OPTIMAL DESIGNS TO DEVELOP AND SUPPORT AN EXPERIMENTAL STRATEGY ON INNOVATION OF THERMOFORMING PRODUCTION PROCESS

Fabrizio Ronchi, Luigi Salmaso[1]
Dipartimento di Tecnica e Gestione dei Sistemi Industriali, Università di Padova, Vicenza, Italia

Mattia De Dominicis
Reckitt Benckiser plc, R&D center, Slough, United Kingdom

Juergen Illert, Krzysztof Krajewski, Rainer Link
Reckitt Benckiser plc, R&D center, Heidelberg, Germany

Luca Monti
Reckitt Benckiser plc, R&D center, Mira, Italia

1. INTRODUCTION

Innovation in thermoforming is a complex challenge. Thermoforming process is affected by various controlled and uncontrolled factors. Definition of factors, operating ranges and a deep understanding of their impact on the final product, is fundamental. Furthermore, innovating a process requires to exit the paradigm under which people are used to operate, so a full understanding of the existing process has to be achieved in order to be able to explore new solutions.

A research aimed to design a new thermoforming production process has been conducted. The new process should allow to produce a packaging releasing detergent in dishwasher at a well-defined moment during main wash. Key part of the process and focus of this study is the sealing of polymeric film. As result of the research, an experimental strategy on innovation of thermoforming for functional packaging has been developed.

Previous studies on multilayer polymeric films investigated the influence of production parameters on their performances (Butler and Morris, 2012), the heat sealing properties of packages (Yamada *et al.*, 2012), and the influence of processing conditions on heat sealing behavior (Iwasaki *et al.*, 2016; Zhao and Lu, 2008). Use of statistical approach in thermoforming is well spread. Design of experiment (DOE) is used for process optimization (Morales and Candal, 2006; Labonte and Dubois, 2011; Smith and Vaidya, 2013), and for quality improvement (Sameh Ibrahim *et al.*, 2012), but there is no evidence in scientific literature of use of DOE for innovation of thermoforming production processes for active packaging.

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[1] Corresponding Author. E-mail: luigi.salmaso@unipd.it

In this study the approach to the problem was based on the principle of DOE. In fact, "The application of experimental design techniques early in process development can result in: improved process yields; reduced variability and closer conformance to nominal or target requirements; reduced development time; reduced overall costs" (Montgomery, 2000). DOE was applied starting from the choice of raw material to the final test in dishwasher. Three control factors were initially chosen by a team of expert in thermoforming, that are temperature, time and pressure. These factors are commonly used to control material sealing in production line. The experimental approach revealed larger impact with current common technology of other factors such as pre-heating temperature and line pace. Final result was the development of a procedure that could allow selection of raw material and of factors' levels combination according to the desired packaging performance by means of an advanced DOE. There are several contributions and examples in scientific literature on techniques used in this study such as optimal design construction (Onukogu and Iwundu, 2007) and response surface (Berti *et al.*, 2006).

This paper illustrates the experimental strategy. A case study provides an example of its development and helps to illustrate steps and benefits of a structured experimental design.

2. PRODUCT AND PRODUCTION LINE

The product under development is a single-dose bottle for detergent. It is made out of a polymeric film bent, formed and sealed. The bottle consists of two chambers. Polymeric material is sealed all around the bottle. Two different seals are required. The first seal, from now on named Strong seal, is all around the bottle, apart from the upper part. The second seal, from now on named Weak seal, is in the top of the two chambers. Strong seal is commonly easy to achieve and it is a common application of polymeric film for packaging. Strong seal has to guarantee resistance to shock during transportation, safe use for customers handling packaging, and permanent sealing in time. Weak seal is the challenge of this research. Weak seal has to open at a well-defined moment under external conditions created during washing cycle in dishwasher. It has to be weaker than strong seal that does not have to open under the same conditions. Nevertheless, it has to resist to transportation shocks and guarantee safety of final customer.

In order to understand the complexity of the analysis it is key to comprehend the product mechanism principle. The two seal types' relation is used with combination of the specific polymer film with ability to retain the memory of shape. During thermoforming process the film is being heated to the glass transition temperatures softening the flat 2D structure to be ready for forming into the new 3D shape defined by the mould. At this stage the material is being stretched, building tensions between polymer chains. Then the mould is cooling down the polymer rapidly "freezing" the tensions. This phenomenon is later used as engine for product release performance in the dishwashing machine. In the cleaning cycle temperature rises above critical level again for the film, unfreezing the tensions, causing material shrinkage. This opposite direction movement of the film (from 3D to 2D) is creating pressure inside of the container. When the pressure is reaching critical level weak seal is breaking but the strong seal remains intact. Thus maintaining a safe margin between strengths ratio of the two seals is critical for the effective opening and release of the product in
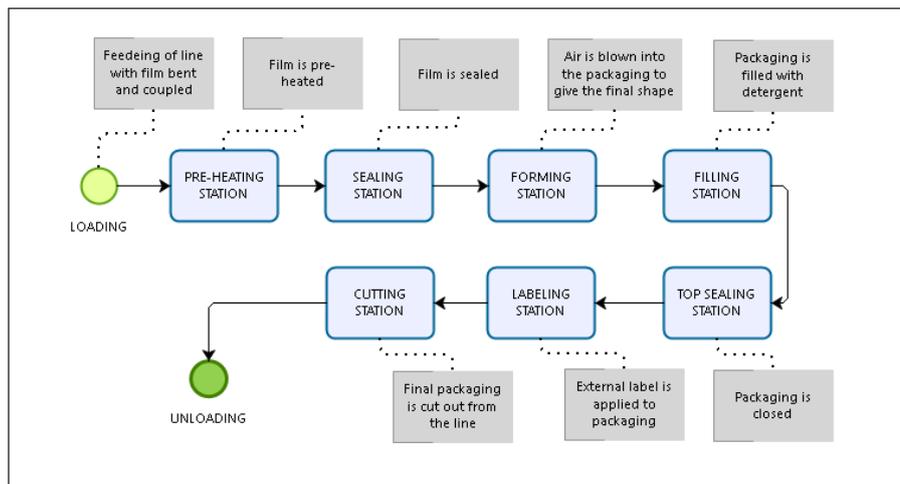
Figure 1 – Scheme of production line.

the right moment in the given environment.

Production lines of packaging based on thermoforming of a polymeric film have a modular structure. Each module or station is devoted to a specific task, and assembly of stations is designed according to the desired packaging following general and established rules of thermoforming production. Film is in big rolls and production is continuous. Scheme of a production line devoted to production of a generic packaging for detergents is given in Figure 1.

3. THE EXPERIMENTAL STRATEGY

The experimental strategy developed thanks to principles of DOE is composed of three phases:

1. *Material characterization.*
   Objective: material selection.
   Success criteria: i) differentiability between Weak and Strong seal; ii) low variability of seal performance related to small fluctuation of production control factors; iii) low variability of seal performance related to level of control factors.
   Factors: i) temperature of sealing jaws; ii) time of sealing; iii) force per cm^2
   Response: seal strength value.

2. *Production in pilot line.*
   Objective: Weak seal investigation, and selection of samples for dishwasher test.
   Success criteria: i) same behavior between left and right chamber; ii) low variability of seal performance related to levels and small fluctuation of production control factors.
   Factors: i) pre-heating station temperature; ii) pace of the line; iii) time of sealing.
   Response: burst pressure value.

3. *Performance of final product.*
   Objective: find successful factor level combinations, and correlation between pressure test results and dishwasher test results.
   Success criteria: i) time of opening; ii) complete release of detergent.
   Response: electric conductivity of water.

Final result is the selection of factor level combinations in production according to the desired packaging performance taking into account peculiar features of material, and correlation between factor level combination and performance. The strategy is summarized in Figure 2.

4. MATERIALS CHARACTERIZATION

The market of polymeric film for thermoforming applications is huge and so is products offer from each supplier. Coupled PET/PE films in the market have different features, and selection is made according to the specific industrial application. In the present specific case, novelty is given by the fact that same material is required to perform in two clearly different ways when sealed.

Objective of characterization is the description of features of material based on a certain response variable measured on seals obtained according to different configurations of production control factors: i) temperature of sealing jaws, ii) time of sealing, and iii) force per cm^2 applied to the material. Sealing was performed controlling the three factors. Response variable is measured by a tensile strength test giving the force needed to open the seal tearing apart the two extremities. The idea is that the force needed to open the seal is representative of the final performance required to the packaging. The measure is obtained by a dynamometer.

Most important features of material according to the purpose of this study are:

1. *Differentiability between weak and strong seal.*
   Differentiability between weak and strong seal is fundamental. Response variable cannot vary within a small interval. It has to show enough difference between values observed at different factor level combinations in order to achieve the control in production of weak and strong seal.

2. *Low variability of seal performance related to small fluctuation of production control factors.*
   Industrial production is in general affected by variability of input factors compared to laboratory experimental environment. To gain the control of production process, levels of control factors have to be chosen among those configurations that shows small effects on response variable as consequence of common fluctuations of input factors.

3. *Low variability of seal performance related to level of control factors.*
   Previous experiences of the team involved in the project revealed high variability of strength test at certain levels of control factors depending on the material. In presence of such high variability, the phenomenon under investigation is out of control. Industrial production of packaging would be impossible at those factor level combination that gives high and uncontrolled variability in response.
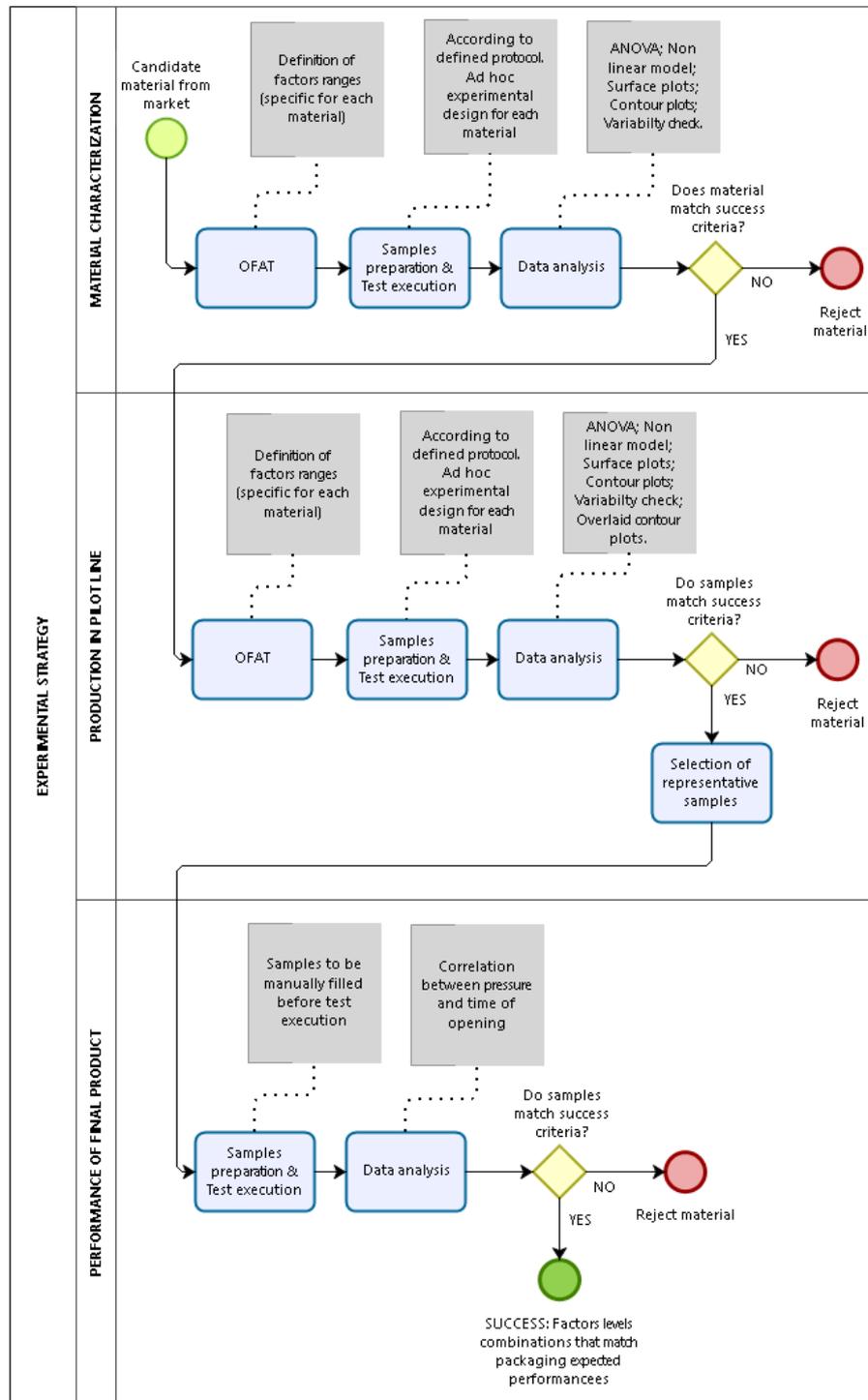
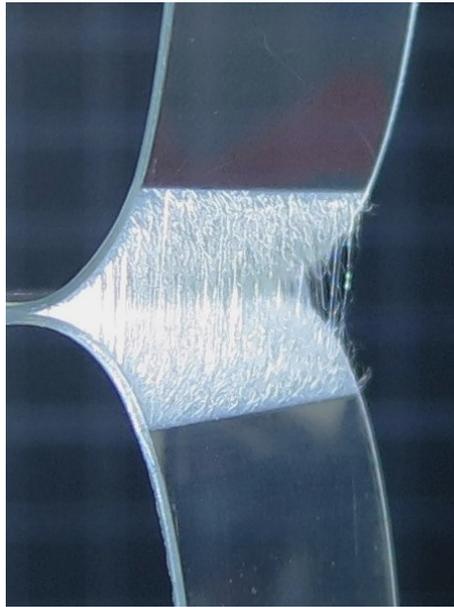*Figure 2* – Scheme of the experimental strategy.

Figure 3 – The seal opening during the tensile strength test of material A. The test measures the force necessary to open the seal tearing apart the extremities of the stripe.

4.1.   The experiment

Characterization of first candidate material was performed. In laboratory flat foils were sealed and tested. Sealing procedure was according to a protocol in order to reduce variability of response: flat foil were cut in squares 10 cm x 10 cm, randomly coupled and sealed always at the same distance from the border; sealing bars were steel made Teflon coated in order to avoid sticking to foil surface; temperature stabilization time of sealing bars was 10 minutes; once sealed, an overnight curing time have been respected in order to allow stabilization of polymeric bonds; 15 mm width stripes were cut out from the sealed squares and prepared to be tested; external stripes were excluded from analysis to avoid distortions given by border effects. There was a protocol for tensile strength test as well: threshold force before start 0.1 N; displacement speed of grippers 50 mm/min; initial gap between grippers 35 mm. Both sealing and testing were performed by the same expert operator for the whole experimental design to avoid variance introduced by different operators. The result of tensile strength test is the maximum force measured to open the seal tearing apart the two extremities, from now on named Seal strength. Measure of seal strength value is in N/15 mm because the opening of the seal is orthogonal to the length that is 15 mm. In Figure 3 seal opening during the tensile strength test.

4.2.   Experimental design

At the beginning a one factor at time (OFAT) experiment allowed to identify operative ranges of factors for candidate material. Some ramps have been performed for each factor with the other two factors fixed at common levels for sealing, and

ranges for factors were defined: Temperature 120 - 150 °C; Time 1 - 1.6 s; Force 20 - 60 N/cm². Operative ranges are specific for candidate material. Their definition should avoid inconsistent or inhomogeneous seal pattern that means too weak seal. At the opposite ranges definition should avoid even too strong seal that lead to material delamination that occurs when the two layers of PET and PE detach and material breaks but seal does not open.

There are different options to design the experiment. Central composite designs are commonly used but best option for this study is optimal design to avoid problems with non-cuboidal regions (Anderson and Whitcomb, 2014). In fact, preliminary experiments revealed that the factorial space defined according to selected ranges couldn't be explored entirely without falling into meaningless data regions, that is inconsistent seal pattern or material delamination. Therefore an optimal factorial design (Draper and Pukelsheim, 1996; Johnson *et al.*, 2011) with 3 factors and 4 levels each was used to explore the response space in an effective and resource saving way. Levels of each factor have been selected in order to have almost homogenous intervals in the ranges (Table 1). Reduction of number of combination from the full factorial design (64 combinations) to the fractional factorial design (38 combinations) were achieved according to D-optimality criteria to minimize the variance in the regression coefficients of the fitted design model. The model selected includes terms up to order two, so that second order interactions can be estimated (D-efficiency=0.6825575). Sequence of factors levels combination was random during specimens preparation. Number of replicates was 8. The number of replicates is precautionary in order to guarantee high power of inferential tests that could be necessary in this explorative phase and taking into account that this test does not need long time to be performed. When needed a sequential experiment is useful to zoom in the factors levels.

TABLE 1
Levels of control factors selected for the experimental design.

Temperature		Time		Force per cm²	
level code	value [°C]	level code	value [s]	level code	value [N/cm²]
1	120	1	1.0	1	20
2	130	2	1.2	2	35
3	140	3	1.4	3	45
4	150	4	1.6	4	60

4.3. Analysis of data

The analysis of response variable aims to understand the impact of control factors on the most important features of the material, and to describe how response varies according to different factor level combinations. Analysis of variance of a full quadratic model allowed evaluating both main factors and interactions. A full quadratic model takes into account all linear terms, all quadratic terms, and all two-way (linear*linear) interactions. Then a backward stepwise model selection procedure at a significance level $\alpha = 0.05$ has been applied to obtain the most parsimonious model with all the significant effects and interactions (Table 2).

TABLE 2
ANOVA table of the final model for material A.
T = Temperature, t = Time, F = Force per cm².

Source of variation		DF	Sum of squares	Mean square	F-value	p-value
Model		7	588.270	84.039	188.30	0.000
	T	1	351.874	351.874	788.44	0.000
	t	1	39.248	39.248	87.94	0.000
	F	1	4.310	4.310	9.66	0.002
	T^2	1	131.648	131.648	294.98	0.000
	t^2	1	4.054	4.054	9.08	0.003
	T*t	1	8.919	8.919	19.98	0.000
	T*F	1	5.655	5.655	12.67	0.000
Residual		296	132.102	0.446		
Total		303	720.372			

According to p-values the model and the three main factors are significant to explain the response variable. That is the means of the response variable are different at a confidence level of 95% between at least two factor levels. Only two interactions are significant. Interaction between Time and Force per cm² has not a significant impact on the seal strength. Coefficient of determination (R^2) of model is 81.66%. Analysis of residuals for ANOVA assumptions does not emphasize particular trends in data patterns, that is no violation of assumptions.

The impact of the terms of final model can be assessed (Table 3). To reduce the impact of non-orthogonal terms, the model was fitted in coded units. The effect of Temperature is much bigger than effect of Time, which in turn is bigger than effect of Force per cm². Low level of variance inflation factor (VIF) shows absence of multicollinearity and confirms the goodness of the model.

TABLE 3
Effects of the terms of the final model in coded units and VIF.
T = Temperature, t = Time, F = Force per cm².

Term	Effect	VIF
T	2.9388	1.04
t	0.9818	1.03
F	0.3412	1.04
T^2	-2.9797	1.01
t^2	-0.5319	1.05
T*t	-0.6427	1.04
T*F	-0.5334	1.07

The model allows obtaining a nonlinear regression equation for Seal strength:

$$S = -154.60 + 2.014T + 18.96t + 1.286F - 0.006622T^2 - 2.955t^2$$
$$- 0.0714T \cdot t - 0.00889T \cdot F,$$

where S = Seal strength, T = Temperature, t = Time, and F = Force per cm².

The equation provides "prediction" of response variable for those factors values
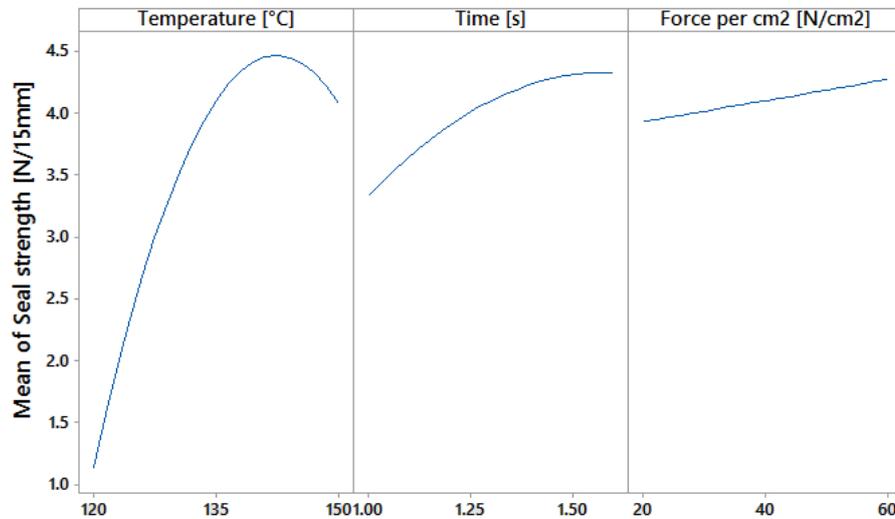
Figure 4 – Main effects plot for Seal strength, fitted means: mean of Seal strength vs. Temperature, Time, and Force per cm².

that have not been tested during the experiment (only in the investigated ranges of factors values).

The effect on the response variable of each factor can be graphically analyzed (Figure 4). The analysis is based on the nonlinear regression equation. Temperature has a high impact on Seal strength. The range of observed values of Seal strength mean is wide (between 0.92 and 4.35 N/15 mm) at Temperature varying. There are steep slopes moving from a level of Temperature to the next one. There is a maximum at approximately 140 °C, then Seal strength decreases at 150 °C. Time effect plot has generally a positive slope with a flat zone at higher values. Force per cm² effect is the less evident. Range of observed values of Seal strength mean is between 2.93 and 3.41 N/15 mm at Force per cm² varying. Graphical analysis of interactions revealed general interaction between Temperature and Force per cm², and between the Temperature and Time only at high levels.

Nonlinear regression equation allows graphical analysis thanks to surface and contour plots. Surface plots show response variable in a 3D space for each couple of factors, while contour plots show response variable in a 2D space for each couple of factors. The remaining factor is fixed at a predefined level.

Surface plot gives a clear vision of the factors levels impact on response variable. Surface plots in Figures 5, 6 and 7 are the most representative of the plots analyzed. They show Seal strength versus each couple of factors holding the third at intermediate level. So, for instance, Figure 5 show Seal strength versus Time and Temperature, while Force per cm² is fixed at 40 N/cm² that by the way is not one of the levels of experimental design. This type of graphs provides interesting information about the impact of factors. In Figure 5 Flat area and Steep slope area are highlighted. Flat area individuates configurations of the two plotted factors levels that have a stable impact on response given the third level fixed. In fact, small variations of the factors don't
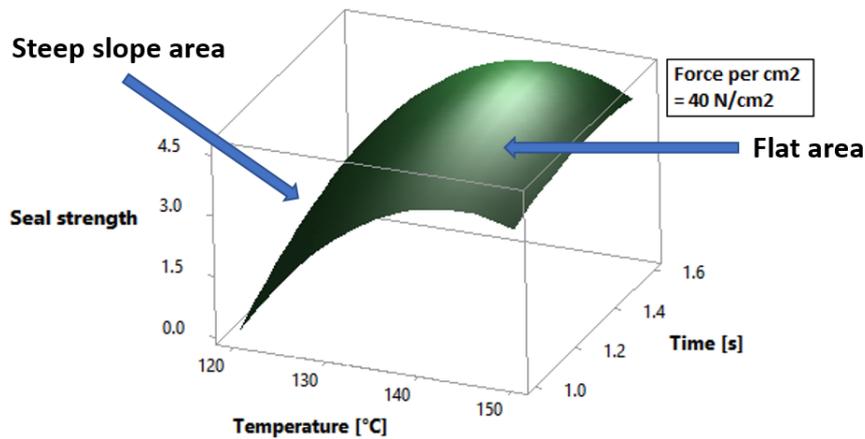
Figure 5 – Surface plot of Seal strength vs. Time and Temperature.

show effects on response. This information is very important in industrial production to have full control of production process. Small fluctuations of input factors are common and they could have an impact on the stability of output. This information must be crossed with impact of the third factor taking into account at the same time the plots versus the three couple of factors. On the other hand, steep slope area reveals high impact of variation of factors.

The three plots have been analyzed varying the third factor along the whole range. Flat area and steep slope area have been individuated and assessed for candidate material.

Contour plot is similar to surface plot but it is in two dimensions. In a contour plot two factors are in the axes, while the response variable is shown by different colored areas. The remaining factor is fixed at a predefined level. Contour plots in Figures 8, 9 and 10 are the most representative of the plots analyzed. They show Seal strength versus each couple of factors holding the third at intermediate level. In Figure 8 there is an example of how differentiation between strong and weak seal can be achieved by changing the level of Time and Temperature. Seal strength goes from lowest values in point A (approximately 0.5 N/15 mm) to the highest in point B (approx. 4.2 N/15 mm) holding Force per cm^2 at 40 N/cm^2. Areas contours are not straight but elliptical because of the combined effect of Temperature and Time. In Figure 9 there is a different representation of the concept of flat area and steep slope area. In fact from 120 °C an increase of 5 °C temperature produces an increase in the Seal strength of more than 1 N/15 mm (steep slope area). At 140 °C the same increase does not produce an increase in the Seal strength (flat area). Areas contours are almost straight, the higher impact of Temperature compared to Force per cm^2 is evident.

The three plots have been analyzed varying the third factor along the whole range. Differentiability between weak and strong seal has been individuated and assessed for candidate material.

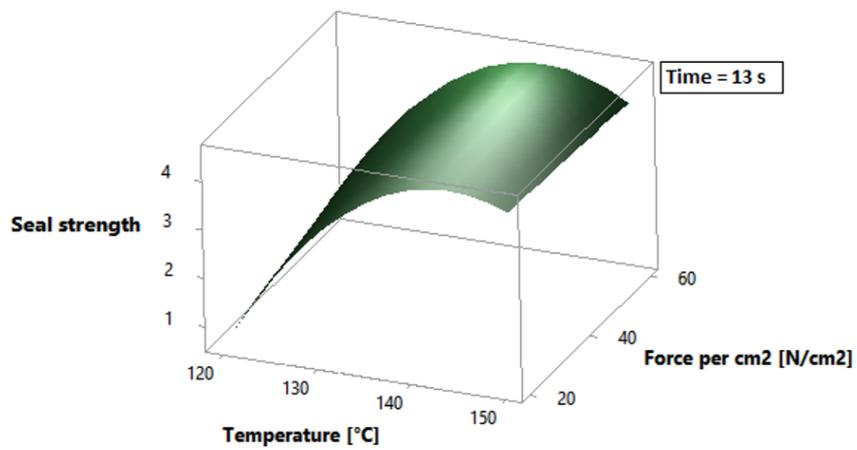Last graphical analysis according to the most relevant features of material is the

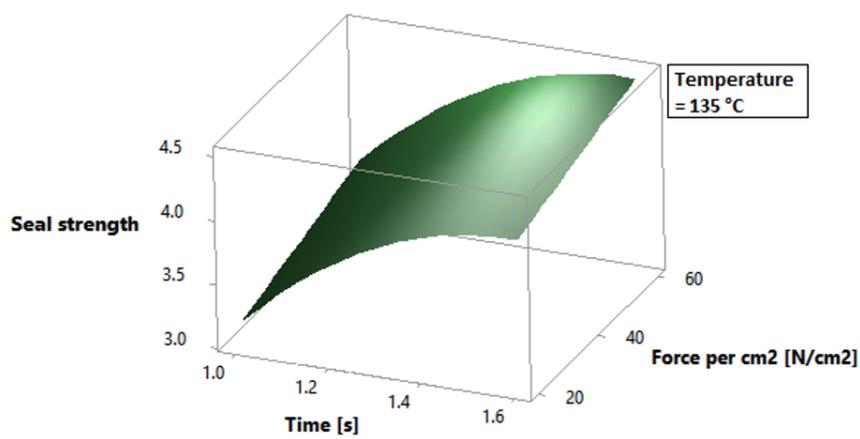*Figure 6* – Surface plot of Seal strength vs. Force per cm² and Temperature.

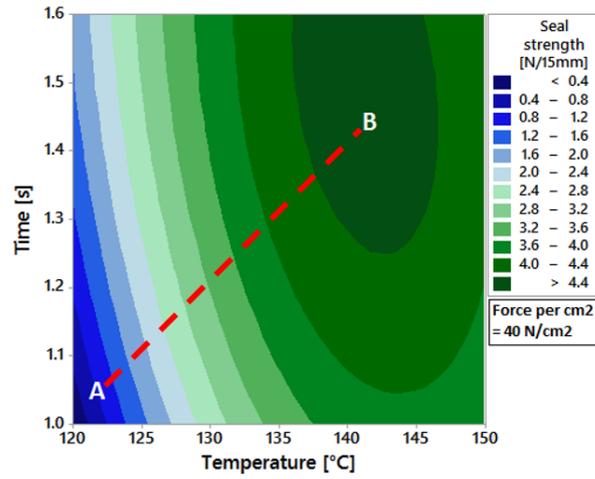*Figure 7* – Surface plot of Seal strength vs. Force per cm² and Time.

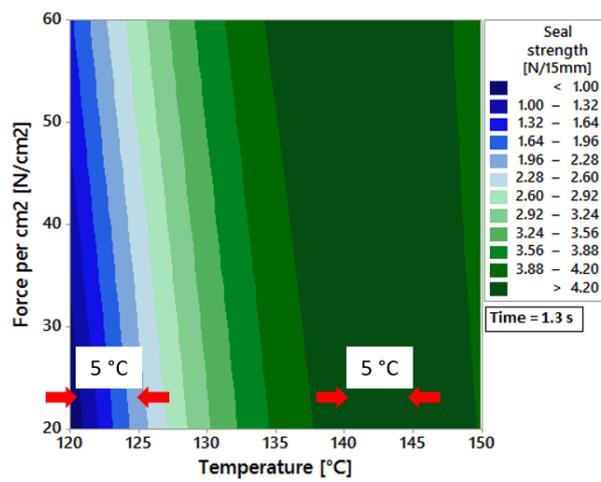*Figure 8* – Contour plot of Seal strength vs. Time and Temperature.

Figure 9 – Contour plot of Seal strength vs. Force per cm² and Temperature.
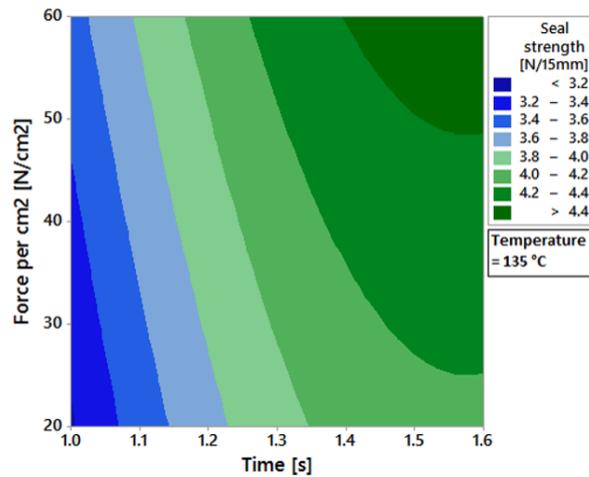
Figure 10 – Contour plot of Seal strength vs. Force per cm² and Time.

one concerning data variability at certain factor level combinations. Graph in Figure 11 shows Seal strength distributions according to the 38 factors levels combinations of the experimental design. Response of candidate material has large variability at certain factor level combinations. Variability could affect properties of final product since a narrow window is expected for weak seal acceptable performance.

Material characterization is performed to select candidate materials from market according to the most important features required for production. It allows studying differentiability between weak and strong seal, variability of seal performance related to small fluctuation of production control factors, and variability of seal performance related to level of control factors. Candidate material revealed to be a good candidate for scale up to production line.

5. PRODUCTION IN PILOT LINE

The objective of the research is production of packaging from one material, and the challenge is to achieve weak and strong seals. Stations that could impact on seal performance are pre-heating station and sealing station. Pre-heating station prepares film before sealing. Film goes through the station, and two hot plates provide thermal energy. Temperature of pre-heating station is a control factor. Sealing station is composed of two faced molds that close cyclically and seal the film. Temperature, time and force per cm² are control factors in sealing station.

Traditional approach to packaging production by thermoforming process considers temperature, time and force per cm² the most important factors to control seal features, while temperature in pre-heating station is used to control film performance in forming station. In fact, pre-heated material is soft and can be easily modelled by blowing air to achieve the final shape, then packaging is cooled down to fix the shape. Traditional approach make sense for traditional packaging, but production of weak seal for the new functional packaging challenged it.

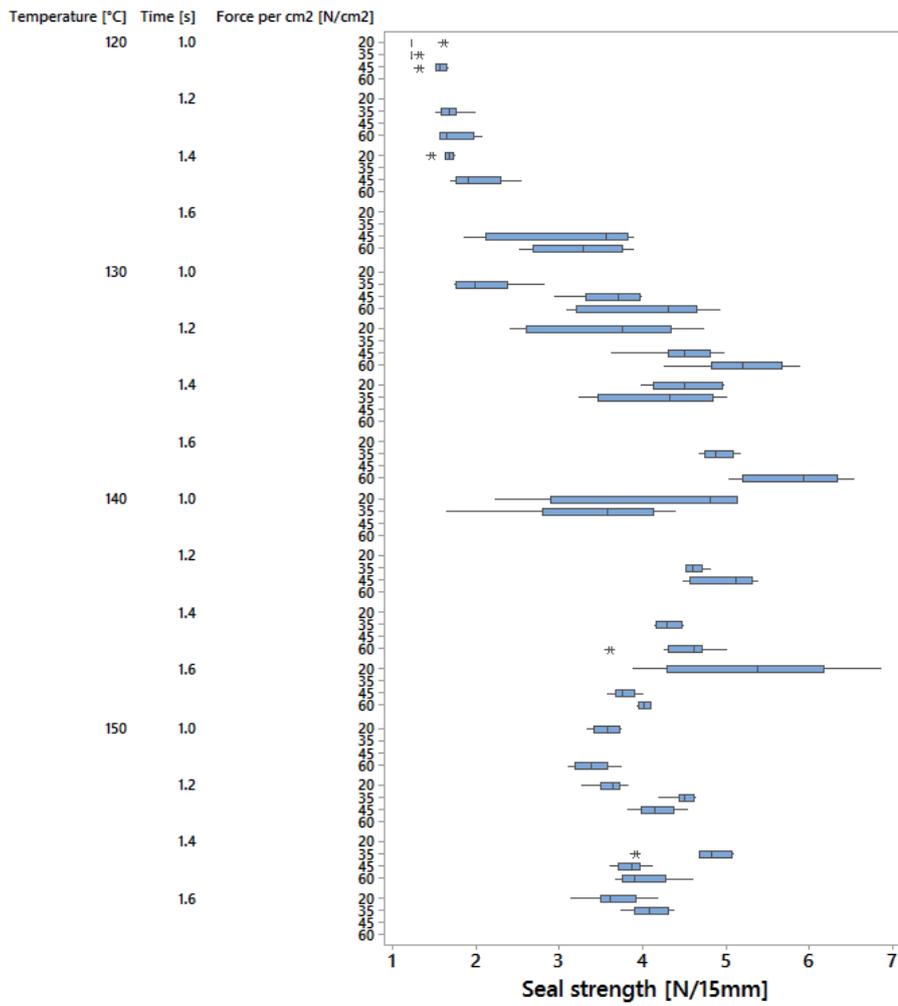Molds in sealing station are made of two different materials, one for the weak

Figure 11 – Boxplot of Seal strength according to factors levels combinations.

seal and one for the strong seal. These two materials are embedded in one mold, and control of time and force per cm^2 is independent for the two materials so differentiation is allowed according to these two factors. Temperature control is unique for the whole mold and differentiation is achieved by the different thermal conductivity. A confirmatory experiment based on levels of factors used in laboratory for candidate material was thus performed. Despite temperature, time and force per cm^2 conditions for weak sealing jaws were the same as the ones used for the experiments in the laboratory, results obtained in the pilot production line were completely different. Weak sealing failed. The systematic experimental approach allowed to individuate the source of failure in the differentiation by thermal conductivity. Laboratory experiment settings on flat film are not transferrable to pilot production line taking into account current production system.

As per results of lab experiments for characterization of candidate material, temperature has the biggest impact on seal performance compared to time and force per cm^2. New factors that could allow control of transferred thermal energy were investigated. Pre-heating station gives thermal energy to the film. Preliminary experiments allowed to select three main control factors for weak seal production in pilot line: i) pre-heating station temperature; ii) pace of the line; iii) time of sealing. Pace of the line is measured in cycles per minute and it allows control of the time the film spends in the preheating station. Control of thermal energy transferred to the film is achieved by combination of pre-heating station temperature and pace of the line. Note that according to material characterization results, force per cm^2 was considered negligible as control factor in production line. Sealing was performed controlling the three factors.

The experimental response variable is measured by a burst test giving the pressure needed to open the seal by injecting air into the bottle. The idea is that the packaging shrinks when temperature in dishwasher raises because of the memory form effect of thermoformed polymeric material. The effect of shrinkage is the increase of internal pressure that at the end leads to the packaging opening. The pressure needed to open the seal is thus representative of the final performance required to the packaging.

The final objective is to produce a packaging releasing detergent in dishwasher at a well-defined moment during main wash. Response variable analysis should allow to select samples for dishwasher test in order to find a correlation between packaging performance in dishwasher and production factors levels, and in case of success to find the best factors setting for production. The reason of making a selection by means of pressure test before dishwasher test is that the latter is highly time consuming.

5.1. The experiment

Candidate material was used to produce batches of finite empty bottles according to an experimental design. In production line presence of uncontrolled factors and of fluctuations of controlled factors is higher compared to lab environment. Production was according to a protocol in order to reduce variability of response: production was in steady state; temperature stabilization time of pre-heating station was 10 minutes; sealing station temperature was fixed at 120 °C; force per cm^2 applied to weak seal was 410 N/cm^2. A curing time of at least 1 day have been respected in order to allow stabilization of polymeric bonds. There was a protocol for burst test as well:

air is injected in a flat area of packaging far from seals; air is blown into the bottle according to a pressure ramp with steps of amplitude 0.02 bar; interval between steps is 5 seconds; pressure ramp starts at 0.05 bar and upper limit is 0.5 bar. Burst test were performed by the same expert operator for the whole experimental design to avoid variance introduced by different operators. The result of burst test is the minimum pressure needed to open the bottle. Pressure test device provides the relative pressure (over atmospheric pressure) as percentage of 1 bar.

The experiment is focused on weak seal. In fact, proven that candidate material allowed differentiability of performance in material characterization section, time and force per cm^2 for strong seal in sealing station were tentatively chosen in order to guarantee a minimum pressure at burst test of 0.5 bar.

5.2.   Experimental design

At the beginning a OFAT experiment allowed to identify operative ranges of factors (Table 4). Some ramps have been performed for each factor with the other two factors fixed at common levels for production, and ranges for factors were defined: pre-heating temperature 120 - 130 °C; pace of the line 11 - 14 cycles per minute; sealing time 1.6 - 2.2 s. Operative ranges are specific for candidate material. Defined ranges should avoid inconsistent or inhomogeneous seal pattern that means too weak seal. At the opposite ranges definition should avoid even too strong seal that prevents bottle opening in dishwasher. As per the first experimental campaign best option to design the experiment is Optimal Design in order to avoid problems with non-cuboidal regions. Therefore an optimal factorial design with 3 factors and 4 levels each was used to explore the response space in an effective and resource saving way. Levels of each factor have been selected in order to have almost homogenous intervals in the ranges. Reduction of number of combination from the full factorial design (64 combinations) to the fractional factorial design (38 combinations) were achieved according to D-optimality criteria to minimize the variance in the regression coefficients of the fitted design model. The model selected includes terms up to order two, so that second order interactions can be estimated.

Packaging is composed of two chambers: left chamber and right chamber. They were tested separately, that is for each packaging only left or right chamber were tested. In fact packaging is asymmetric and different geometry between the chambers could have an impact on weak seal performance. Number of replicates was 10, 5 to test left chamber and 5 to test right chamber. The number of replicates is precautionary in order to guarantee high power of inferential tests that could be necessary in this explorative phase.

TABLE 4
Levels of control factors selected for the experimental design.

Pre-heating temperature		Pace of the line		Sealing time	
level code	value [°C]	level code	value [cycles/min]	level code	value [s]
1	120	1	11	1	1.6
2	123	2	12	2	1.8
3	127	3	13	3	2.0
4	130	4	14	4	2.2

Figure 12 – Weak seal of left chamber and right chamber of the bottle.

5.3. Analysis of data

Data analysis was based on the same approach used for material characterization. General steps of analysis are here briefly summarized. For each chamber, Analysis of Variance of a full quadratic model allowed evaluating both main factors and interactions taking into account all linear terms, all squared terms, and all two-way (= linear*linear) interactions. Then a backward stepwise model selection procedure at a significance level $\alpha = 0.05$ has been applied to obtain the most parsimonious model with all the significant effects and interactions. The impact of the terms of final model was assessed. VIF was useful to exclude multicollinearity. The models allowed obtaining a nonlinear regression equation for response variable. Graphical analysis of the effect on the response variable of each factor and second order interactions was performed as well as analysis of surface and contour plots. Last but not least, graphical analysis of data variability at different factors levels combinations.

In general, an experiment performed in industrial plant presents a limit in data analysis compared to one performed in laboratory. Surface and contour plots graphical analysis in material characterization allowed to assess impact of fluctuation of factors. In fact, laboratory environment enables high control of factors, and variability observed at a certain factors levels combination is mainly related to uncontrolled factor, as for instance homogeneity of material. In production line, control of factors is affected by typical variability of industrial environment and sensors are in general less sensitive. The effect of fluctuation of control factors is thus confounded with the effect of variability of uncontrolled factors. This limit should be taken into account when drawing conclusion from data analysis.

Range of observed values was from 0% to 50%, meaning that choice of factor levels allowed investigation of the whole interval of interest of response values. The three factors pre-heating station temperature, pace of the line and time of sealing are significant in the model of both chambers. Pace of the line is the most important factor when considering the impact on pressure value required to open the weak seal of both chambers of the samples. Pressure value increases at low levels of cycles per minute, while it decreases at high levels of cycles per minute.

One important result was that the test revealed a non-symmetric behavior of the left and right chamber of the bottle. Geometry influences the way the chambers open. In Figure 12 weak seal area of the two chambers. Different distributions of response values were observed between the two chambers for many factor level combinations. As a consequence, nonlinear regression equations obtained from the models of left and right chambers were different. A well performing packaging should guarantee that detergent is delivered from the two chambers at around the same time during washing cycle. Therefore, those factor level combinations that could allow
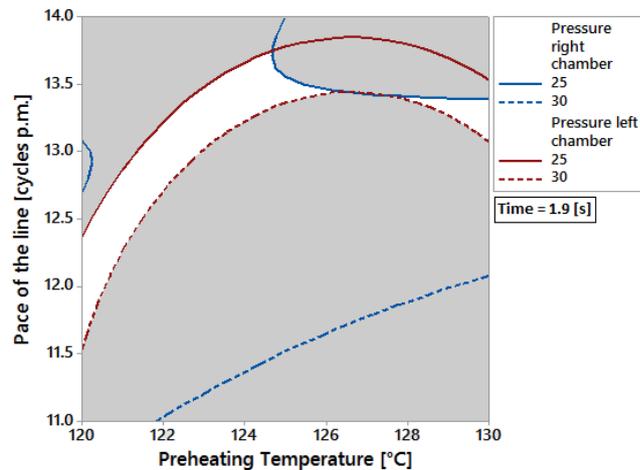
Figure 13 – Overlaid contour plot of Pressure of right chamber and left chamber vs. Pace of the line and Preheating temperature. Interval of response from 25% to 30%.

similar performance were investigated.

Response contour plots of the two chambers were compared by overlapping them two by two. The analysis of overlaid contour plots was performed for each couple of factors. Analysis allowed identification of areas where predicted responses were similar. Examples of overlaid contour plots in Figures 13, 14 and 15 are for interval of response from 25% to 30%. Intervals in the whole range of response have been investigated. Furthermore, the three plots have been analyzed varying the third factor along the whole range. The statistical model allowed to select those factor level combinations that results in a similar behavior of the two chambers.

Pilot line experiments are performed on those materials that show promising results according to material characterization. The objective is the selection of samples showing different performances at pressure test to perform the most significant test: the test in dishwasher. Selection must consider: i) coverage of different pressure test performances, ii) similar behavior of the two chambers, and iii) low variability of response according to factor level combinations. Candidate material allowed selection of samples to be tested in dishwasher.

6.   Performance of final product

Performance of final product can be evaluated according two dimensions: i) time of opening during washing cycle, ii) complete vs. partial release of detergents. These two dimensions are representative of the expectations of final customer. In fact, packaging has to deliver detergent on or before a defined threshold so that there is enough time for detergent for an effective cleaning. Furthermore, complete release of detergent is given as qualifier by marketing.

The two dimensions were investigated by a dishwasher test. Time of opening was established measuring the electric conductivity of water in dishwasher. Electric conductivity changes when detergent is released because of chemical composition.
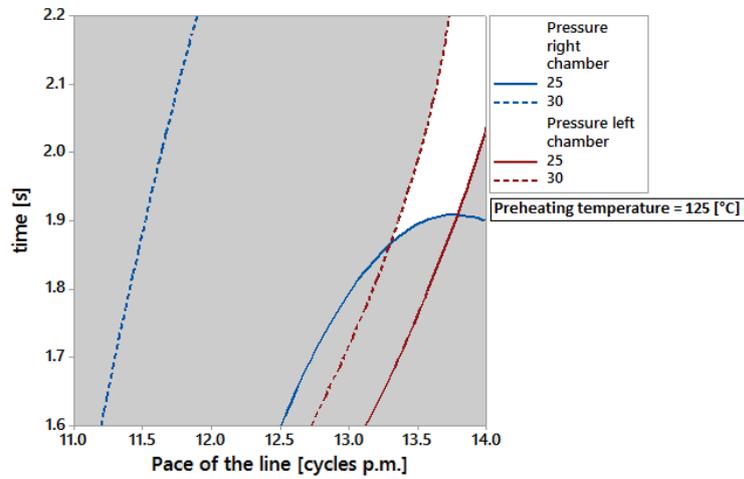
Figure 14 – Overlaid contour plot of Pressure of right chamber and left chamber vs. Time and Pace of the line. Interval of response from 25% to 30%.
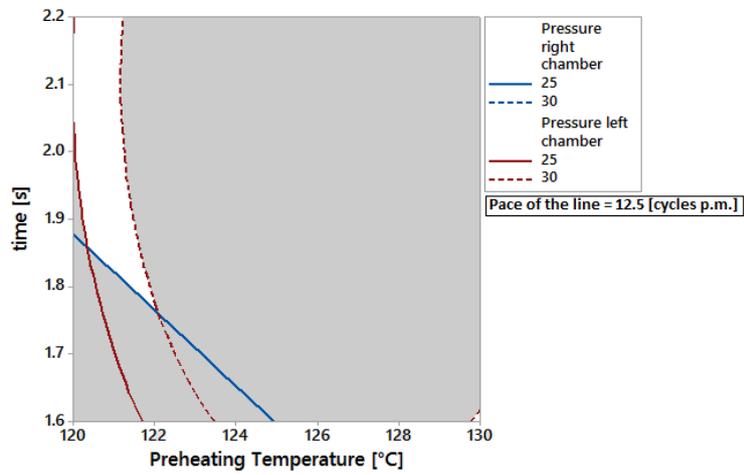

Figure 15 – Overlaid contour plot of Pressure of right chamber and left chamber vs. Time and Preheating temperature. Interval of response from 25% to 30%.
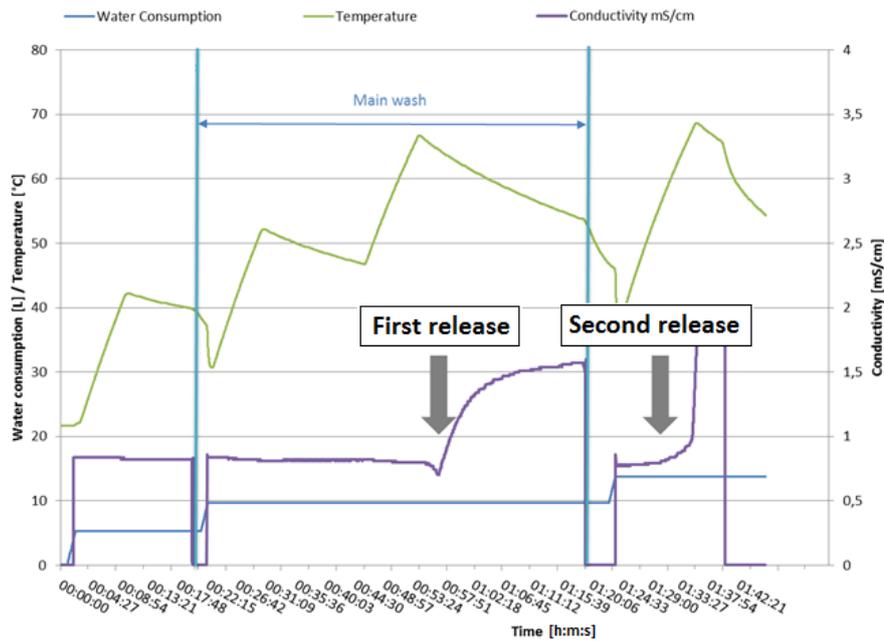
Figure 16 – Observed electric conductivity of water [mS/cm], water temperature [°C], and amount of water [l] during a washing cycle.

The amount of delivered detergent was observed at the end of washing cycle. This test is time consuming (a washing cycle is about 100 minutes) so packaging samples to be tested were selected according to their response at the pressure test.

6.1. The experiment

Packaging samples were manually filled with detergent. They were tested individually in an intensive washing cycle (70 °C) without dish load, all with the same dishwasher. Sensor measured i) electric conductivity of water [mS/cm], ii) water temperature [°C], and iii) amount of water [l]. In Figure 16 there is an example of measured values during an experimental run.

Measured data provide the exact moment in which the two chambers open. Observer can't discriminate between left and right chamber, but only between first and second release. Crossing electric conductivity data with water temperature and amount, it can be established even in which part of washing cycle the two chambers open. Main wash last from minute 22 to minute 80. In Figure 16, for instance, first chamber opens during main wash, while second chamber opens during rinse that by the way is too late.

Samples produced according 8 different factor level combinations were tested. Factors levels combination were classified according to pressure test in three groups: pressure < 20%, pressure 20% - 30%, and pressure > 30%.

6.2. Analysis of data

Analysis of data aims to individuate those factors levels combinations that satisfy minimum criteria in the dimensions under investigation. Furthermore, a correlation between pressure test and opening time is investigated. Mean data observed are in Table 5.

TABLE 5
Observed opening time [minutes] and leftover liquid [%].

Classification in pressure test	Pressure test		Opening time		Leftover liquid	
	left chamber	right chamber	1st release	2nd release	left chamber	right chamber
$< 20\%$	15.4	13.8	N/A	N/A	N/A	N/A
20%-30%	21.4	21.4	57	91	20	10
20%-30%	22.5	22.2	56	89	20	10
20%-30%	29.0	25.8	58	90	10	10
20%-30%	29.4	23.0	56	86	10	10
20%-30%	28.2	25.0	58	90	10	10
$> 30\%$	40.0	33.8	98	N/A	100	40
$> 30\%$	39.4	33.8	99	N/A	100	50

In general, test reveals a basic correlation between product release and pressure:

— Pressure test value $< 20\%$: packaging does not satisfy minimum safety criteria for customer; detergent leak out during manual activation before placing in dishwasher.

— Pressure test value 20 - 30%: first detergent release is in the main wash at minute interval 56 - 58, second release is during rinse cycle; product release is in the range from 80% to 90%.

— Pressure test value $> 30\%$: only one chamber opens at the end of the rinse cycle; there is partial detergent release.

None of the selected samples reached the minimum quality criteria. Candidate material revealed unsatisfactory performance in the final product. Only a systematic approach allowed the investigation of the whole factorial space and the final evidence based assessment of the material.

7. CONCLUSIONS

An experimental strategy on innovation of thermoforming production process has been developed. DOE techniques were used in designing and analyzing all the phases of the strategy. DOE enhanced innovation capability allowing reduction of systematic errors and distortions, full exploration of factorial space, and reduction of number of tests (Montgomery, 2000; Johnson *et al.*, 2011). The experimental strategy allows selection of material and correlation of control factor levels to packaging performance for each tested material.

Traditional approach (Iwasaki *et al.*, 2016; Zhao and Lu, 2008) to production control in thermoforming process was challenged. DOE allowed to identify and overcome the mismatch between control factors in laboratory and in production line.

Anyway, mismatch suggests development of 2 separated sealing stations: one for strong seal and one for weak seal. In this way same control factors could be used in laboratory and in production line. Result would be a direct correlation between performance in dishwasher and control factors in laboratory. The experimental chain would be shorter and a significant reduction of number of tests should be allowed.

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SUMMARY

The experimental strategy developed thanks to the DOE methodology enhance innovation capability in the field of thermoforming production processes for active packaging. It is composed of three phases: i) material characterization, ii) production in pilot line, and iii) performance of final product. It allows selection of material, and correlation between control factors in production and performances of final product. Use of optimal designs allows reduction of number of tests, and in the meantime a full exploration of factorial space.

Keywords: Thermoforming; Production process; Active packaging; Optimal design; Design of experiment.