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Evaluation and optimization of the peel performance of a heat sealed topfilm and bottomweb undergoing cool processing

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Abstract

Easy opening of heat sealable tray and topfilm food packaging concepts is often realized by using a peelable seal layer. Peel strength needs to be strong enough to prevent the package from opening during and after storage, transportation and handling at different environmental temperatures. It also needs to be weak enough to be convenient for an ageing population. When designing packaging concepts, a balance needs to be found assuring food quality, safety and convenience. This study presents a method to evaluate and optimize peel performance of a packaging concept with polyethylene seal layer undergoing cool processing at −18°C and 4°C. A design of experiment approach is used as a basis. With a limited amount of tests, models are fitted and experimentally validated to obtain an optimal peel strength of 0.5 N mm^{-1} at an environmental temperature of 23°C. When measured during thermal treatment at −18°C and 4°C, the peel strength increased but no effect was seen once the seals are tested at 23°C after treatment. Bending of the bottomweb during the peel test slightly decreased peel strength while bending stiffness increased at low temperatures, suggesting a minor impact of bending of the bottomweb related to environmental temperature on the observed peel performance. Increased peel strengths during cool processing were clearly related to a change in seal failure mechanism. Besides cohesive peeling, partial delamination occurred in these stronger seals.

KEYWORDS
cool processing, food packaging, heat seal, peel performance, polyethylene

1 | INTRODUCTION

Tight packages are crucial to ensure food quality and food safety throughout the process chain. Perishable food products such as meat, cheese, ready meals and others are often packed in a rigid thermoformed tray, heat sealed with a thin flexible topfilm, in vacuum or with a modified atmosphere to extend the shelf life. In 90% of all thermoform fill and seal machines, only the bottomweb is formed. Besides heat sealability, materials are selected based on the barrier and mechanical properties.[1] These properties are determined by the chemical composition, production process and the thickness. 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°C to −18°C where the presence of H_2O in a solid state extends the shelf life. Cool processing generally

extends the shelf life by decreasing microbial activity and biochemical reactions.[2] In the last decades, the tray and topfilm concept is expected by consumers to open by peeling off the topfilm at a low peel strength. To meet the needs of the rapidly growing segment in the population of those aged 65+ with reduced muscle strength, increasingly living in single-person households, packaging solutions with easy opening features and smaller size are suggested.[3] Industrial guidelines[4] and research[5] are published to address the suggestion of easy opening of thermoformed trays with peelable seals for this segment of the population. Seal quality must be ensured at all temperatures of the process chain. For cool processing, no study is available on the peel performance (peel strength and peel energy) of heat sealable tray and topfilm materials. Test procedures to efficiently evaluate and optimize the peel performance of packaging materials before, during and after cool processing are missing. As a result, insight into this matter is somewhat limited.

2 | OBJECTIVES

The main objective of this study is to present a method to optimize the peel performance of packaging concepts undergoing cool processing. This method is based on previous studies with a similar methodology to optimize seal strength with a limited amount of tests.[6,7] A second objective is to evaluate the relation between peel performance and cool processing by applying the proposed method on a commercial packaging concept with polyethylene (=PE) seal layer.

3 | MATERIALS AND METHODS

3.1 | Materials

The topfilm is composed of a blown coextruded structure of 45 μm with three layers (PE, ethylene vinyl alcohol [=EVOH] and cohesive peelable PE at the seal surface), laminated to a 12-μm-thick polyethylene terephthalate (=PET) outer layer. The bottomweb is composed of a PE seal layer of 35 μm laminated to an outer PET layer of 250 μm. These materials were provided by Südpack Verpackungen GmbH & Co KG (Germany). The bottomwebs are not thermoformed and characterized as films to eliminate the impact of the thermoform process.

3.2 | Methods

Previous studies on fracture mechanics have shown that peel energy results of experimental tests is the sum of the energy of creating new interfacial area, which is referred to as the energy of fracture (Ga), the energy to extend the peel arm (Ge) and the energy to bend the peel arm (Gb).[8,9] The following formula for peel strength illustrates this sum of impacting components and considers the geometry of the test by including the peel angle θ. $Peel\ Strength\ \left(\frac{N}{mm}\right) = \frac{Ga + Ge + Gb}{1 + \varepsilon a - cos\theta.}$

ε_a represents the inelastic extension.[10]

Besides presenting an optimization method, this study evaluates the relation of cool processing on peel performance by applying the proposed method and performing additional mechanical tests for seal and film characterization.

3.2.1 | Seal preparation and characterization

Samples are cut to a width of 30 mm and a length of 100 mm in the machine direction of the film. The seal width, this is the width of the jaws, is 10 mm. The upper jaw is heated at high temperatures, while the lower jaw is kept at 50°C to simulate the sealing process in the industry where the lower jaw is not actively heated by itself, but only through the frequent touching of the heated upper jaw. Seal temperature in this study refers to the temperature of the upper jaw. Seal times from 1 to 3 s are used to simulate the sealing process of the topfilm and tray packaging concept in the industry. Seal pressures from 1 to 4 N mm^{-2} are used to cover the full working range of the lab sealer. A peel strength test with a peel angle of 180° is performed within 4 hr after sealing. In order to do so, the bottomweb is clamped at the bottom and the topfilm from above. Clamp distance is set at 20 mm, and testing speed is 300 mm min^{-1}. A preload force of 1 N is used.

Three results characterize the peel performance. The maximum peel strength is calculated by dividing the maximum force measure by the sample width. The average peel strength is calculated by dividing the average force of the central 30% of the position of the peel curve with the sample width. Peel energy is the energy below the force–elongation curve. Samples are visually analyzed afterwards to study the impact of the peel test and temperature treatment on peeled multilayer structures. Discussed seal failure mechanisms of ASTM F88 such as cohesive peel and delamination, as shown in Figure 1, are differentiated amongst combinations of these mechanisms by eye. Microscopic cross section of peeled samples with amplification of 10 × 20 and 10 × 50 is made to visualize the layer distribution.

In order to determine the cooling time of the experiments to optimize peel performance, the following test is carried out. Samples are sealed with a seal temperature of 150°C, a seal time of 0.7 s and a seal pressure of 1.0 N mm^{-2}. Using these seal settings, samples peeled cohesively in a peel test. Directly after sealing, samples are transferred to temperature chambers of −18°C, 4°C or 23°C, and samples are tested in triplicate after 30 min, 1 hr, 2 hr, 4 hr, 6 hr, 1 day, 2 days, 4 days, 8 days, 11 days, 1 month and 2 months. Five minutes before testing the samples are kept at 23°C, and the peel test is also carried out at 23°C to measure the influence of processing time on maximum peel strengths and standard deviations.

3.2.2 | Film characterization

All materials are stored in a room with standard environment conditions (23°C, 50% relative humidity) 8 days before testing.

A three-point flexural test is performed on bottomweb samples to determine the impact of environmental temperature on bending

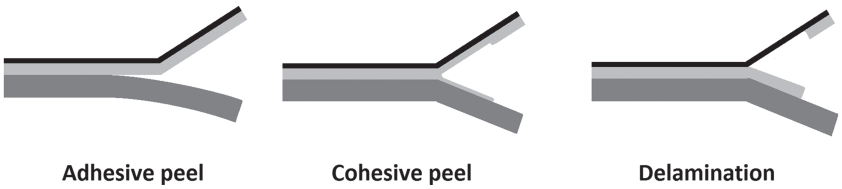

FIGURE 1 Seal separation modes of topfilm, with outer layer (black) and seal layer (light grey), and bottomweb (dark grey)

properties. The bottomweb sample is cut to a width of 30 mm and a length of 50 mm in machine direction. In this direction, the sample is naturally slightly bended because of the winding on a roll with 76-mm core diameter. The sample is placed on two supports, with the bend facing upwards, in a temperature chamber of −18°C, 4°C or 23°C. The length of the span between these supports is 20 mm. The radii of the supports and loading edge are 5 mm. The position, and thus resulting strain, is zeroed at a preload force of 0.3 N, corresponding closely with a straight parallel sample at considered temperatures. The testing speed is set at 1 mm min^{-1}, and a comparison is made of the flexural stress (σ_f)–strain (ε_f) curves until 2% strain. Flexural stress and strain are calculated according to the ISO 178 standard with the formulas (Equations 1 and 2) shown below.

$$\sigma_f = \frac{3FL}{2bh2} \quad (1)$$

and

$$\varepsilon_f = \frac{600sh}{L2}\% \quad (2)$$

F is the applied force (N), L is the span (mm), b is the width (mm), h is the thickness (mm) and s is the deflection (mm).

A tensile test on the topfilm is done to determine its tensile properties. The 15-mm-wide rectangular topfilm samples are tested in machine direction at 300 mm min^{-1} and a clamp distance of 20 mm to match the settings of the peel strength test.

As the topfilm material is a commercial material and the composition of the seal layer remains unknown, additional tensile tests were performed on low density PE (LDPE) film samples to visualize how cool processing impacts a PE stress–strain diagram at the test temperatures, cool time and test speed in this study.

3.2.3 | Seal optimization

To evaluate the impact of the individual parameters seal temperature, seal time, seal pressure, treatment temperature and their interactions on the peel performance (peel strength and peel energy) a design of experiment approach was followed according to previous research.[6,7]

- In a first step, a design space is defined using predefined limits of all individual parameters. The limits are based on preliminary tests, industrial relevance and the working range of the equipment. In this study, the minimum and maximum design limits for continuous parameters such as seal temperature, seal time and seal pressure are respectively 130–180°C, 1.0–3.0 s and 1.0–4.0 N mm^{-2}. Processing temperature is considered a categorical parameter because there is no interest in intermediate temperatures.
- In a second step, an experimental design is defined within the design space. The combination of continuous and categorical parameters requires a custom design. An I-optimal design with 24 experimental runs is proposed.[11]
- Each of the runs is tested in duplicate. Additionally, samples *during and after* cool processing are tested, summing up four samples for each run. Each sample generates three results: maximum and average peel strength, and peel energy.
- In a next step, a response surface model is fitted to the obtained data. Factors were mean centred before calculating interactions or quadratic terms.
- This model is then used to optimize settings to obtain certain target values for peel performance at 23°C and to predict values at −18°C, 4°C and 23°C during and after cool processing. The optimized peel performance is based on the capacity of the packaging concept and on target values that can be achieved by 95% of the population.[4]
- In a last step, the optimized seals are validated by testing five samples, sealed at optimum settings. For more detail on this methodology, the reader is referred to a previous study.[7]

The influence of bending movement of the bottomweb on the peel performance is evaluated by comparing the peel performance of optimized seals at 23°C with seals that are reinforced by gluing the bottomweb to a 1-mm-thick metal plate. The influence of processing temperature on bond strength and elongation is evaluated by testing the optimum seals with a reinforced metal plate during −18°C, 4°C and 23°C and thus eliminating the difference in bending stiffness of the bottomweb at different temperatures.

3.2.4 | Statistical analysis

Results from the higher mentioned experiment were analyzed using a response surface model, considering main effect, interactions and quadratic effects. An all-possible subset model selection was performed to define the final model that was used for the optimization. For all analyses, the JMP version 14 software (JMP 14, The SAS institute, Inc, NC, USA).

3.2.5 | Apparatus

Sealed samples are prepared with a Labthink HST-H3 heat seal tester (Labthink Instruments Co Ltd, People's Republic of China). Peel and

flexural tests are carried out with the Tinius Olsen 5ST universal testing machine (Tinius Olsen Ltd, United Kingdom), the tools and clamps are inside a TH 2700 temperature chamber (Thümler GmbH, Germany). The combination of both instruments is installed by Benelux Scientific BVBA (Belgium). A Nikon Eclipse ME600 microscope and NIS-Elements D4.10.00 software (Nikon, Japan) are used to visualize cross sections.

4 | RESULTS AND DISCUSSION

4.1 | Influence of cooling time

Figure 2 shows the maximum peel strength results at different cooling times. There is a very small impact of cooling time on maximum peel strength, the average values increase slightly after one day of cooling. However, the increase of average values lies within a 95% confidence interval (shown by the error bars) of the maximum peel strengths at low cooling times.

Because of this limited impact and to be able to perform many tests in a short amount of time, the following ageing and cooling time restrictions are followed in the optimization experiments: Sealed samples are tested in a 4-hr timeframe after sealing in the optimization tests. Samples *during* sealing are kept in the temperature chamber for 15 min prior to the start of the test. Samples *after* sealing are also kept in the temperature chamber but transferred after 15 min to cool down or heat up to 23°C. These samples are eventually tested at 23°C in the temperature chamber.

4.2 | Seal optimization

The experimental design with results is shown in Table 1.

Table 2 shows a summary of the coefficients of the terms which are included in the models for each response. Parameters estimates for nonsignificant terms are not shown in the table because they are not retained in the models. The table shows the complexity of parameters (first order, second order and interactions) that impact the results for peel strength and peel energy. As an example, the polynomial model for maximum peel strength during thermal processing is given, factors are mean centred:

$$\begin{aligned}\textit{Maximum peel strength} = {} & -4.598 + 0.028 * \textit{Tseal} + 0.468 * \textit{tseal} + 0.244 \\ & * \textit{pseal} + \textit{Match Tprocessing}\,[-18 \rightarrow 0.082; 4 \rightarrow 0.042; 23 \rightarrow -0.124] + 0.009 \\ & * \textit{Tseal} * \textit{tseal} - 0.212 * \textit{tseal}^2 + 0.009 * \textit{Tseal} \\ & * \textit{pseal} + 0.124.\textit{tseal}.\textit{pseal} + 0.032 * \textit{pseal}^2 + \textit{Tseal} \\ & * \textit{Match Tprocessing}\,[-18 \rightarrow 0.008; 4 \rightarrow -0.007; 23 \rightarrow -0.002] + \textit{tseal} \\ & * \textit{Match Tprocessing}\,[-18 \rightarrow -0.044; 4 \rightarrow 0.118; 23 \rightarrow -0.074] + \textit{pseal} \\ & * \textit{Match Tprocessing}\,[-18 \rightarrow -0.014; 4 \rightarrow -0.129; 23 \rightarrow 0.143]\end{aligned}$$

Using these models, seal settings are optimized. Based on a maximum peel line of 22 mm for a thermoform-fill-seal machine and a minimum opening force that can be achieved by 95% of elderly female population[4], and considering the potential peel strength of the packaging concept at 23°C as shown in Table 1, average and maximum peel strengths of 0.5 N mm^{-1} are considered as optimal. Target values of 0.5 N mm^{-1} are matched for average and maximum peel strength during and after cool processing, and peel energy is maximized using linear desirability functions to optimize peel performance. It is shown in Table 1 that the target peel strength is achievable with the considered packaging concept. The maximization of peel energy is chosen to generate a peelable seal that maintains this strength over the full length of the sealed surface.

The optimal settings to match the target values at 23°C during and after cool processing are given by a seal temperature of 170°C, a seal time of 1.0 s and a seal pressure of 2.0 N mm^{-2}. The predicted values for peel strength of the optimal sealed samples, prepared with these settings at processing temperatures of −18°C, 4°C and 23°C, are compared with confidence intervals based on validation experiments, corresponding with the CICon approach.[12,13] The results are shown in Table 3. The predicted values are a good indication of what can be expected; however, these values are slightly underestimated. A higher accuracy can be reached by adding repetitions or by adding extra points to the design. Even when both responses average and maximum peel strength is matched to an equal value of 0.5 the maximum value is slightly higher than the average value, another outcome

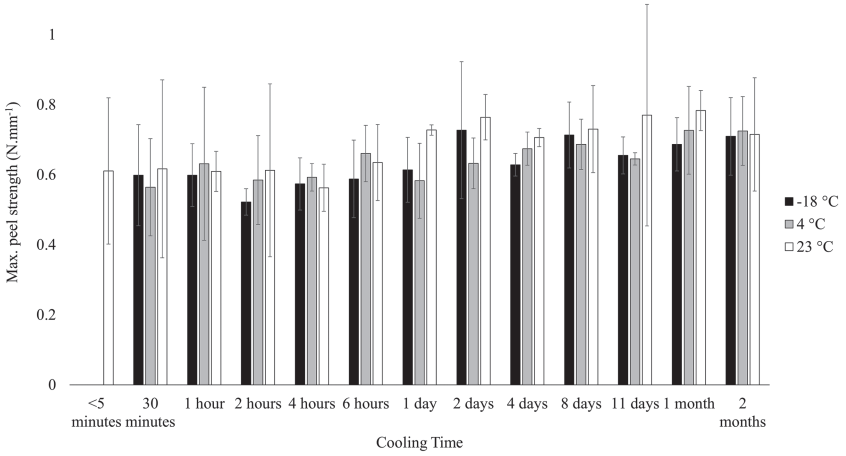

FIGURE 2 Influence of cooling time on maximum peel strength (n = 3)

TABLE 1 Experimental design with parameters (seal temperature, seal time, seal pressure and processing temperature) and responses (average peel strength, maximum peel strength and peel energy) during and after cool processing (n = 2)

					During cool processing			After cool processing		
	T_{seal} (°C)	t_{seal} (s)	p_{seal} (N mm^{-2})	$T_{processing}$ (°C)	Average peel strength (N mm^{-1})	Maximum peel strength (N mm^{-1})	Peel energy (J)	Average peel strength (N mm^{-1})	Maximum peel strength (N mm^{-1})	Peel energy (J)
1	155	2.0	2.5	−18	1.1	1.2	0.34	0.8	0.8	0.20
					1.1	1.2	0.33	0.7	0.8	0.18
2	180	1.0	4.0	4	1.0	1.0	0.26	0.7	0.7	0.17
					1.0	1.0	0.27	0.8	0.8	0.20
3	155	1.0	1.0	4	0.8	1.0	0.21	0.2	0.3	0.02
					0.3	0.7	0.10	0.6	0.7	0.13
4	180	3.0	1.0	23	0.7	1.3	0.23	0.7	1.3	0.23
					0.7	0.9	0.21	0.7	0.9	0.21
5	155	2.0	2.5	23	0.7	0.8	0.18	0.7	0.8	0.18
					0.7	0.7	0.19	0.7	0.7	0.19
6	130	3.0	4.0	−18	0.7	1.0	0.21	0.4	0.6	0.08
					0.5	0.7	0.14	0.5	0.6	0.12
7	180	2.0	1.0	4	0.8	1.0	0.28	0.8	0.8	0.23
					1.1	1.1	0.29	0.8	0.9	0.24
8	180	3.0	2.5	4	2.6	2.7	0.24	2.0	2.1	0.29
					3.1	3.4	0.54	2.3	2.4	0.44
9	155	3.0	4.0	23	1.7	1.8	0.14	1.7	1.8	0.14
					1.7	2.0	0.11	1.7	2.0	0.11
10	130	1.0	4.0	23	0.1	0.2	0.02	0.1	0.2	0.02
					0.0	0.1	0.00	0.0	0.1	0.00
11	130	3.0	2.5	23	0.5	0.6	0.13	0.5	0.6	0.13
					0.5	0.6	0.13	0.5	0.6	0.13
12	130	3.0	1.0	4	1.0	1.0	0.21	0.7	0.7	0.13
					0.2	0.6	0.06	0.6	0.7	0.10
13	130	1.0	1.0	−18	0.0	0.1	0.01	0.0	0.0	0.00
					0.0	0.1	0.01	0.0	0.0	0.00
14	180	1.0	1.0	−18	1.1	1.3	0.42	0.4	0.6	0.10
					0.6	1.1	0.18	0.6	0.7	0.16
15	130	1.0	2.5	4	0.1	0.3	0.02	0.0	0.0	0.00
					0.2	0.3	0.03	0.0	0.1	0.01
16	155	3.0	1.0	−18	0.9	1.3	0.39	0.7	0.7	0.17
					1.2	1.3	0.35	0.8	0.8	0.20
17	155	1.0	4.0	−18	1.0	1.2	0.30	0.5	0.6	0.14
					0.8	1.0	0.21	0.4	0.6	0.12
18	160	1.0	2.5	23	0.7	0.7	0.19	0.7	0.7	0.19
					0.7	0.7	0.17	0.7	0.7	0.17
19	139	1.3	1.0	23	0.4	0.6	0.07	0.4	0.6	0.07
					0.4	0.6	0.09	0.4	0.6	0.09
20	155	2.0	2.5	−18	1.1	1.3	0.44	0.7	0.7	0.18
					1.2	1.2	0.31	0.7	0.7	0.17
21	180	2.0	4.0	23	3.3	3.3	0.38	1.3	1.4	0.05
					3.3	3.3	0.48	2.2	2.3	0.11

(Continues)

TABLE 1 (Continued)

					During cool processing			After cool processing		
	T_{seal} (°C)	t_{seal} (s)	p_{seal} (N mm^{-2})	$T_{processing}$ (°C)	Average peel strength (N mm^{-1})	Maximum peel strength (N mm^{-1})	Peel energy (J)	Average peel strength (N mm^{-1})	Maximum peel strength (N mm^{-1})	Peel energy (J)
22	180	3.0	4.0	-18	3.3	3.3	0.38	2.2	2.2	0.33
					3.3	3.3	0.48	1.8	2.1	0.15
23	155	3.0	4.0	4	2.5	2.7	0.38	2.1	2.1	0.27
					0.9	0.9	0.25	0.7	1.2	0.21
24	130	2.0	4.0	4	0.8	0.8	0.12	0.4	0.6	0.08
					0.8	0.9	0.13	0.4	0.5	0.09

would not make sense. The calculated confidence intervals 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.

4.3 | Film characterization

The results of film characterization are shown below in Figures 3–5. These results are discussed in relation with peel performance in Section 4.4.

Figure 3 shows the flexural stress–strain curves of five bottomweb samples for each evaluated environmental temperature. The samples at low temperature (−18°C and 4°C) reach higher stress values when strain increases, compared to samples at standard temperature. The samples at −18°C tend to have the highest stress; however, variation is too high to distinct clearly with the samples at 4°C. A flexural stress of 70 N mm^{-2} corresponds with normalized strength value of 0.2 N mm^{-1}.

Figure 4 shows tensile stress–strain curves of the topfilm at −18°C, 4°C and 23°C. At low temperature, the elongation decreases, whereas the yield and peak strength increase. Stress values of 40 and 60 N mm^{-2} correspond, respectively, to normalized strength values of 2.5 and 3.7 N mm^{-1}. The average values (not shown) of yield stress at −18°C, 4°C and 23°C are statistically different at a 95% confidence level.

Figure 5 shows stress–strain curves during tensile tests of 60-μm-thick standard LDPE blown monolayer film. Increase of yield and peak stresses is observed at low temperature, comparable with the effects illustrated in Figure 4. Stress values of monolayer PE film are lower, and strain values are higher in comparison with multilayer film. This is caused by the presence of a thin PET outer layer in the multilayer topfilm. Stress values of 20 and 30 N mm^{-2} correspond, respectively, with normalized strength values of 1.2 and 1.8 N mm^{-1}. The average values (not shown) of yield and peak stress at −18°C, 4°C and 23°C are statistically different at a 95% confidence level.

4.4 | Evaluation of peel performance *during* cool processing

This section discusses the impact of temperature on peel strength and peel energy during cool processing.

Figure 6 shows bending movements of the sealed bottomweb that occurs during the peel test. Once a pulling load is exerted on the seal, and peeling initiates, the sealed bottomweb will slightly bend. The bottomweb straightens when the seal is peeled towards the end of the seal. In a previous study on peel films of LDPE, with minor contents of isotactic polybutene-1, bending force and bending energy was neglected because the values were 200 and 100 times smaller as peel force and peel energy.[9] In the flexural test of this study, flexural stress reached values up to 75 N mm^{-2} around 2% flexural strain, corresponding with respective normalized strength values of 0.2 N mm^{-1} at 6 mm. Although different test protocols were used, these values indicate a higher proportion of bending force to peel force, which reaches around 0.5–1.2 N mm^{-1} in Table 3 as maximum peel strength. This was expected as the bottomweb is a more rigid material because of the presence of a thick PET outer layer of 250 μm.

The bending of the bottomweb causes a change in peel angle during the test. If the bottomweb is fixed, a peel angle of 0° would be assumed for the bottomweb and 180° for the topfilm. The bottomweb is not fixed in the peel test of this study causing a change in peel angle partitioning over bottomweb and topfilm during the peel test.

Root rotation, which is described in a previous study,[8] is another important factor that could impact the peel performance. The angle of root rotation (θ_0) is dependent on the peel angle (θ), with values between 0° and θ. The applied peel energy will be partitioned between the part that bends the peel arms and the part that creates new interfacial area. The previous study also showed the dependency of θ_0 with yield stress. As this material property increases typically at decreased temperature, it is likely that decreasing processing temperature will increase θ_0 and more in general impact the peel performance during cool processing.

TABLE 2 Significant terms with parameter estimates (*V*) and corresponding *p* values (*p*–*V*) of average peel strength, maximum peel strength and peel energy during and after cool processing

	During cool processing						After cool processing					
	Average peel strength ($N\ mm^{-1}$)		Maximum peel strength ($N\ mm^{-1}$)		Peel energy (J)		Average peel strength ($N\ mm^{-1}$)		Maximum peel strength ($N\ mm^{-1}$)		Peel energy (J)	
Term	V	*p*–*V*	V	*p*–*V*	V	*p*–*V*	V	*p*–*V*	V	*p*–*V*	V	*p*–*V*
Intercept	−4.7550	<0.0001	−4.5976	<0.0001	−0.5638	<0.0001	−3.0738	<0.0001	−3.2361	<0.0001	−0.3324	<0.0001
T_{seal}	0.02793	<0.0001	0.0278	<0.0001	0.0046	<0.0001	0.0177	<0.0001	0.0186	<0.0001	0.0027	<0.0001
t_{seal}	0.45050	<0.0001	0.4676	<0.0001	0.0531	0.0006	0.3789	<0.0001	0.4118	<0.0001	0.0493	<0.0001
p_{seal}	0.28074	<0.0001	0.2438	<0.0001	–	–	0.1485	<0.0001	0.1509	<0.0001	–	–
$T_{processing\ [-18]}$	–	–	–	–	0.0674	0.0003	–	–	−0.1083	0.0400	–	–
$T_{processing\ [4]}$	–	–	–	–	–	–	–	–	–	–	0.0211	0.0163
$T_{processing\ [23]}$	–	–	–	–	–	–	–	–	–	–	–	–
$T_{seal}{}^{*}t_{seal}$	0.0102	0.0044	0.0094	0.0104	–	–	0.0051	0.0264	0.0059	0.0056	–	–
$t_{seal}{}^{*}t_{seal}$	–	–	–	–	−0.0690	0.0191	–	–	–	–	–	–
$T_{seal}{}^{*}p_{seal}$	0.0103	<0.0001	0.0087	0.0010	–	–	0.0055	0.0009	0.0049	0.0012	–	–
$t_{seal}{}^{*}p_{seal}$	0.1246	0.0272	0.1264	0.0315	–	–	0.1058	0.0053	0.1212	0.0007	–	–
$p_{seal}{}^{*}p_{seal}$	–	–	–	–	–	–	–	–	–	–	−0.0206	0.0015
$T_{seal}{}^{*}T_{processing\ [4°C]}$	–	–	–	–	–	–	–	–	–	–	0.0011	0.0079
$T_{seal}{}^{*}T_{processing\ [23°C]}$	–	–	–	–	–	–	–	–	–	–	–	–
$t_{seal}{}^{*}T_{processing\ [4°C]}$	–	–	–	–	–	–	0.1350	0.0440	0.1465	0.0176	0.0274	0.0083
$p_{seal}{}^{*}T_{processing\ [23°C]}$	–	–	–	–	–	–	–		–	–	–	–

TABLE 3 Validation of statistical optimum at various processing temperatures during and after cool processing (n = 5)

	Average peel strength (N mm^{-1})		Maximum peel strength (N mm^{-1})	
Processing temperature	Predicted value	CI measured	Predicted value	CI measured
−18°C—during cool processing	0.80	[1.02, 1.24]	0.99	[1.17, 1.25]
−18°C—after cool processing	0.54	[0.56, 0.67]	0.62	[0.62, 0.68]
4°C—during cool processing	0.47	[0.94, 1.04]	0.63	[0.97, 1.07]
4°C—after cool processing	0.48	[0.60, 0.77]	0.47	[0.68, 0.77]
23°C	0.45	[0.51, 0.62]	0.58	[0.60, 0.66]

Abbreviation: CI, confidence interval.

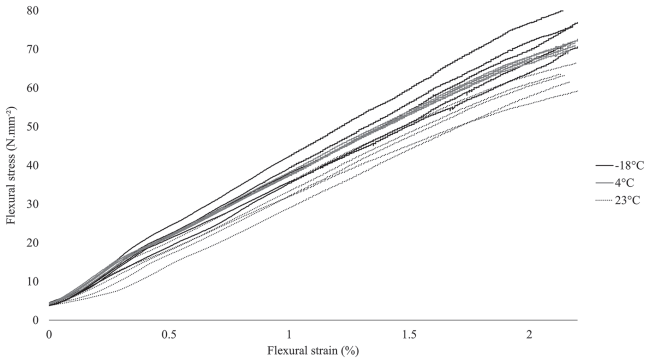

FIGURE 3 Influence of environmental temperature on flexural stress–strain curves of a polyethylene terephthalate/polyethylene (PET/PE) bottomweb (n = 5)

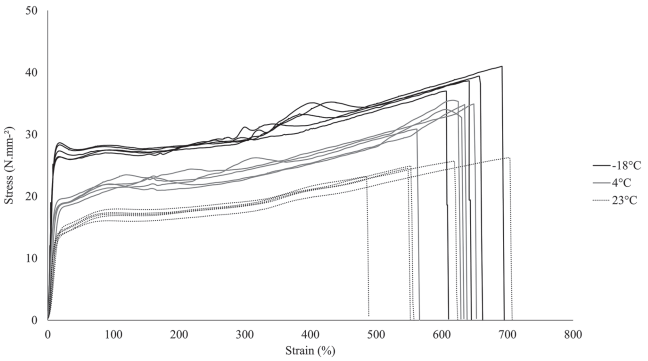

FIGURE 5 Influence of environmental temperature on tensile stress–strain curves of a 60-μm-thick blown extruded monolayer low density polyethylene (LDPE) (LDPE FE8000, Total) film, tested at 300 mm min^{-1} on 15-mm-wide rectangular shaped samples (n = 5)

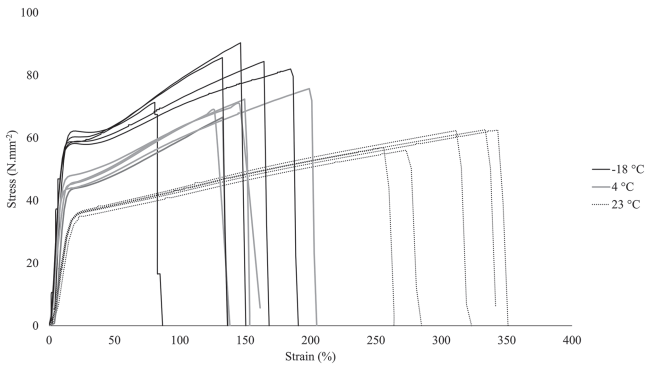

FIGURE 4 Influence of environmental temperature on tensile stress–strain curves of a polyethylene terephthalate/polyethylene (PET/PE)-EVOH-PE topfilm (n = 5)

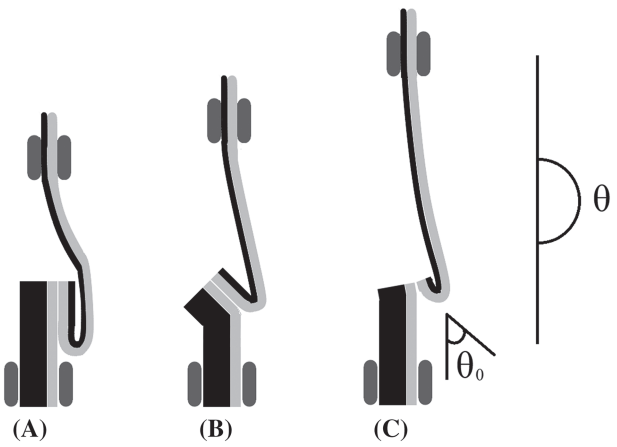

FIGURE 6 Bending of sealed bottomweb during a peel test. A, B and C represent respectively start situation, peel initiation and peel end

Figure 7 shows all raw peel strength-position curves, used to calculate the average and maximum peel strengths in Table 3, and compares it with sealed samples with a reinforced bottomweb to eliminate the differences in bending movement of the bottomweb, and the changes in peel angle partitioning as a consequence of this, at considered temperatures −18°C, 4°C and 23°C.

In all tests, sealed samples with regular bottomwebs tend to achieve lower peel strengths than those that are reinforced with a thin metal plate. These results indicate a slightly negative impact of bending movement on peel strength and peel energy (area under the curve).

The total distance or end position of the peel tests at 23°C is around 20 mm. The end position is the sum of the deformation of the peel arm(s), the peeled distance and the deformation of the peel area.

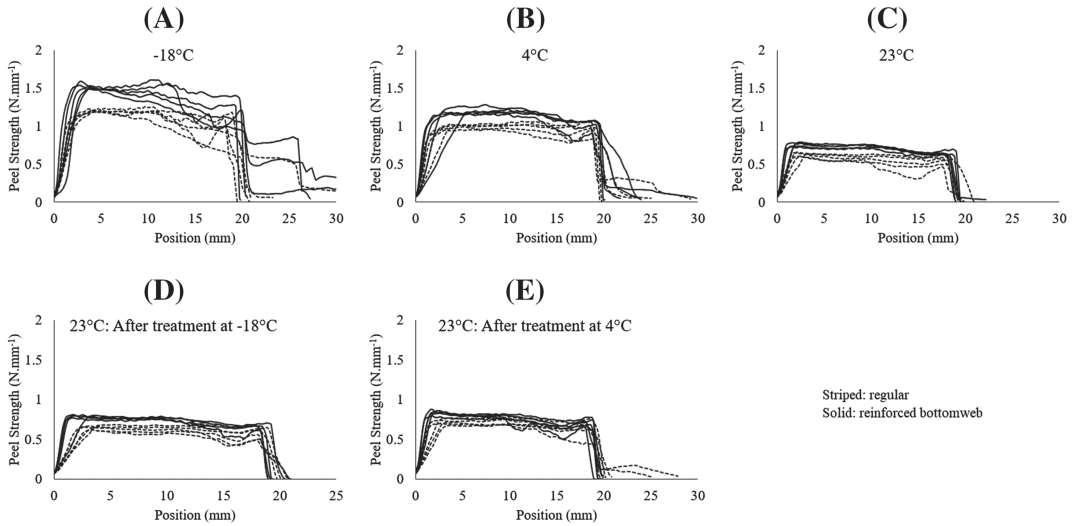

FIGURE 7 Influence of thermal treatment on peel performance of regular and reinforced samples, sealed at 170°C, 1.0 s, 2.0 N mm^{-2}, during (A–C) and after (D and E) thermal treatment at −18°C, 4°C and 23°C (n = 5)

In the beginning of each curve, deformation of the peel arms takes place. This deformation can be differentiated in tensile deformation of the topfilm and bending deformation of the bottomweb. With the reinforced samples, bending deformation is eliminated and a trend of slightly steeper initial slopes can be observed. Initial slopes of regular and reinforced samples are however both very steep and only take a small amount of the total distance. In this regard, the observed higher yield stresses at low temperature in Figure 4 have a zero to minimal impact on total distance, especially with the corresponding normalized strength values that are multiples of the observed peel strength values.

In a T-peel test, the peeled distance corresponds to twice the width of the seal area (W). In a fixed arm peel test, the peeled distance is $W - W * cos\theta$.[9] In the peel tests of this study, which are carried out at a peel angle of 180°, peel distance values of 20 mm are expected because the seal width is 10 mm. In this test, deformation of the peel area is very limited because peeling ends around 20 mm. The impact of the observed higher yield stresses at low temperature with the standard LDPE in Figure 5 has zero to minimal impact on peel distance because of the lack of deformation.

In the curves of the tests during −18°C and 4°C, peel strength is not decreasing as sharply when compared to other tests. This can be explained by the seal failure mechanism. With cohesive peel failure, the materials will be opened around 20 mm and the strength very sharply drops to zero. With combined failure of cohesive peeling and delamination, a small area of the topfilm delaminates during and shortly after cohesive peeling. This results in a less sharp decrease of strength compared to the samples that are fully cohesive peeled. With the regular samples, tested at, respectively, −18, 4 and 23 during thermal treatment, full cohesive peel failure is observed at 3, 4 and 5 out of 5 samples. With the reinforced samples, it was observed at, respectively 1, 4 and 5 out of 5 samples. Other samples were partially delaminated; the occurrence increases at cool temperatures and even more with the use of metal plates as reinforcement for the bottomweb. In a previous study on peelable PE films, translaminar crack propagation was observed with 180° fixed arm peel test. It caused peel force to increase compared to samples with interlaminar crack propagation.[9] As temperature decreases, density of PE will increase because of the decrease in free volume of the amorphous regions in the polymer skeleton.[14] A decreased chain mobility of the polymers in the seal layer at 4°C and especially at −18°C is suggested to be the general cause to promote brittle failure in the peel test of this study. The full cohesive peeling is the preferred failure mechanism because of the clean look and absence of delaminated plastic parts. After thermal treatment, once the samples are tested at 23°C, delamination of the topfilm is rarely observed.

Figure 8 shows images of one sealed sample and peeled surfaces of topfilm and bottomweb. At the right side, microscopic cross sections are shown to visualize the impact of the peel test during thermal treatment on the layer distributions of the peeled topfilm and bottomweb. Partially delaminated samples of the tests at −18°C and 4°C are selected to show more details of the undesired seal failure mechanism. As previously mentioned, full cohesive peeling occurred in the majority of samples. To prevent delamination, seals can be optimized towards a specified performance at −18°C; care must be taken to reach sufficient peel strengths at higher temperature that prevent opening during transportation, storage and/or handling.

A cross section of a sealed sample in Figure 8a shows that the thickness is around 360 μm; this is a result of sealing a 60-μm topfilm against a 290-μm bottomweb. Small deviations can occur because of heterogeneity of the thickness of commercial plastic films. Cross sections of cohesive peeled topfilms are shown in Figure 8b,d,h. At all considered temperatures, thickness is around 50 μm. This consistent slight decrease of total thickness can be a

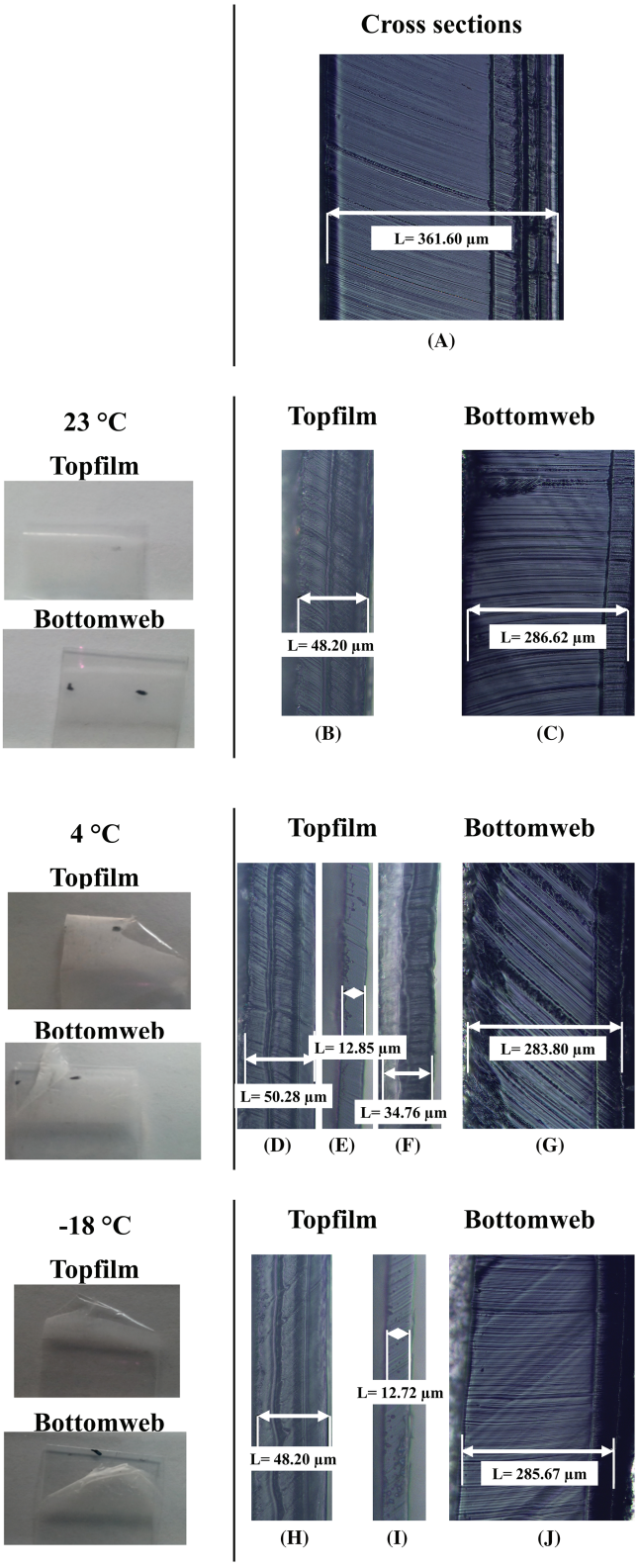

FIGURE 8 (A–J) Pictures and cross sections of sealed and peeled samples that are tested in a peel test during thermal treatment

result of thin layers that are peeled off because of the cohesive failure. Cross sections of cohesive peeled bottomwebs are shown in Figure 8c,g,j. At all considered temperatures, thickness is around 285 µm, which is very close to the original material thickness. Possible effects of thickness increase because of a sticking layer of the topfilm are not clear. With a thick commercial web with a heterogeneous thickness distribution, it is harder to observe slight differences in micrometre range compared to similar differences in the thin topfilm. Cross sections of delaminated topfilms after peel tests at 4°C and −18°C are shown in Figure 8e,f. This cross section is cut out of the transparent part of the topfilm on the left side of the image. The thickness of 12 µm indicates that the 35-µm blown extruded part sticks against the bottomweb and that only one layer remains at the topfilm, PET. This 12-µm part is highly transparent compared to rather hazy elongated seal materials. One cross section (Figure 8i) is made of a stretched out hazy plastic part that remains attached at the bottomweb after peel testing during 4°C. The resulting thickness of 35 µm indicates that the blown extruded part of the topfilm (PE-EVOH-PE) is delaminated and elongated because of the peel test.

Humidity was neglected during this work; in a next study, it can be added as treatment parameter. The proposed method can also be applied with different temperatures, such as pasteurization and sterilization temperatures, relevant for retort packages.

5 | CONCLUSIONS

This work presents a method to evaluate and optimize the peel performance of a packaging concept with a peelable topfilm sealed to a bottomweb. Models are fitted and experimentally validated at optimal settings to achieve a peel strength of 0.5 N mm^{-1} in 23°C. During thermal treatment at −18°C and 4°C peel strength increased, after thermal treatment, there was no impact of treatment temperature on peel strength.

Peel strength increased in a peel test with reinforced bottomweb at all considered environmental temperatures. Bending stiffness of the bottomweb increased during −18°C and 4°C, suggesting a minor impact of environmental temperature on bending of the bottomweb during the peel test. The increase in peel strength is clearly related with a change in seal failure mechanism. Seals peeled cohesively when tested at 23°C, partial delamination occurred during 4 and, more often, during −18°C.

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