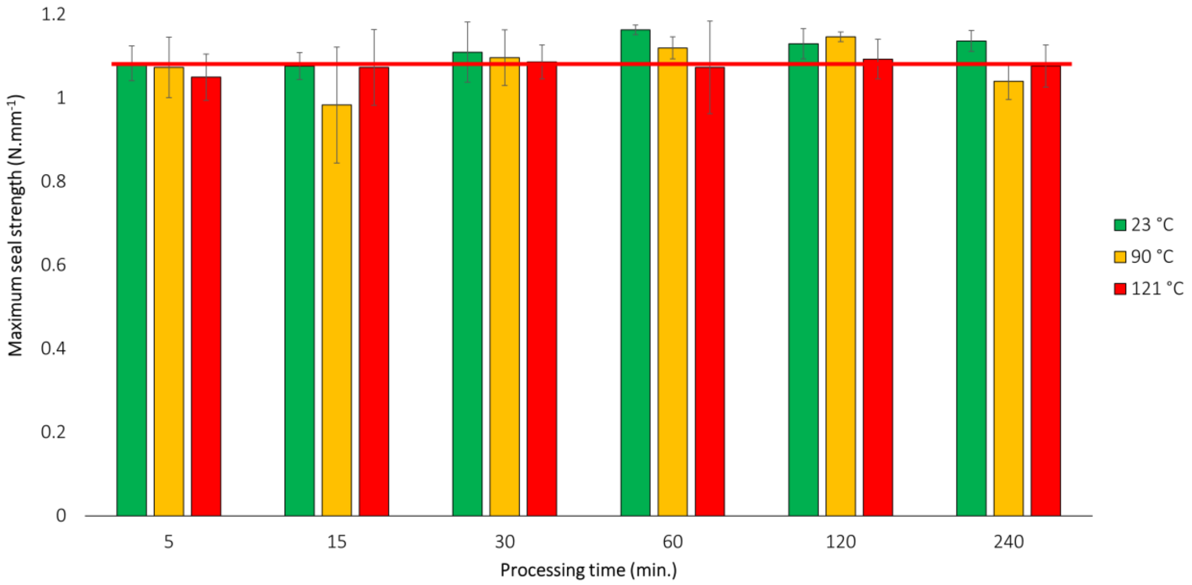

Figure 4: Influence of processing time on maximum seal strength of PET/PP peel 12/45 pouch, n=3: average value and standard deviation

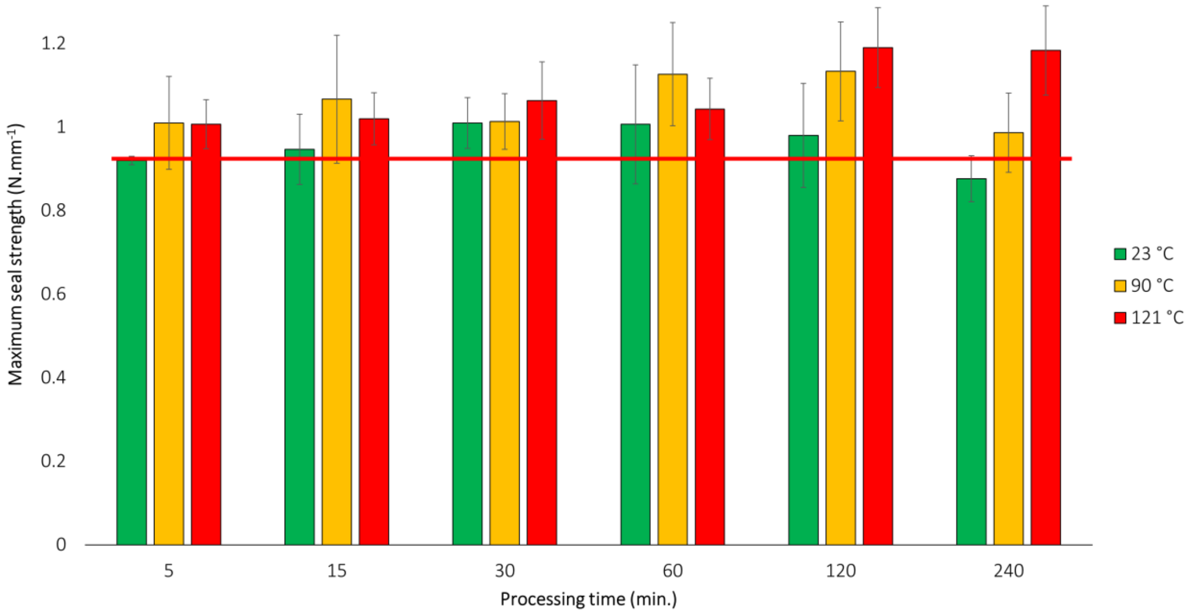

Figure 5: Influence of processing time on maximum seal strength of PET/PP peel 12/45 topfilm, sealed to PA/PP 60/100 non-peel bottomweb, n=3: average value and standard deviation

iii. Conclusion

The influence of processing time on peel performance is material dependent and rather limited. In the ThermoPeel project, processing time is kept constant at 15 minutes. Fixing the process time is important to limit the amount of tests. Seal parameters (seal temperature, seal time, seal pressure) and processing temperature could be varied on several packaging concepts in a design of experiment approach.

2.3 Design of experiment approach

In order to investigate the influence of the considered process parameters on the seal behaviour, we recommend to use a design of experiment approach. We outline a six-step strategy that can be followed to efficiently execute the research.

- <u>Define design space:</u> In this step, the considered process parameters ("factors") are listed, as well as the minimal and maximal values of each of these. Both the choice of the considered parameters and their limits can be based on prior knowledge / experiments, recommendations from the film producers, or simply based on practical requirements and limits. It is noted that not all factors are truly continuous and come with minimal and maximal values. Indeed, for the processing temperature interest could only be for two or three discrete values, or for some applications non-numeric factors might be considered.
- <u>Set up an experimental design:</u> The number of factors as well as their characteristics drives the choice of the experimental design. When only continuous factors are considered, central composite designs are simple yet effective designs for finding an optimum. If a combination of continuous and categorical factors is considered, or when prohibited combinations of factors exist, one should use so-called optimal designs. Such designs can be tailored to the application considered and, thus, are extremely flexible. Many software packages such as JMP®, DesignExpert, R and Minitab offer these types of designs.
- <u>Perform experiments:</u> In this third phase, the actual experiments are performed, and responses are recorded.
- <u>Fit response surface model:</u> In order to relate the factors to the responses, a statistical model has to be fitted to the data. This can be performed in the exact same software packages mentioned in previous point. A full quadratic model is a good starting point, from which non-significant terms are removed to come up with a simplified final model.
- <u>Optimize input parameters:</u> The goal of the model is to find those combinations of the factors that result in a desirable outcome (seal quality). In order to do so, the outcomes are re-scaled or re-defined into so-called desirability functions. The desirability function maps the outcomes between 0 (not desirable) to 1 (highly desirable). When multiple responses are considered, a desirability function is defined for each response separately, and these individual desirabilities are combined into one overall desirability. This is done by computing the geometric mean of the individual desirabilities.
- <u>Experimental validation:</u> The optimal setting as found in the optimization step has to be validated in this last step. Hereto, a number (typically between 3 and 10) of seals are created at the optimal settings, and the 95% confidence interval of these measurements is calculated. If this confidence interval holds the predicted value as obtained by the model, the model is considered as validated. If it is not the case, it is recommended to augment the design and to re-calculate (update) the model.

2.4 Finite-Element load simulation

i. Burst pressure

The burst test is a method to evaluate the seal strength **on packaging level** and to investigate the internal pressure built up due to thermal processing. It enables a defined pressurizing of the package. In ThermoPeel the inflation rate is set to 5 mbar/s and the internal pressure is increased up to the point where the package fails. Two different test set-ups are used to determine the parameters (film combination, seal parameter settings, deformation) influencing the seal strength. A unrestrained burst test resulting in a free deformation of the package and a restrained burst test with a limitation on the deformation due to a defined, adjustable gap between the plates of the test rig as shown in Figure 6. At a package height of 21 mm the gap size is set between 26 – 46 mm. The packages are compared regarding their deformation during pressure increase, their burst pressure and failure pattern in order to identify the load distribution on the package and the sealed seal.

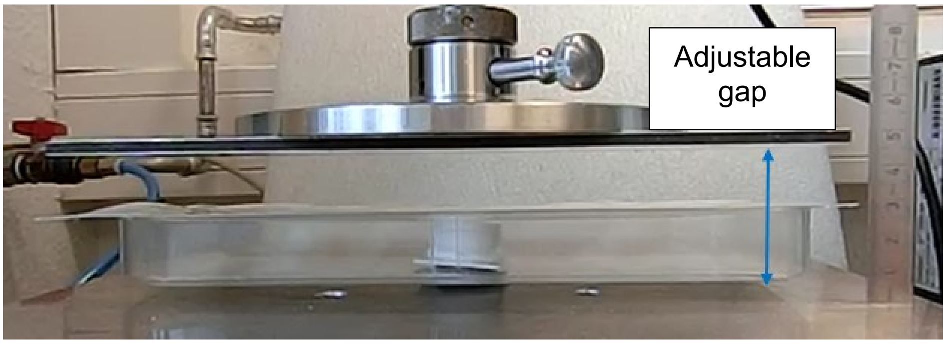

Figure 6: Test rig for conducting burst pressure tests with adjustable gap between the plates

ii. Simulation set up

A whole set of forming and sealing tools must be developed to test the effect of changes in geometry (e.g. seal contour, tear contour, tray geometry, etc.) or packaging concept on the load of packages experimentally. Therefore, a numerical simulation model of the burst test scenario is set-up, to evaluate the applied load to the seal at packaging level virtually. The results of the aforementioned experiments are used to validate the model. The model is implemented and calculated with the commercially available software LS-DYNA. The model structure, as described in the following, enables a quick modification of model parts (e.g. tray geometry, film combination) in order to conduct parameter studies:

- <u>Geometry model:</u> The geometry model of the packaging is set up in a CAD program and meshed within the commercially available software ANSYS Mechanical. The tray and top film are depicted by shell elements, the sealed seal by solid elements. The element size varies dependent on the required calculation accuracy (Figure 7). The highest accuracy is required in the area of the sealed seal. In ThermoPeel the element size at the sealed seal is set to 0.5 mm. This enables a sufficient accurate calculation with the shortest possible calculation time. Subsequently, the generated geometry model is transferred into LS DYNA. The geometry model defines the parameters tray and top film geometry, seal contour and film thickness by defining the thickness of the shell elements.

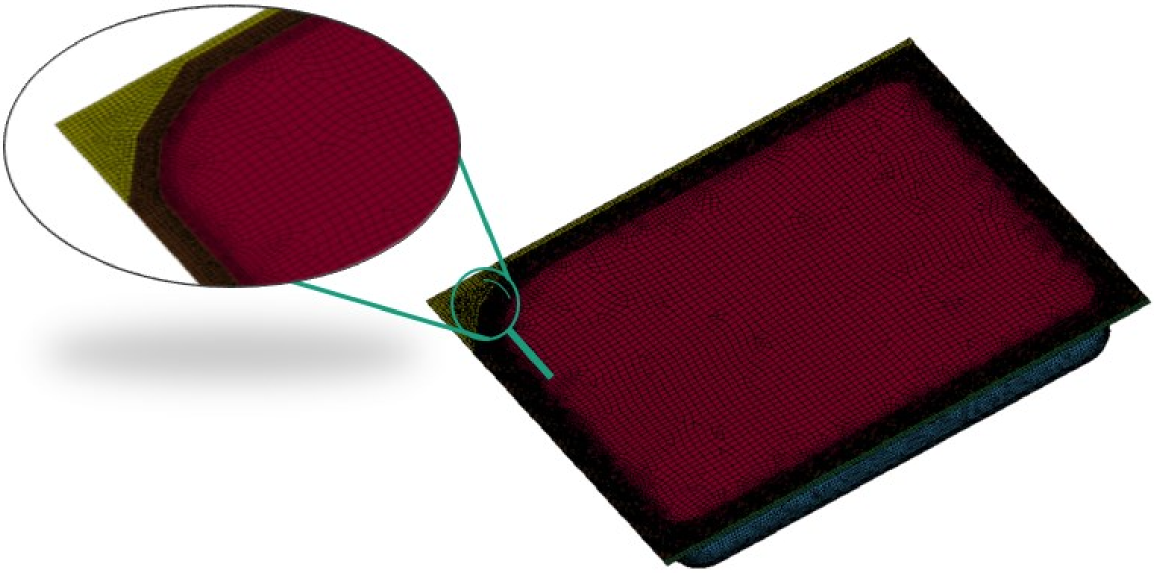

Figure 7: Meshed geometry model of the package with a locally finer element size in the area of the sealed seal

- Material model: The tray and top film are both mechanically characterized by performing uniaxial tensile tests according to DIN EN ISO 527-1[xii]. An elasto-plastic material model is used to model the film material. The material model parameters, effective plastic strain and yield stress, are calculated based on the measured force-displacement curves.
- Failure model: To set up the failure model and to determine the required peel force to open the sealed seal, peel tests are performed on sample level based on DIN SPEC 91441[xiii], but with varied tear angle. Measurements were conducted up to four tear angles between 45° and 135° for each simulated packaging concept in ThermoPeel. This is necessary because the required peel force is highly dependent on the peel angle and therefore, the seal strength of the package also varies along the seal contour depending on the deformation of the package due to the internal pressure increase. The material parameters, required to model the cohesive behaviour of the sealed seal, are derived based on the approach described in Geißler[xiv]. At this, the separation energy and maximum traction of the sealed seal, required as material parameters for the cohesive element formulation, are calculated based on the experimentally determined seal strength at different tear angles. Therefore, an optimization of the aforementioned material parameters was conducted by using adaptive simulated annealing by means of the software LS-OPT. The aim of the optimization study was to determine the optimal material parameters in tangential and normal direction of the cohesive elements to achieve the required peel force depending on the varying peel angles during the internal pressure increase.
- Simulation load scenario and boundary conditions: The internal pressure load scenario is set up by applying the recorded pressure course from the experiments into the simulation software. Therefore, the recoded pressure course is extrapolated as shown in Figure 8. Finally, an implicit time integration is applied to calculate the simulation model. The simulation results are used to compare the load on different packages.

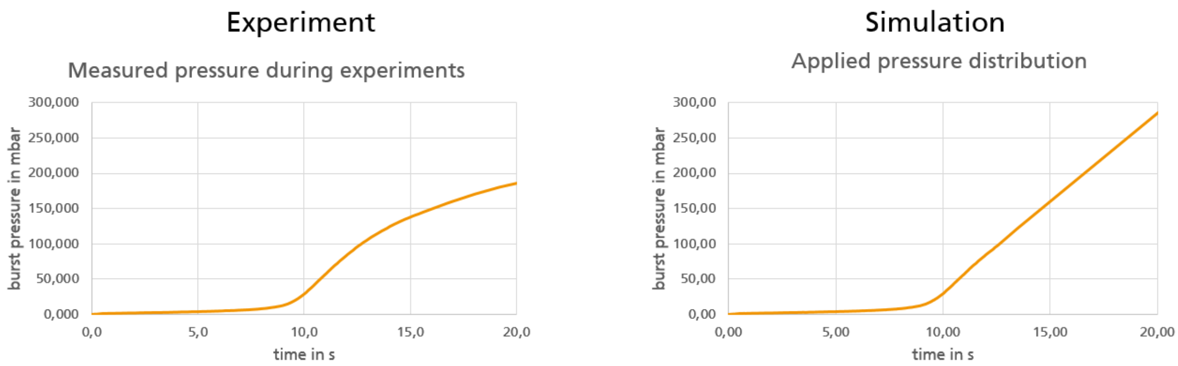

Figure 8: Measured pressure curve recorded during burst pressure measurements (left), extrapolated pressure curve applies within the simulation (right)

2.5 Pre-calculation of opening force

Based on the correlations identified in the IGF-project 18613 "EasyReliablePeel" a calculation tool was developed to pre-calculate the opening force of a package. With the help of this web-based software tool, the expected opening force is predicted based on the seal contour and the seal strength of the packaging material to be used. For the seal strength, values can be entered lengthwise and crosswise to the machine running direction. The values in between are interpolated.

The tool enables the evaluation of the opening force already in the design process by comparing the expected opening force with the reference values for easy opening (IVLV-Merkblatt 106[xv]). Therefore, the seal contour can be optimized to achieve an easy-to-open package.

- Input data:
    - Sealed seal contour as dxf-file
    - Running direction of the film based on the machine direction
    - Opening direction in relation to the sealed seal contour
    - Seal strength lengthwise and crosswise to the running direction of the film.
- Results:
    - Peel line along the opening path
    - Opening force along the opening path.

3 Practical examples

In this third chapter, methods of the previous chapter are applied on practical examples. In the end of this chapter a summarizing figure shows how methods can be combined to evaluate and optimize the peel performance of packaging concepts undergoing thermal processing.

3.1 Sample level: six step approach

The quality of peelable packaging materials can be evaluated and optimized in a six steps approach: defining a design space, setting up an experimental design, performing experiments, fitting a response surface model, optimizing input parameters and validating the optimum. The experiments in the first, third and last step are seal strength tests. This approach would be also applicable with other tests with numerical output. Seal separation modes are reported but not optimized because of the difficulty of optimizing non-numerical data with this approach. All steps are shown in one example for cool processing of peelable topfilm, sealed against a bottomweb with PE seal layer. A peelable PET/PE-EVOH-PE 12/45 film is sealed as pouch and as topfilm against a PET/PE non-peelable bottomweb. The detailed method is shown for the topfilm-bottomweb combination.

i. Define design space

The influences of processing temperature and seal parameters (temperature, time and pressure) on the peel performance (maximum and average seal strength, peel energy) are of high interest. 3 processing temperatures are considered: freezing at -18 °C, cooling at 4 °C and the standard temperature of 23 °C. The boundaries of seal pressure and time can be respectively set to the working range of the sealer (1 → 4 $N.mm^{-2}$) and the relevant range for the considered packaging concept (topfilm-bottomweb: 1.0 → 3.0 s). The boundaries of seal temperature are not known, so **preliminary tests should be performed to determine the minimum and maximum seal temperature of the design space and to have a first impression of peel performance**. Seal strength tests are performed in standard conditions at relevant fixed settings for seal pressure (1.0 $N.mm^{-2}$) and seal time (2.0 s), while seal temperature is varied to check the peel performance in a relevant temperature range (100→180 °C of the upper jaw, lower jaw is kept constant at 50 °C to simulate the industrial process). From upper jaw temperatures of 140 → 180 °C cohesive peeled seals are generated with the following characteristics: no clear opening and end peaks, maximum seal strength ≈ average seal strength ≈ 8 N. 15 mm^{-1}. A raw seal strength curve is shown in Figure 9. Figure 10 shows the influence of upper jaw temperature on maximum seal strength. Max seal strength rises from temperatures of 110 → 140 °C to a plateau value between 140 → 180 °C of 8 N. 15 mm^{-1} or 0.5 $N.mm^{-1}$. Based on these results the boundaries of seal temperature are 130 and 180 °C. 130 °C is a good minimum because it is just before the edge with 2.0 s seal time and probably on the edge at 3.0 s seal time. 180 °C is a good maximum because the concept is still cohesively peeled and 180 °C was previously considered as a maximum temperature for this application.

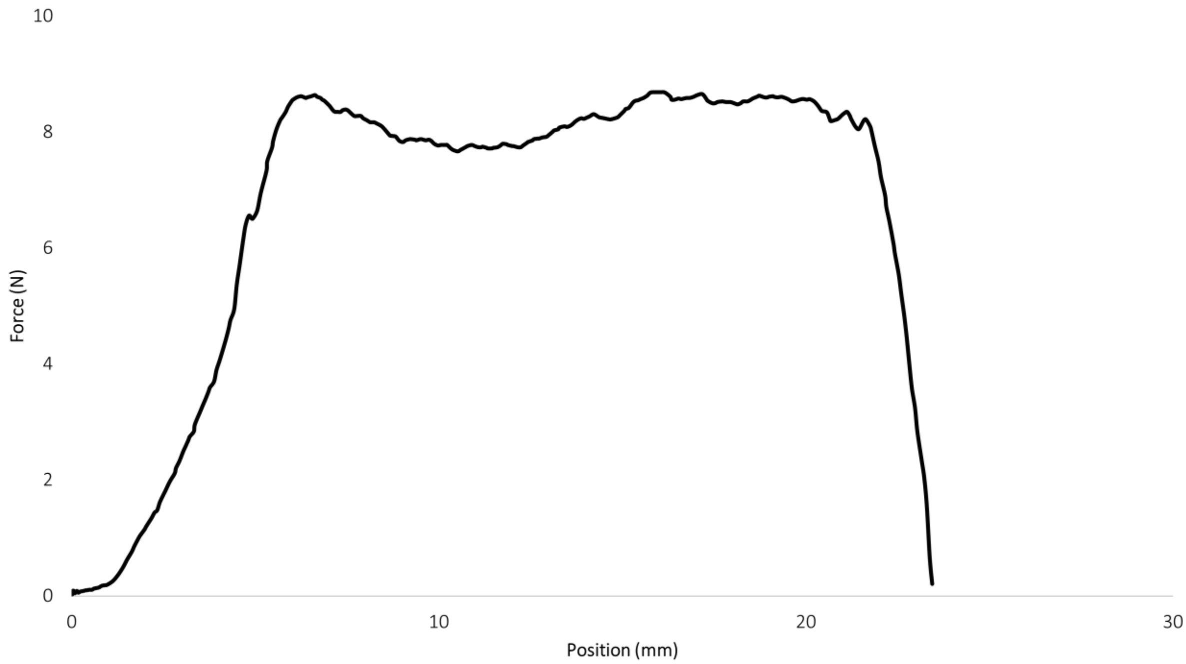

Figure 9: Raw seal strength curve of peeled PET/PE-EVOH-PE (peel) 12/45 topfilm, sealed against a PET/PE 250/35 bottomweb

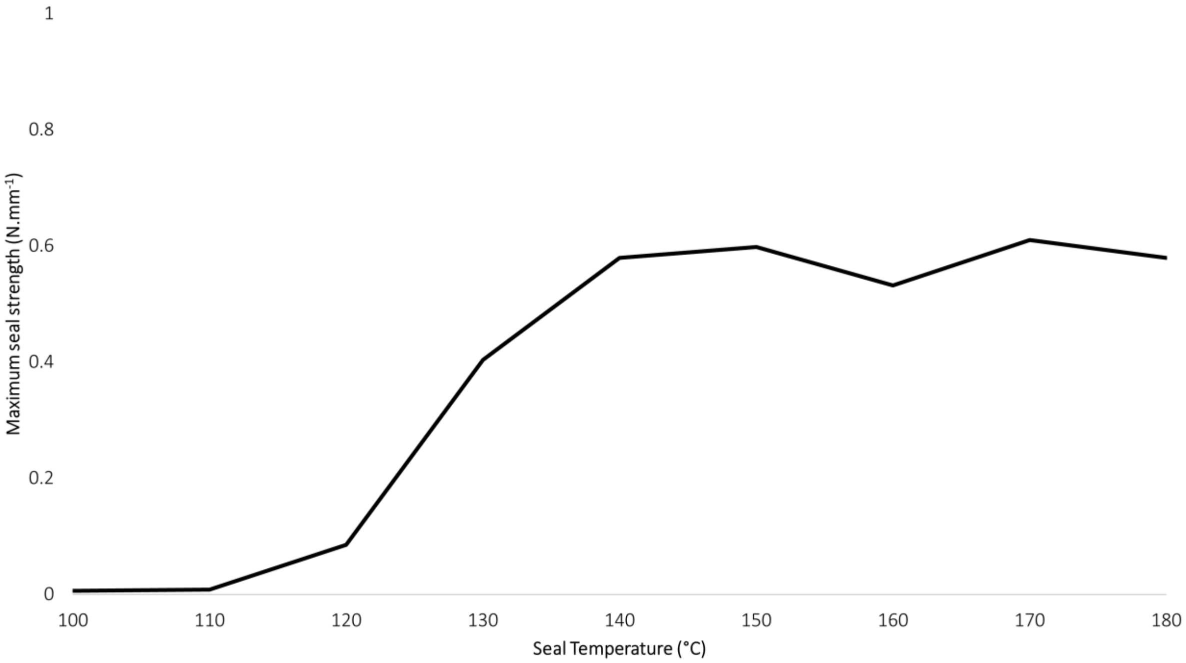

Figure 10: Influence of upper jaw temperature on maximum seal strength of PET/PE-EVOH-PE (peel) 12/45, sealed against a PET/PE 250/35 bottomweb

ii. Set up an experimental design

The processing temperature is a categorical (nominal) variable because there is only an interest in these temperatures and not in the values in between. Seal parameters (temperature, time and pressure) are continuous variables because there is an interest in the values in between. Combining these 4 variables is not a standard setting, and requires so-called optimal designs that are tailored to the application. They are not pre-defined and cannot be found in textbooks, instead, they are generated for the specific application. We chose a custom design that allows for fitting a full response surface model for the three continuous variables. This allows to find a true optimum, or to match a given target value. We combine this capability with the possibility to estimate the effect of the categorical processing temperature, and allow the processing temperature effect to be different for the different continuous variables. These requirements ask for performing experimental runs using 24 different combinations of these 4 variables. This is far less than performing all possible combinations, which would lead to at least 34 = 81 runs. Table 1 shows the experimental design in the first 5 columns in a random order of runs.

iii. Perform experiments

For each of these runs 2 samples were tested. One sample during processing, with 15 minutes fixed processing time. The other sample is tested after processing, also with this sample processing time was fixed at 15 minutes, in standard conditions (23 °C, 50 % RH) within a timeframe of 4h after processing. As previously mentioned in section 2.1 three responses were considered: average seal strength, maximum seal strength, and peel energy. Table 1 shows the results of all responses in the last 6 columns.

Table 1: Experimental design + results of PET/PE-EVOH-PE (peel) 12/45, sealed against a PET/PE bottomweb

					During processing			*After processing*		
run	T (°C)	t (s)	p ($N.mm^{-2}$)	$T_{processing}$ (°C)	Av. seal strength ($N.mm^{-1}$)	Max. seal strength ($N.mm^{-1}$)	Peel energy (J)	Av. seal strength ($N.mm^{-1}$)	Max. seal strength ($N.mm^{-1}$)	Peel energy (J)
1	155	2.0	2.5	-18	1.07	1.24	0.34	0.75	0.82	0.20
2	180	1.0	4.0	4	0.95	0.98	0.26	0.65	0.67	0.17
3	155	1.0	1.0	4	0.81	0.98	0.21	0.19	0.33	0.02
4	180	3.0	1.0	23	0.67	1.26	0.23	0.67	1.26	0.23
5	155	2.0	2.5	23	0.68	0.81	0.18	0.68	0.81	0.18
6	130	3.0	4.0	-18	0.7	1.04	0.21	0.41	0.55	0.08
7	180	2.0	1.0	4	0.75	1	0.28	0.78	0.8	0.23
8	180	3.0	2.5	4	2.62	2.66	0.24	2.03	2.06	0.29
9	155	3.0	4.0	23	1.74	1.77	0.14	1.74	1.77	0.14
10	130	1.0	4.0	23	0.11	0.17	0.02	0.11	0.17	0.02
11	130	3.0	2.5	23	0.5	0.61	0.13	0.5	0.61	0.13
12	130	3.0	1.0	4	0.96	1.03	0.21	0.66	0.72	0.13
13	130	1.0	1.0	-18	0.02	0.05	0.01	0.01	0.01	0.00
14	180	1.0	1.0	-18	1.06	1.26	0.42	0.39	0.62	0.10
15	130	1.0	2.5	4	0.13	0.26	0.02	0.01	0.01	0.00
16	155	3.0	1.0	-18	0.91	1.26	0.40	0.66	0.72	0.17
17	155	1.0	4.0	-18	1.03	1.19	0.30	0.51	0.63	0.14
18	180	1.0	2.5	23	0.69	0.71	0.19	0.69	0.71	0.19
19	139	1.3	1.0	23	0.4	0.63	0.07	0.4	0.63	0.07
20	155	2.0	2.5	-18	1.14	1.27	0.43	0.71	0.74	0.18
21	180	2.0	4.0	23	3.3	3.33	0.38	1.34	1.38	0.05
22	180	3.0	4.0	-18	3.3	3.33	0.38	2.18	2.2	0.33
23	155	3.0	4.0	4	2.52	2.65	0.38	2.08	2.09	0.27
24	130	2.0	4.0	4	0.76	0.82	0.12	0.44	0.6	0.08

iv. Fit response surface model

A quadratic model with interactions for the three continuous variables of the following form is chosen: $\hat{y} = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{12} x_1 x_2 + \beta_{23} x_2 x_3 + \beta_{13} x_1 x_3 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{33} x_3^2$
This model contains main effects (plane), interactions (twisted plane) and quadratic effects (curvilinear).

The effect of the categorical processing temperature is introduced by adding interactions between the three continuous variables and the categorical variable. Non-significant effects are not included in the model. For average seal strength during processing a model is obtained with a least squares fit as shown in Figure 11. This figure shows main effects (seal temperature, seal time and seal pressure), interactions (seal temperature * seal time, seal temperature * seal pressure, seal time * seal pressure) and quadratic effects (seal temperature * seal temperature). In this example processing temperature interacts with seal temperature and seal time.

$$
\begin{aligned}
&-4.673113721 \\
&+0.0268247934 \bullet \text{Tseal (°C)} \\
&+0.4425594745 \bullet \text{tseal (s)} \\
&+0.2966783844 \bullet \text{pseal (N.mm-2)} \\
&+\text{Match}\left(\text{Ttreatment (°C)}\right)\begin{pmatrix} -18 \Rightarrow 0.0085322244 \\ 4 \quad \Rightarrow 0.1246165641 \\ 23 \ \Rightarrow -0.133148789 \\ \text{else} \Rightarrow . \end{pmatrix} \\
&+\left(\text{Tseal (°C)} - 155.375\right)\bullet\left(\left(\text{Tseal (°C)} - 155.375\right)\bullet -0.000104222\right) \\
&+\left(\text{Tseal (°C)} - 155.375\right)\bullet\left(\left(\text{tseal (s)} - 2.0141666667\right)\bullet 0.0065832494\right) \\
&+\left(\text{Tseal (°C)} - 155.375\right)\bullet\left(\left(\text{pseal (N.mm-2)} - 2.5625\right)\bullet 0.010419286\right) \\
&+\left(\text{tseal (s)} - 2.0141666667\right)\bullet\left(\left(\text{pseal (N.mm-2)} - 2.5625\right)\bullet 0.1817064975\right) \\
&+\left(\text{Tseal (°C)} - 155.375\right)\bullet\text{Match}\left(\text{Ttreatment (°C)}\right)\begin{pmatrix} -18 \Rightarrow 0.0102415083 \\ 4 \quad \Rightarrow -0.009691825 \\ 23 \ \Rightarrow -0.000549683 \\ \text{else} \Rightarrow . \end{pmatrix} \\
&+\left(\text{tseal (s)} - 2.0141666667\right)\bullet\text{Match}\left(\text{Ttreatment (°C)}\right)\begin{pmatrix} -18 \Rightarrow -0.107165922 \\ 4 \quad \Rightarrow 0.2729325668 \\ 23 \ \Rightarrow -0.165766645 \\ \text{else} \Rightarrow . \end{pmatrix}
\end{aligned}
$$

Figure 11: Example of prediction expression (JMP®) of average seal strength during processing of PET/PE-EVOH-PE (peel) 12/45, sealed against a PET/PE bottomweb

v. Optimize input parameters

In this example a target value of 0.5 $N.mm^{-1}$ for average and maximum seal strength, a maximization of peel energy, during and after thermal processing is considered as optimal performance. **Desirability functions are set accordingly to obtain input parameters to reach optimal peel performance.** This is shown in Figure 12. The desirability functions are shown in the last column. For seal strength a target value of 0.5 $N.mm^{-1}$ is matched, obtained by a desirability function with a narrow peak at 0.5 $N.mm^{-1}$ and 100 % desirability. Peel energy is maximized, obtained with a linear desirability function. The parameters to reach the optimal performance at a processing temperature of 23 °C are a seal temperature of 143 °C, a seal time of 2.0 s and a seal pressure of 1 $N.mm^{-2}$. The responses during and after processing are predicted in red in the left column. The software can give predictions with the obtained parameters at processing temperatures of -18 and 4 °C. It is also possible to obtain optimal parameters for each individual processing temperature.

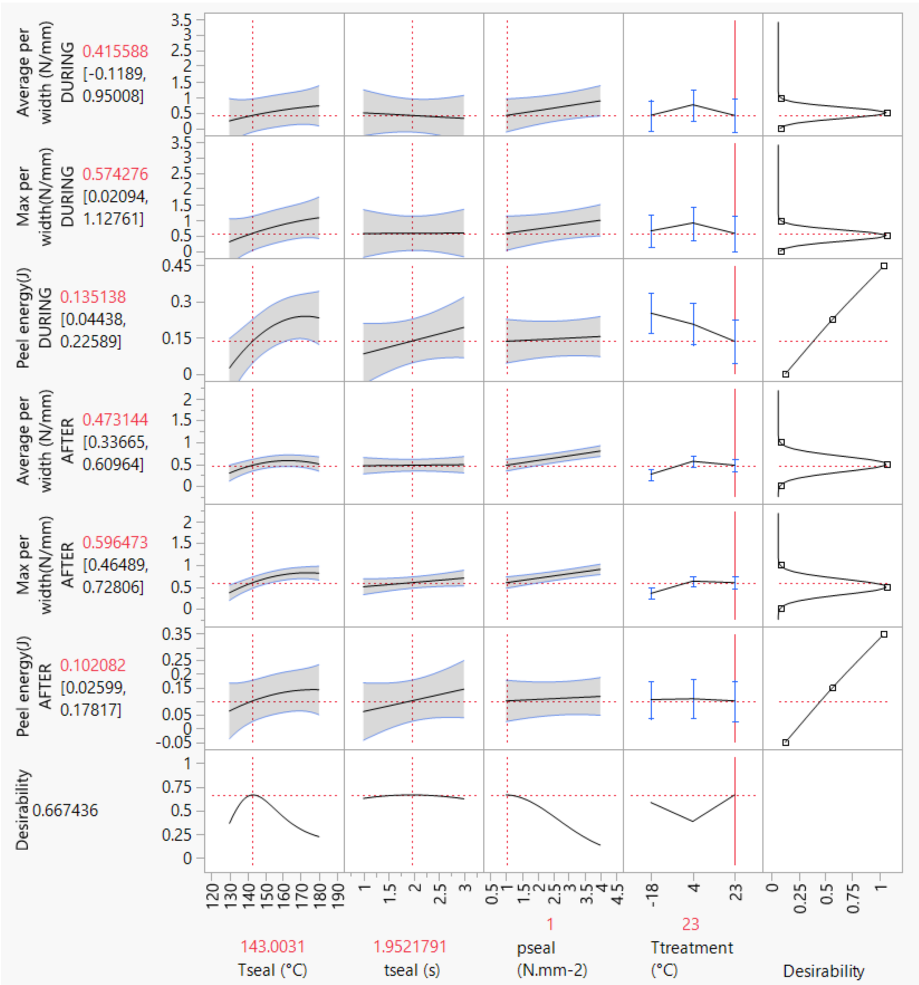

Figure 12: Optimization (JMP®) of peel performance of PET/PE-EVOH-PE (peel) 12/45, sealed against a PET/PE bottomweb

vi. Experimental validation

In a last step **confirmation runs are done at optimal settings to calculate a confidence interval of 95 %**. This will show if the predicted optima are ok. Figure 13 and Figure 14 show the validation results of the maximum seal strength during and after thermal processing. In both figures the predicted values are lower and outside the 95 % confidence interval (= error bars) of the measured values. A higher accuracy can be reached by adding repetitions or by adding extra points to the design. The measured values follow the trend of the predicted values that during cool processing peel strength increases at -18 °C, however, also at 4 °C increased peel strength is measured. Cool processing has no impact on peel strength when seals are heated up to 23 °C. The impact of processing temperature on average seal strength and peel energy is similar. In some cases (during -18 °C and in lesser extent during 4 °C thermal processing), besides cohesive peeling, samples delaminated partially. The increase in seal strength seems to be related with a change in seal separation mode.

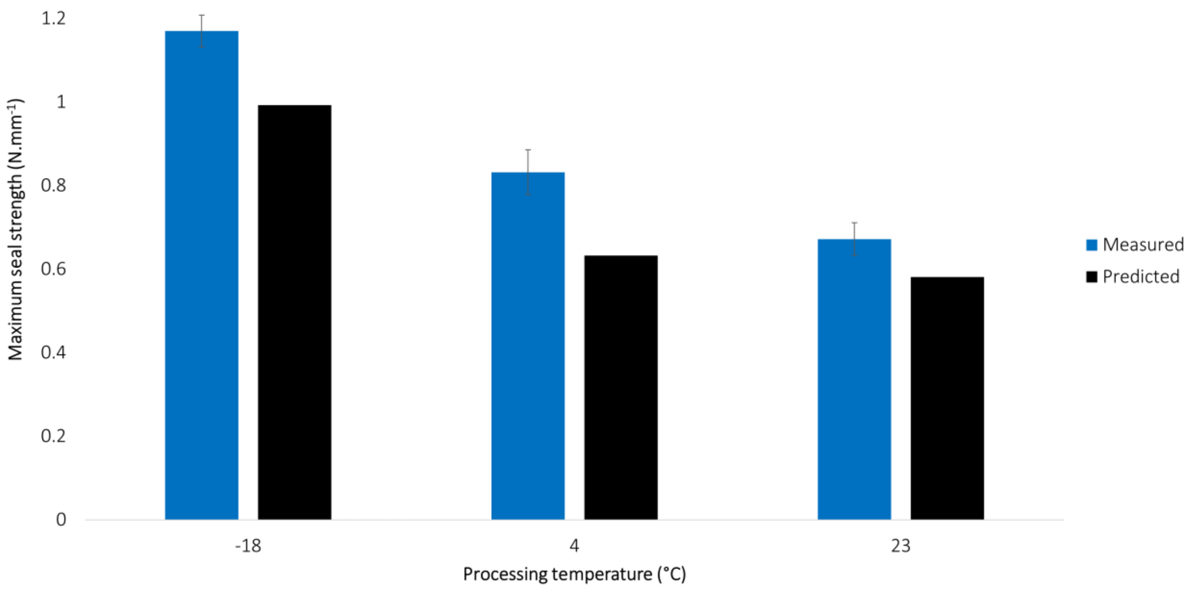

Figure 13: Experimental validation of optimal maximum seal strength <u>during</u> processing of PET/PE-EVOH-PE (peel) 12/45, sealed against a PET/PE bottomweb

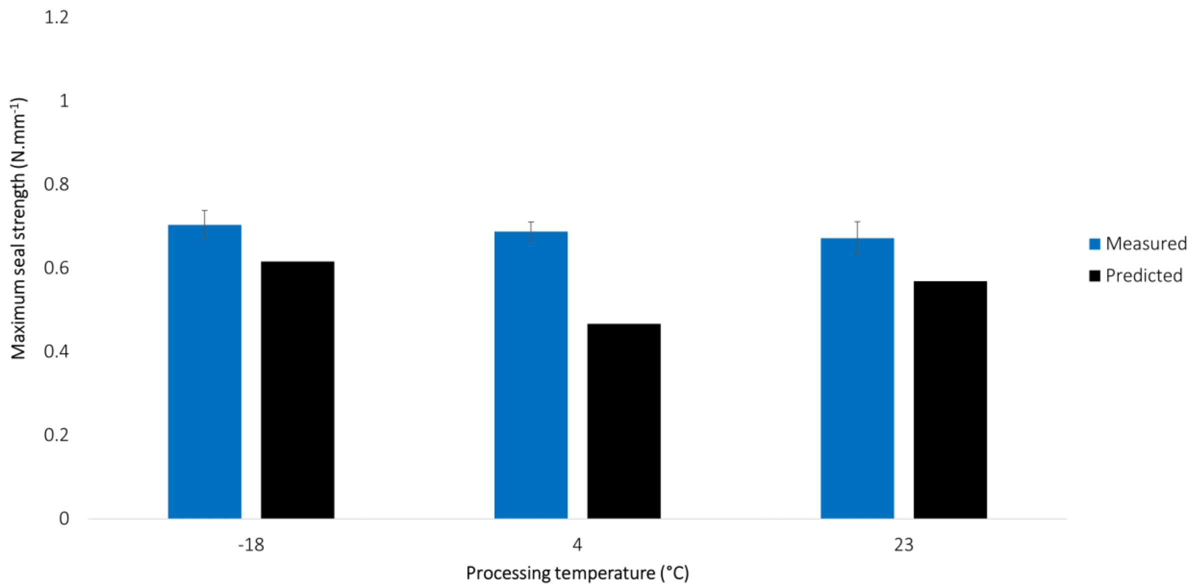

Figure 14: Experimental validation of optimal maximum seal strength after processing of PET/PE-EVOH-PE (peel) 12/45, sealed against a PET/PE bottomweb

3.2 Packaging level

i. Experimental results of burst test

At first, the burst tests enabled the investigation of the influence of the packaging design on the seal integrity during thermal processing. Therefore, different types of tray packaging, different film combinations and sealing parameter settings were considered. The burst pressure is used to evaluate the withstanding of the sealed seal.

1. Failure behaviour dependent on the peel system

For the film combinations with cohesive peel system,

- a PE cohesive peel system(PET/PE-EVOH-PE 12/45 sealed against PET/PE 250/40 non peelable bottomweb) and
- a PP cohesive peel system (PET/OPA/PP 12/15/70 sealed against PA/PP 60/100 bottomweb).

Burst pressure at failure increases with an increasing seal strength as shown in Figure 15. Despite the higher seal strength of the PP cohesive peel system (12 N/15mm compared to 8 N/15mm), the burst pressure is in the same range as the peak rupture pressure of the PE cohesive peel system. A reason for this is possibly the different deformation behaviour of the trays. In contrast to the cohesive peel systems, the burst peel system (PET/PP-EVOH-BurstPP 12/40 sealed against PA/PP 60/100) does not show any significant influence of the seal strength on the burst pressure values.

Regarding the failure pattern, packages with a low burst pressure fail by slowly peeling the sealed seal (Figure 16, left). Packages, failing at a high burst pressure, burst. However, a closer look at the failure pattern reveals differences between the cohesive (Figure 16, middle) and the burst peel system (Figure 16, right). At first, the seal of the cohesive peel system peels cross the seal. When the seal fails the internal pressure is still high. Further tearing of the seal is now much easier because the seal width to be separated is much narrower. Therefore, it seems like the package burst opens along the seal. Packages with a burst peel system generally fail by bursting. The seal also separates longitudinal after the package is opened. In this case, the top film tears completely.

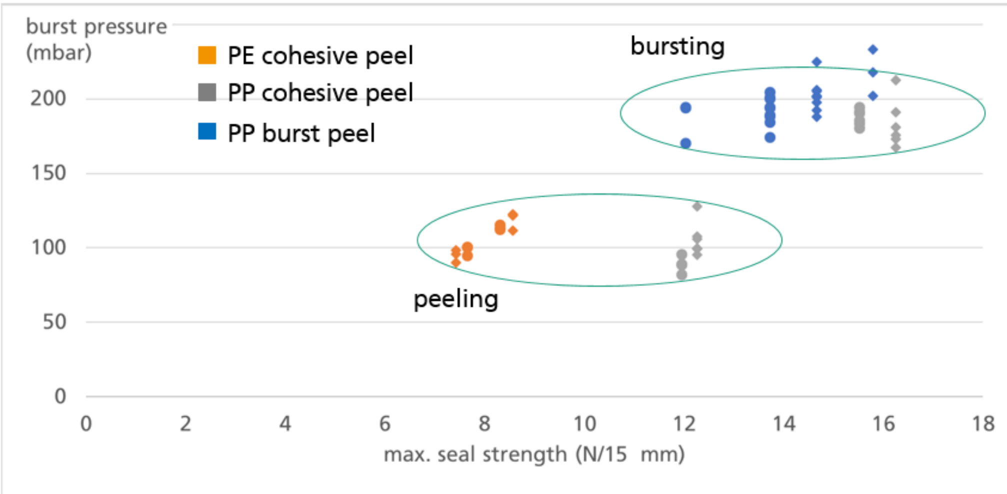

Figure 15: Peak rupture pressure over the seal strength of the package for the three investigated peel systems

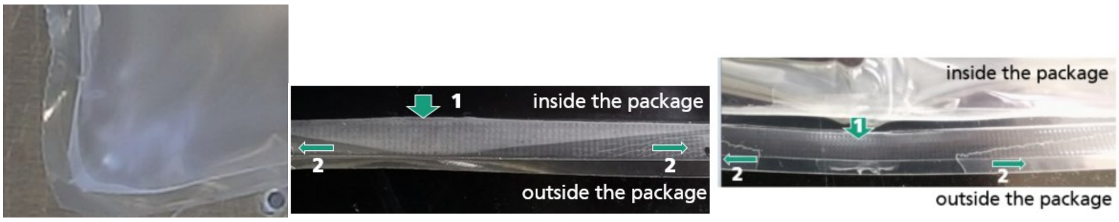

Figure 16: Failure pattern of the sealed seal for the different peel systems and different seal strengths, PP cohesive peel at lower seal strength (left), PP cohesive peel at higher seal strength (middle), PP burst peel (right)

2. Influence of the plate distance on the burst pressure

During thermal processing, it is common for packages to be in a stack or secondary package. This restricts the free deformation of the package. Therefore, burst pressure measurements are conducted with different plate distances.

Figure 17 shows that the burst pressure increases with a decreasing gap size between the plates. The lowest burst pressure is achieved in case of a free deformation of the package. If the seal strength of a package is increased by adjusting the sealing parameters, the burst pressure values also increase. However, this influence decreases at smaller gap sizes.

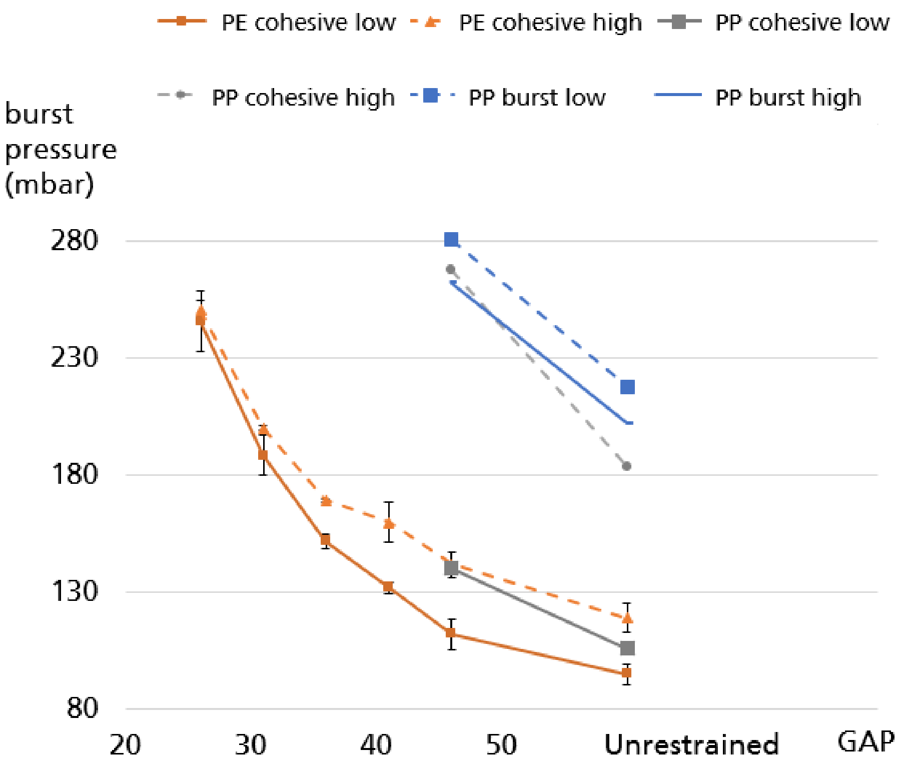

Figure 17: Peak rupture pressure dependent on the gap between the plates for the investigated peel systems

3. Case studies

In the case studies, burst pressure measurements were conducted for packages of different tray geometries and films and therefore, also different PP peel system. At this, no significant influence on the burst pressure was evaluated regarding different heat processes, applied to the packages prior the measurement., and different volumes. It was also shown in the case studies that the burst pressure increases for different PP peel systems with an increase in seal strength.

ii. Results of the finite element simulation

The validation of the simulation model was carried out based on selected results of the experimental burst tests. Therefore, the packaging designs and film concepts of the experimental studies were modelled in the simulation environment. The results of the simulation and experimental studies were compared regarding the deformation of the packages, the occurring failure pattern and the peak rupture pressure.

1. Deformation

Figure 18 and Figure 19 show the deformation of different packages during the experiments in comparison to the simulation results shortly before failure. Looking at the folding up of the edges, good accordance is achieved for both geometries. Due to the idealised material model, the round package folds up more evenly in the simulation than in the experiment.

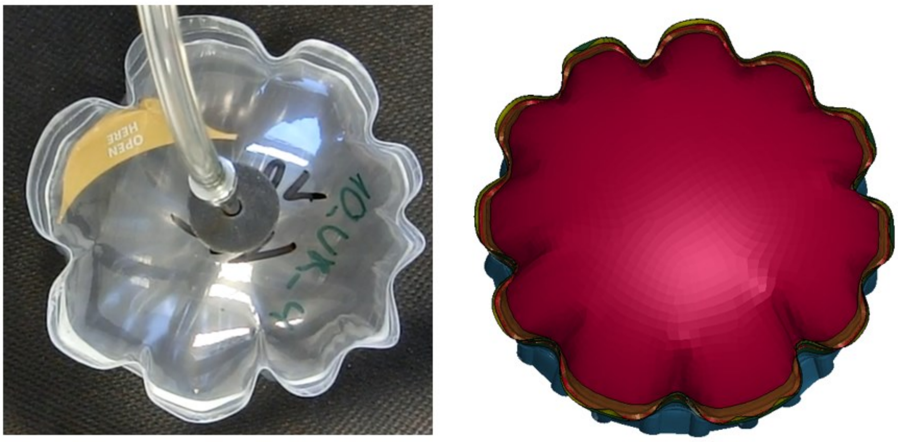

Figure 18: Validation of the simulation results regarding the deformation of a round package with a oPA/PP peel top film sealed against a PP bottomweb at T_{seal} =165 °C

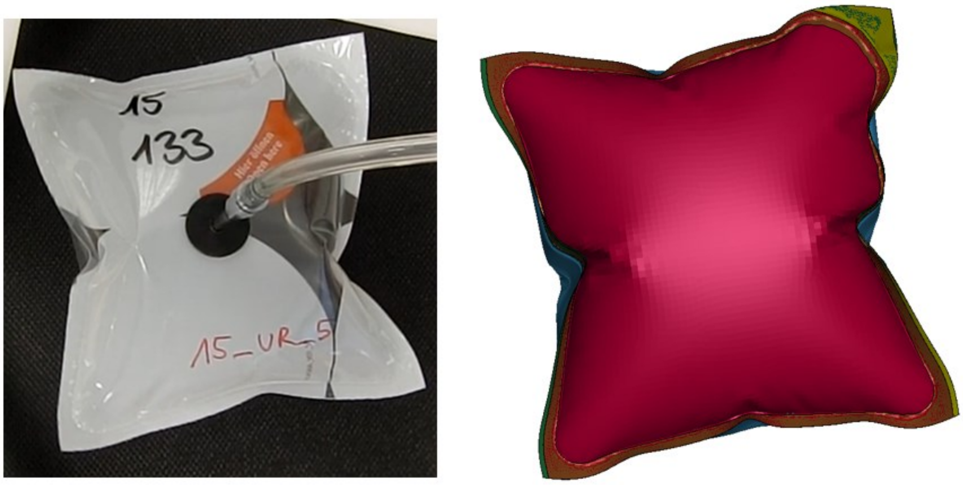

Figure 19: Validation of the simulation results regarding the deformation of a cuboid package with a PET/PP peel top film sealed against a PP bottomweb at T_{seal} =133 °C

2. Failure pattern

Figure 20 and Figure 21 show the failure pattern of different packages during the experiment in comparison to the simulation results. Looking at the round geometry, an equal peeling occurs along the whole circumference of the seal in experiment as well as in the simulation. At the cuboid package, the seal mainly peels at the tear contour, in the corners and along one long edge. A good accordance between experiment and simulation is therefore also achieved with regard to the failure pattern.

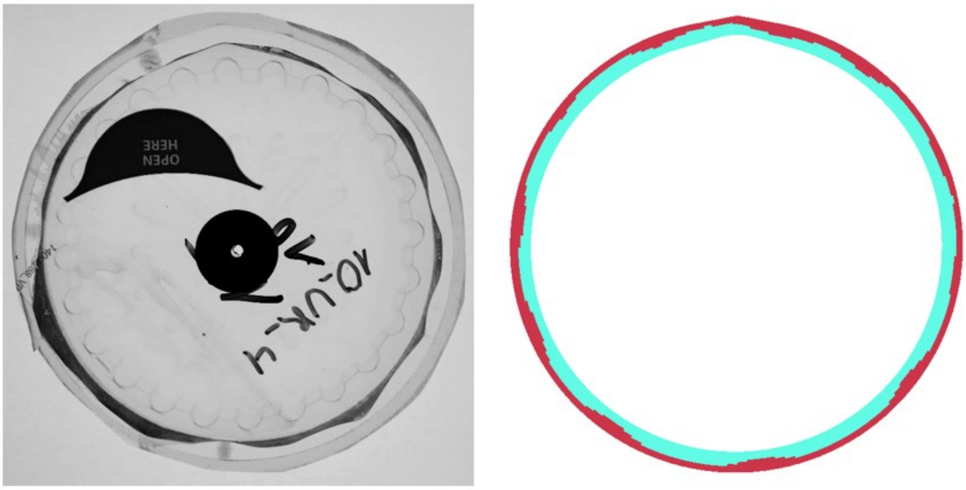

Figure 20: Validation of the simulation results regarding the failure pattern of a round package with a oPA/PP peel top film sealed against a PP bottomweb at T_{seal} =165 °C

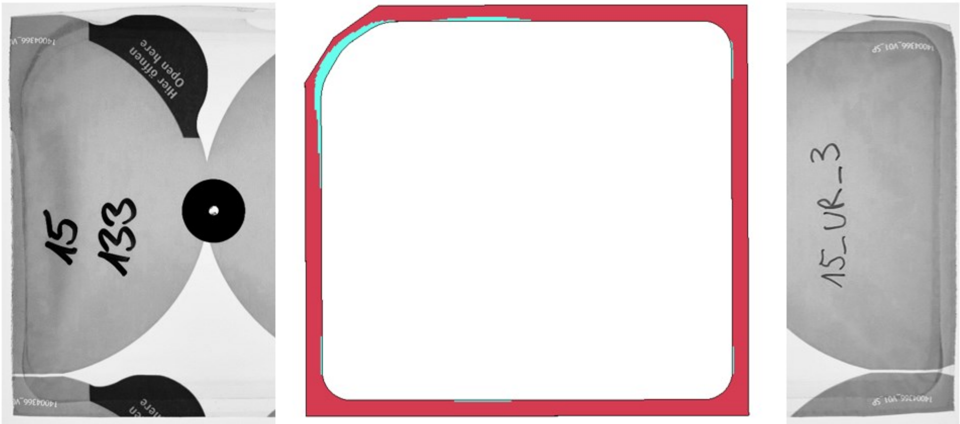

Figure 21: Validation of the simulation results regarding the failure pattern of a cuboid package with a PET/PP peel top film sealed against a PP bottomweb at T_{seal} =133 °C

3. Burst pressure

Comparing the simulation and the experimental results regarding the evaluated burst pressure, as shown in Table 2, a good qualitative accordance can be determined. The burst pressure of the simulation is within the range of the experimental results or slightly below. A comparison of different packages also shows that the experimentally determined tendencies of the packages regarding their resilience against an internal pressure increase correspond to the simulation results. As example, the burst pressure of Package 1 and 2 is almost equal, both, in the experiment and in the simulation, whereas the burst pressures of the packages 3 and 4 are significantly higher. However, a quantitative validation of the simulation model is not feasible due to the highly fluctuating experimental results.

Table 2: Comparison of the peak rupture pressure of different packaging concepts and designs regarding experiment and simulation results

	Package 1	**Package 2**	**Package 3**	**Package 4**
Top film	PET/PP peel	oPA/PP peel	PET/PP peel	oPA/PP peel
Bottomweb	PP	PP	PP	PP
Seal strength	8.3 N/15 mm	7.8 N/15 mm	11.2 N/15 mm	15.0 N/15 mm
Burst pressure experiment	118.4 – 171.9 mbar	115.7 – 132.5 mbar	197.9 – 218.0 mbar	219.0 – 362.1 mbar
Burst pressure simulation	120 mbar	117 mbar	178 mbar	299 mbar

4. Conclusion

The FE simulation is feasible to compare different packaging designs (e.g. trays geometries, tear contours, seal contours) and packaging concepts (e.g. film combinations, seal strength) qualitatively regarding their deformation, failure pattern and burst pressure. This allows drawing conclusions about the resistance of various packages to internal pressure loads due to heat processing.

iii. Results of the opening force calculation tool

The opening force calculation tool was validated by measuring the opening force of packages with a cohesive failure pattern and comparing it to the results calculated within the tool as shown in Figure 22. For this purpose, the opening force was calculated on the basis of the seal contour of the technical drawing of the sealing tool. The results already show a good agreement with the experiments. However, the calculated tear-on force is slightly lower, the peel force slightly higher than the measured values. These deviations are due to a deviation of the real seal contour of the experimentally measured package from the contour of the sealing tool. By calculating the opening force based on the real imprint of the seal, a high calculation accuracy of the tool is shown for films with a cohesive failure pattern.

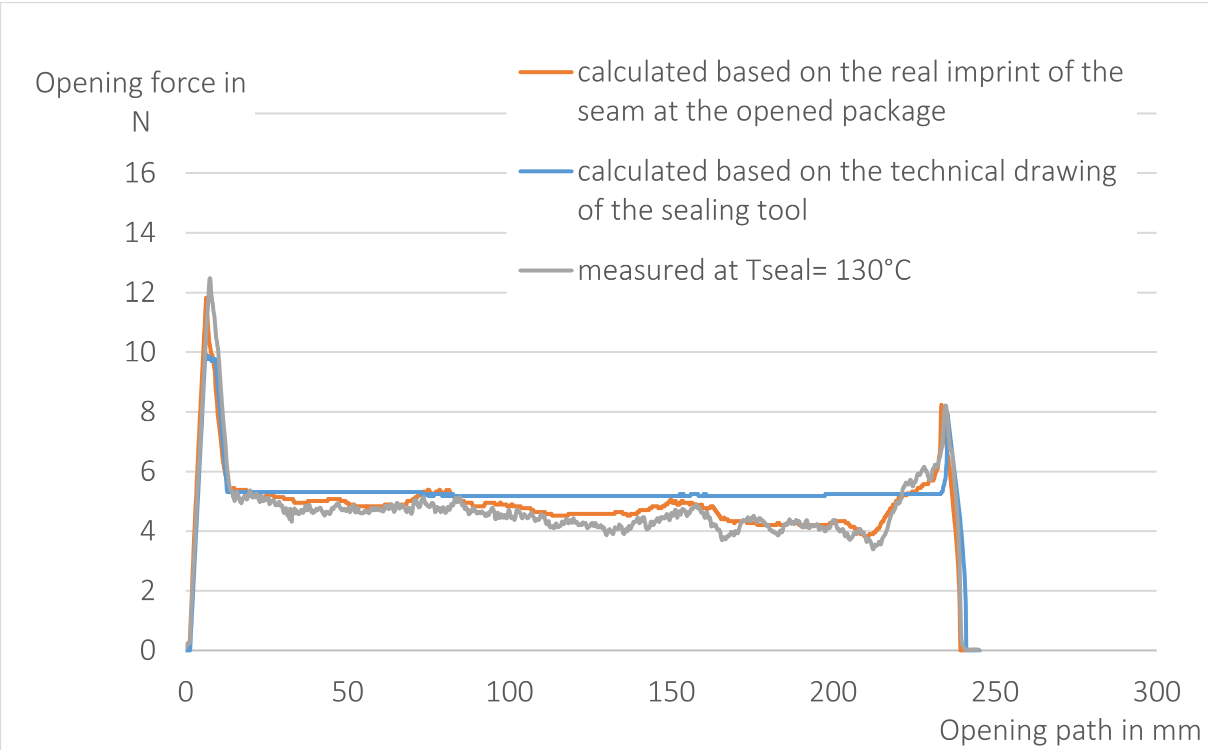

Figure 22: Comparison of measured and calculated opening force curves of a package

3.3 Summary

The three aforementioned steps were developed as a basis to support the design process of peelable packages undergoing thermal processing:

- The design of experiment approach optimizes the sealing and thermal processing parameters on sample level.
- The FE simulation investigates the load applied to a package due to an internal pressure increase caused by thermal processing.
- The opening force calculation tool predetermines the expected opening force along the opening path of a package.

The guideline in Figure 23 describes, how the three steps can be applied together to optimize the package properties and the sealing process. At first, a FE simulation model of the package is used to compare the load capacity of different package designs against an increasing internal pressure. Possible variable input parameters are for example the tray geometry and material, the tear contour and contour of the seal or the seal strength. On the other hand, the opening force calculation tool is used to evaluate the expected opening force of the chosen seal contour and packaging concept. The expected opening force can be compared to the reference values for easy-opening (IVLV-;Merkblatt 106). The application of the FE simulation and the opening force calculation tool can be both, iteratively as well as parallel. In the last step, a target value for the seal strength on sample level is defined based on the results of the pre-calculation of the opening force. In order to achieve this target seal strength, the sealing and processing parameters are optimized with the help of the design of experiment approach.

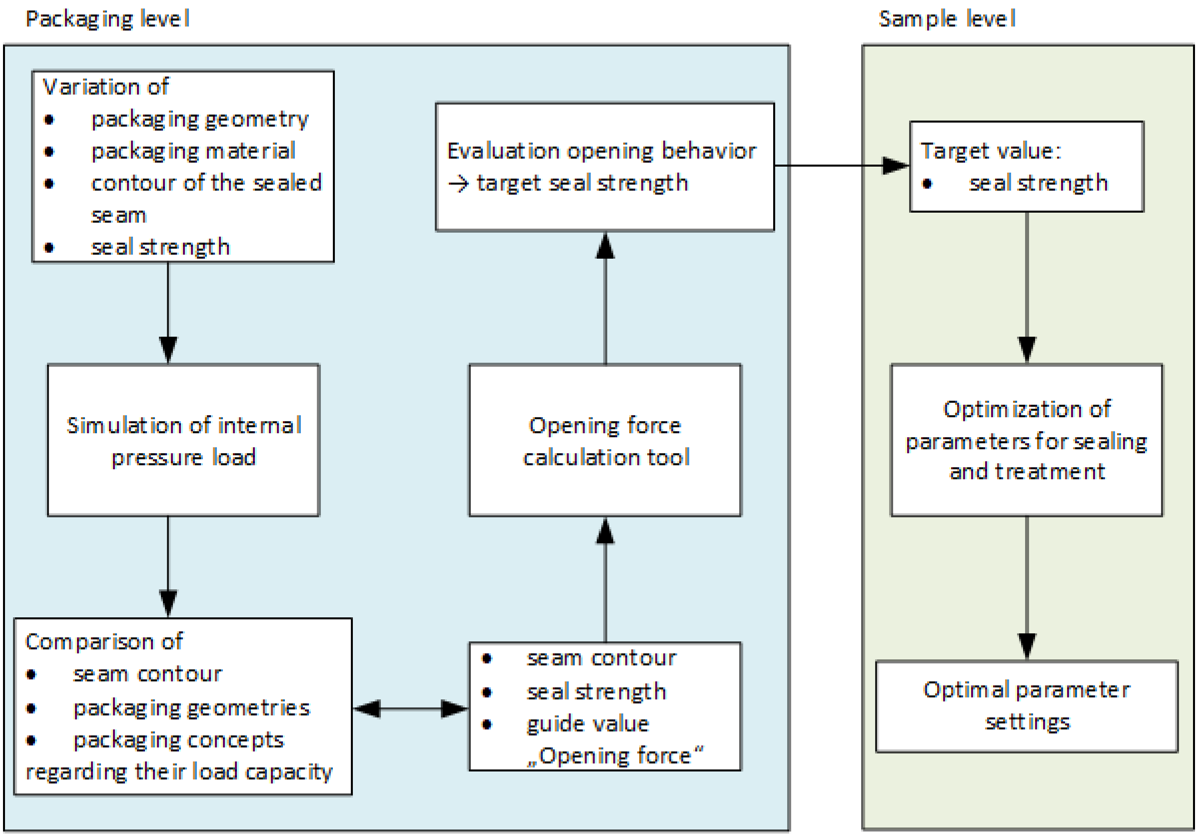

Figure 23: Guideline to evaluate and optimize peelable packaging concepts undergoing thermal processing

4 Contact

If you have questions about this guide, seal performance and/or packaging in general you can contact a member of the ThermoPeel-consortium.

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