

## **THERMAL SEAL FORCE OF COATED BIDIRECTIONAL STRETCHED PET FILM USED IN FOOD PACKAGING INDUSTRY**

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### **Abstract**

In addition to the mechanical and permeability properties of food packaging, heat sealing ability of packaging is critical in order to protect a product and extend its shelf life. Optimum temperature sealing is an essential necessity in the packaging industry. Important control parameters for bonding are jaw temperature, jaw pressing time and jaw clamping force. Sufficient pressure must be applied to the surfaces to achieve acceptable adhesion. The physical evaluation of an adhesion can provide a useful qualitative indicator of the effectiveness of the heat sealing process and is based on optimization of the parameters of dwell time, temperature and process pressure. The purpose of this work is to define the effective jaw heat and compression time for the seal strength of the surface coated PET film to the APET surface. The analysis of the thermal adhesion performance of the copolyester surface coated biaxially oriented PET (BOPET) film is brought to the literature. It will guide the determination of the process conditions of the packaging machine to be used after the film production in the sector. Thus, it will contribute to production and energy efficiency as there will be analyzed data about the required jaw temperature, jaw pressing time and pressing force in packaging machines. The effects of parameters on the seal strength were analyzed using the Box-Behnken design, which is a response surfaces methodology (RSM). Then, the significance of the variables was examined by regression analysis. In the study, the correlation coefficient value was 0.995 and it is seen that the model represents 99.5% of the experiments. The most effective factor determined at the 0.05 statistics significance level was jaw temperature.

**Keyword:** BOPET, Box-Behnken Experiment Design, Response Surface Method, Heat Seal, Coating.

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## 1. Introduction

Polyethylene terephthalate (PET), the third most extensively used packaging material, satisfy 16% of plastic consumption in Europe [1]. Biaxially oriented polyethylene terephthalate (BOPET) film is frequently used in food packaging applications.

In the packaging process, thermo sealing involves joining one structure to another or to trays to provide a seal. For example, the film is adhered to its own surface and turned into a bag. Another typical application is bonding to plastic containers as top covers. Heat sealing helps to protect the product from the external environment until the product reaches the end user. For food packaging, sealing helps protect the product. It acts in most layer sealing in multi-layer packaging films. Sealing materials have been developed to optimize the sealing function [2].

An inelastic container with high thermal sensitivity, with a flexible film attached as lidding is widely used for packaging food products ready-to-eat meal that only need for heating. Heating to meal is realizable with a microwave or classic oven. Due to the thermal load during heating process, different materials such as aluminum, polyester coated card board and polyethylene terephthalate (PET) are used as tray material. Crystal PET (CPET) trays are especially convenient for this and similar applications. To develop the heat seal ability of these trays with lidding films, usually containers include amorphous PET (APET) on the food contact surface. Usually high thermal resistant materials as APET or CPET are used for plastic container. To improve the coarse heat seal performance of the PET top cover film, a heat sealable layer is usually formed on the film from a lower melting material [3].

After the polyester film production is completed, the heat-adhesive layer is added to the film surface with an additional process. During this process, it produces polyester film in a classical method.

Afterwards, heat sealable and peelable structure is coated on the produced polyester film. In the coating process, the heat sealable and peelable polymer is dissolved in the organic solvent, then the solvent is evaporated and the heat sealable polymer remains as a solid layer on the film [3].

The heat sealing process is described as a method for joining and welding thermo plastic materials. It is commonly used to form packages into bags [4]. The key element in heat sealing is to heat both sides of the thermo plastic structure. In the press type heat sealing process, which is the most known type of heat sealing, the heat is transferred to the surface of the films by the jaw and heat is transmitted to the sealing area through the film. In order for the heat sealing process to take place, the surface is first heated to the required temperature. It is then cooled to complete bonding. Heat sealing can be used to create a hermetic combining [5]. The appropriate and required heating temperature depends on the structure of the thermo plastic films to be bonded with heat treatment [5]. Important factors for the heat sealing process are jaw temperature, jaw pressing time, and pressing force. Adjusting the temperature of the heating block is the easiest way to control the heat sealing process [5]. In order to achieve an acceptable bonding, sufficient pressure and sufficient time must be used for the polymer chains to spread and form bonds across the surface [4]. Bags and pockets are the most common use of laminate film packaging materials [6].

Bonding strength of sealable film is described as heat seal strength. It is affected by sealing jaw temperature, sealing pressure, and dwell time. The heat seal strength, which is related to the film polymer melting temperature, is a function of sealing temperature [7].

Factors which affect the thermal bonding are;

Dwell Time: Heat sealing process time and temperature are the two most basic

parameters that affect the sealing performance. Dwell time and jaw temperature are related. Lower dwell time needs higher jaw temperature to obtain similar performance. Otherwise, longer dwell times should be need for lower sealing temperatures. Jaw pressing time on packaging industry is time in which the sealing jaws are contact with the film [2].

**Jaw Temperature:** Heat sealing temperature is so critical throughout the sealing operation as well as in the end use. Time and temperature influence the fusion of the seal along sealing process. This is important for flow, diffusion and wetting. BOPET and BOPP, which are bi-oriented stretched films, tend to shrink when the temperature rises above the thermal setting temperature. Made from other materials films may melt and shrink. As the temperature of the polymer increases, its viscosity decreases. During the day, the temperature may change depending on the working speed of the packaging machine for various reasons. Another factor affecting the thermal adhesion strength is the temperature to which it is exposed during use. If the temperature acting on the packaging machine during filling and the application temperature of the retort is close or higher than the melting temperature of the heat seal material, it may put pressure on the heat seal area. Low temperatures can cause heat seal problems and embrittle the film [2].

**Jaw Pressing Force:** Pressure is necessary for the structures to be heat bonded to come into contact and for the sealing to take place. When working at high temperatures, excessive pressure may cause heat seal material to incarceration. For a good heat seal, sufficient pressure should be used, not high pressing force [2].

Aiyenger and Divecha [7] obtained a second order model for heat seal strength by experimentally working with the response surfaces methodology for heat seal strength optimization for biaxially oriented polypropylene film. The study gives a

quadratic model for the heat seal strength. As a result of this study, it has been determined that the pressing time, temperature and their interaction have important effects. It was determined that seal strength was acceptable at pressing time of 0.9s and temperature of 120 °C.

In the study of Farris et al. [8], the effects of temperature, jaw pressing time and jaw clamping force on the heat bond strength of biaxially oriented polypropylene films coated with a gelatin-based layer were investigated. As a result of the study, it was determined that the most effective factor on the thermal bonding force in the mathematical model obtained with the response surfaces methodology was the jaw pressure. Study showed the jaw clamping force negatively affected the thermal bond strength of the coated polypropylene films, while the jaw temperature had a positive effect. It was also concluded that pressing time as a main factor did not have a significant effect.

In this study, the most effective heat sealing process parameters for bonding force of the coated heat sealable 21  $\mu\text{m}$  BOPET packaging film to the APET structure surfaces are determined. A successful heat sealing can be guaranteed by controlling the heat sealing process parameters. In this examination, optimizability of the heat sealing strength is demonstrated by examining the effects of jaw dwell time, jaw pressing force and jaw temperature. Accordingly, a model was obtained for the heat seal strength. This study gives the result that jaw dwell time, jaw temperature and their interaction are important. It also gives the heat seal process parameter value for the highest heat seal strength that can be obtained according to the model.

## 2. Materials and Methods

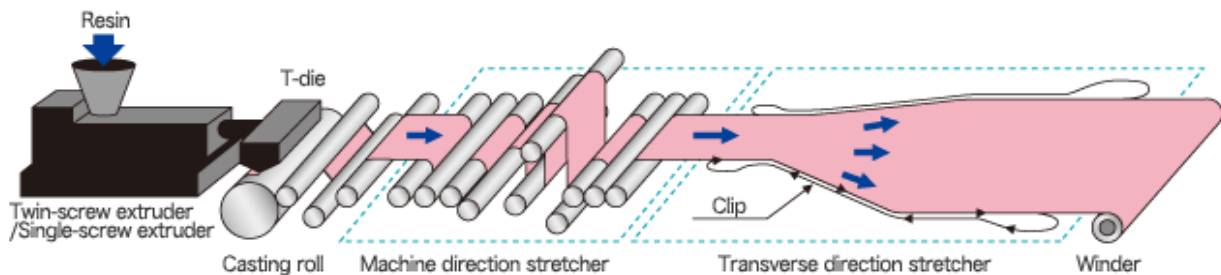
Flexible packaging BOPET 19 $\mu\text{m}$  film coated with  $2\pm 0.2$   $\mu\text{m}$  thick and  $2.5\pm 10$  gr/m<sup>2</sup> copolyester-based heat sealant on film surface by offline engraving coating method

in the surface coating machine. As shown in Figure 2, a heat sealable and peelable layer is applied to the polyester film by offline coating methods in an additional process step following the as Figure 1 traditional film production.

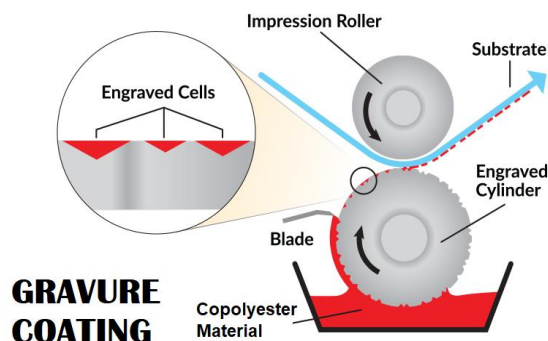
During coating process, heat seal copolyester material was diluted with ethylacetate and transferred to film surface with a gravure roller. After coating, the film surface entered dryer tunnel and ethylacetate was evaporated on the surface and a layer with APET surface sealable was formed on film surface. The production process was carried out based on study of Peiffer et al [3]. As shown Figure 3, final product obtained is used for foods filled in rigid containers and trays as a lidding in food packaging applications.

To review sealing performance of the surface coated biaxially oriented PET film with APET surface, heat sealing processes were carried out conforming to ASTM F88 - 00 standards with Brugger HSG-C device.

Thermal bonding was carried out with a singled well heated. For heat sealing process, 15 mm wide and 76 mm length strips were cut from the APET structure and surface coated film, and bonding was performed with a 10 mm wide thermal jaw. In order to analyze the influence of heat sealing parameters on seals strength, a total of three different heat sealing jaw temperatures (120 °C- 170 °C), three different pressure values (400 N – 600 N) and three different pressing times (0.5 s – 2 s) heating operations were performed combinations of processes. Llyod LS1 tensile strength measuring device was used to examine seal strength of the heat-sealed surface coated BOPET film and APET surface. Seal strength measurement was carried out according to ASTM F88-07 standards. ASTM F88-07 test method is used to measure the heat seal strength in packages. This test method determines the force required to separate the heat-sealed strip.



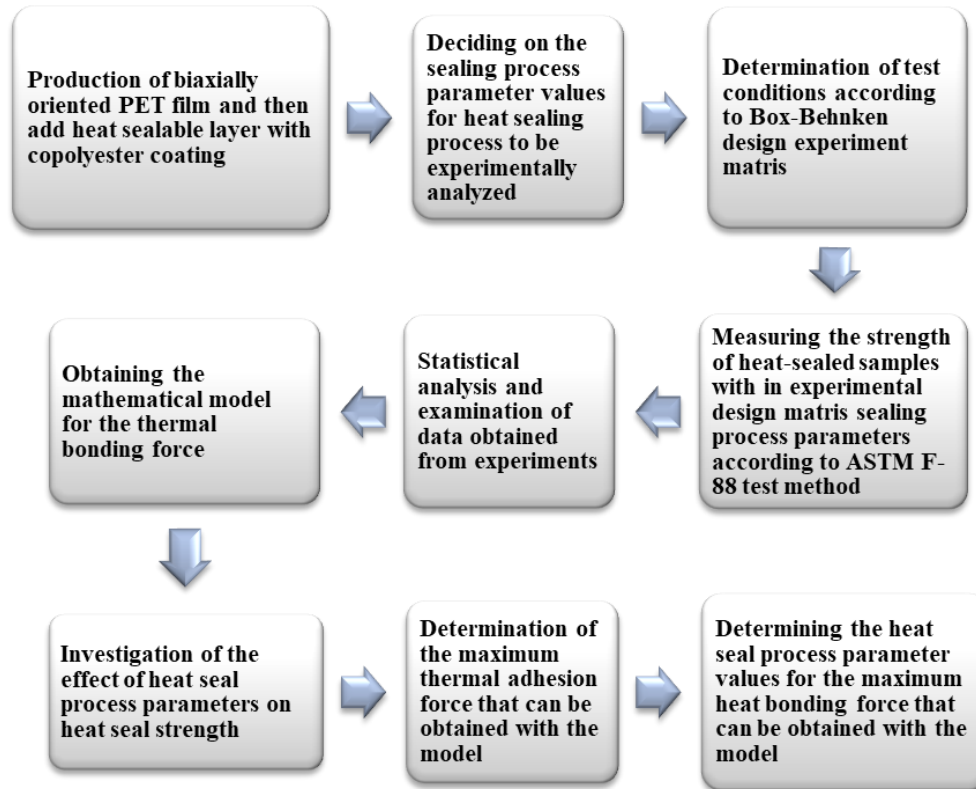
**Figure 1.** Biaxially oriented film production method



**Figure 2.** Offline film coating method



**Figure 3.** Heat sealable coated BOPET film food packaging application



**Figure 4.** Flowchart of the study

Testing steps:

- Calibrate the test machine.
- Prepare samples as specified in ASTM F88 test standards. The edges should be smooth and perpendicular to the heat seal direction.
- Place the samples in the tester. The heat seal zone of the sample should be equidistant between the test device clamps. A distance of 25-50 mm should be left between the clamps. Before the test starts, the heat-sealed specimen should be left loose so that it is not exposed to force. The test loading speed should be between 200 and 300 mm/min [9].

The flowchart summarizing the study is given in Figure 4.

For this study a Box-Behnken design (BBD) response surface (RSM) was selected. This method contains analyzing systematical, simultaneous, and effective variation of critical parameters. Jaw heat sealing temperature ( $x_1$ ), jaw heat sealing pressing

force ( $x_2$ ) and jaw dwell time ( $x_3$ ) were chosen to model the heat seal strength ( $y$ ), determine possible interplays, define higher order effects, and define optimum operating terms. Sealing temperature, dwell time and pressing force were studied at three different levels and sealing temperatures, according to Box-Behnken design (BBD), which was repeated with a total of fifteen process combinations. Each treatment combinations were examined three times and the average seal strength was considered.

The response surface analysis method is a series of experimental procedures applied to optimize the throughout value of a function with various detached unstable. The fundamental approximation is to motion in the direction of maximum increase or decrease to reach the local minimum or maximum value (optimum point) [10]. The designs which are generally used to reach the optimum point are central composite or Box Behnken experimental designs. These designs are second-order models with

quadratic terms, meaning that a link beyond the linear approximation can be expressed between the independent variables and the output value. The results obtained with these models are displayed graphically with contour drawings, which are called response surfaces, and the experimental designs used to estimate these values are called response surface designs [10].

The response surfaces methodology occurs of a set of statistical and mathematical techniques used in system optimization. Thanks to this technique, it is possible to explain the effects of independent variables, singly or in interaction, on the process [11]. The foundation of the response surfaces methodology is based on the measurement of response depending on k number of independent variables (x) of any physical system. This situation can be expressed functionally as:

$$y = f(x_1, x_2, \dots, x_k) \quad (1)$$

In any experiment, discrepancy between observed  $\hat{y}$  value and expected y value is interpreted as the error of the system and is stated with  $\epsilon$ . In this case, equation to be used can be expressed in theoretical form as follows [12].

$$\hat{y} = f(x) + \epsilon \quad (2)$$

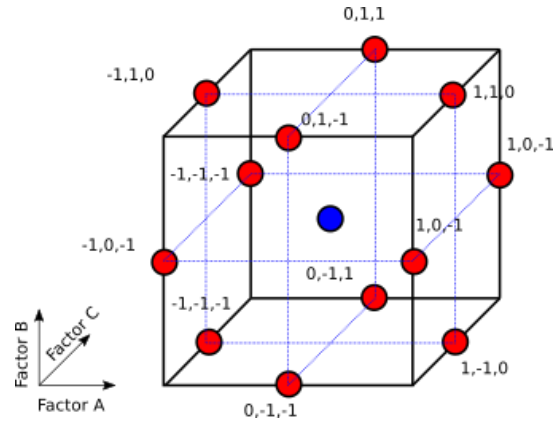
Response surfaces models are mostly quadratic (second order) or cubic (third order) polynomial form, which includes variables and their interactions [12]. Seal strength (y), is modeled as a higher order response surface in three dependent variables given with the equation:

$$y = \beta_0 + \sum_{i=1}^n \beta_i x_i + \sum_{i=1}^n \beta_{ii} x_i^2 + \sum_{i=1}^n \sum_{j=1}^n \beta_{ij} x_i x_j + \epsilon_0 \quad (3)$$

where y: Seal strength,  $\beta_0$ : the constant process effect,  $\beta_i$ : linear effect of  $x_i$ ,  $\beta_{ii}$ : the quadratic effect of  $x_i$ ,  $\beta_{ij}$ : the interaction effect between  $x_i$  and  $x_j$ .

Box-Behnken design is an efficient method for modeling quadratic response surfaces. Factors to be included in the model must have at least three levels. In the Box-

Behnken experiment, the value of one of the factors is fixed at the central value, while combinations of all levels of the other factors are applied [13].



**Figure 5.** Geometrically display of Box-Behnken design

Each factor, or independent variable, is placed at one of three equally spaced values, usually coded as -1, 0, +1. The operation range of the design is geometrically shown in Figure 5. In the Box-Behnken design the levels of the factors are at the midpoints of the edge and in the center point.

**Table 1.** Box-Behnken design experiment matrix

Trial No	Factors		
	A	B	C
1	-1	-1	0
2	1	-1	0
3	-1	1	0
4	1	1	0
5	0	0	0
6	0	-1	-1
7	0	-1	1
8	0	1	-1
9	0	1	1
10	0	0	0
11	-1	0	-1
12	-1	0	1
13	1	0	-1
14	1	0	1
15	0	0	0

**Table 2.** Design plan in coded levels

	-1	0	1
Temperature[°C]	120	150	170
Pressure [N]	300	500	600
Dwell time [s]	0.50	1.00	2.00

**Table 3.** Actual values of experiments

Trial No	Factors			Seal Strength [N/15mm]
	Temp. [°C]	Pressure [N]	Dwell time [s]	
1	120	300	1.00	2.57
2	170	300	1.00	4.33
3	120	600	1.00	2.50
4	170	600	1.00	4.83
5	120	500	0.50	2.10
6	170	500	0.50	4.10
7	120	500	2.00	2.17
8	170	500	2.00	4.83
9	150	300	0.50	3.00
10	150	600	0.50	3.53
11	150	300	2.00	3.50
12	150	600	2.00	3.93
13	150	500	1.00	3.67
14	150	500	1.00	3.67
15	150	500	1.00	3.67

Box-Behnken matrix is presented in Table 1. Coded design plan of three independent variables is presented in Table 2, with corresponding actual values as tabulated in Table 3.

Results were analyzed using MINITAB statistical analysis software. They are disputed and commented in the following part. Seal strength values shown in the Table 3 is the three test average.

**3. Results and Discussion**

Test results of fifteen run repeated Box-Bhenken design for optimization of seal strength of 21µm coated sealable BOPET film are shown in Table 3. It presented seal strength values corresponding to combined effect of three process parameters in their specified ranges, heat sealing temperature: 120–170 °C, jaw dwell time: 0.5–2 s, and jaw pressing force: 400–600 N.

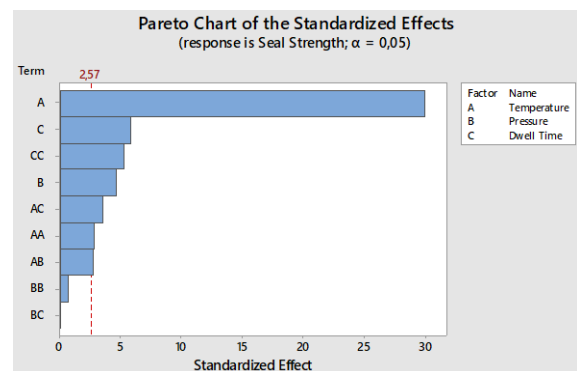
The model obtained for seal strength using the reaction surface methodology and Box-Behnken design approach is expressed in following equation with the coded factors.

$$y = -5.06 + 0.0861x_1 - 0.00587x_2 + 0.359x_3 - 0.000242x_1^2 + 0.000002x_2^2 - 0.579x_3^2 + 0.000038x_1x_2 + 0.00954x_1x_3 - 0.000025x_2x_3 \tag{4}$$

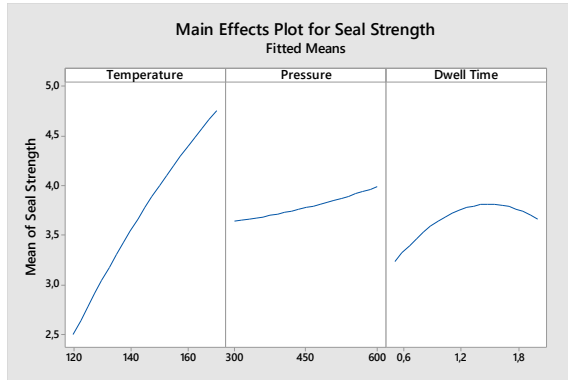
where y: seal strength, x1: temperature, x2: pressure, x3: dwell time.

The heat seal strength in defined combination of three variables can be estimated with equation (4). The heat seal model graphics which were prepared using equation (4) are shown in Figures 6-12. These graphs show the behavior of the heat seal force relative to synchronous changes in the two variables while keeping the third variable constant.

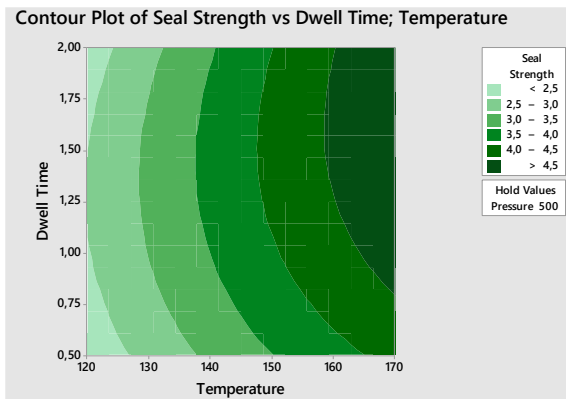
In the study, the correlation coefficient value (R2=0.995) was obtained as higher than 0.80 and it is seen that the model represents 99.5% of the experiments.



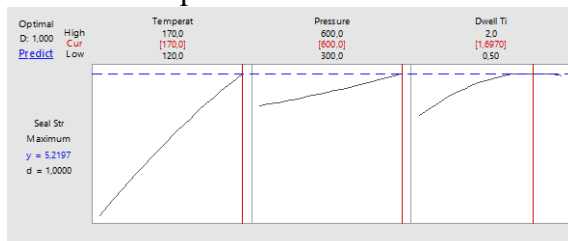
**Figure 6.** Pareto chart of the standardized effects



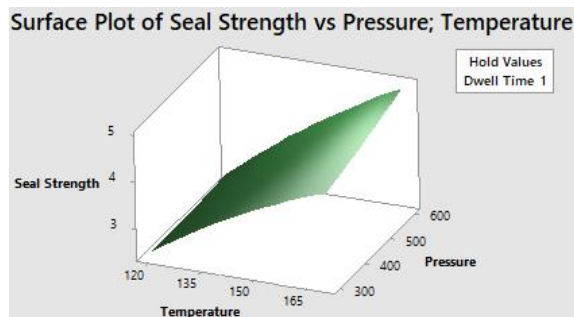
**Figure 7.** Main effects plot of seal strength



**Figure 8.** Contour plot of seal strength at varying temperature and dwell time a pressure of 500N



**Figure 9.** Response optimization for maximum seal strength



**Figure 10.** Surface plot of seal strength with varying temperature and pressure at a dwell time of 1s

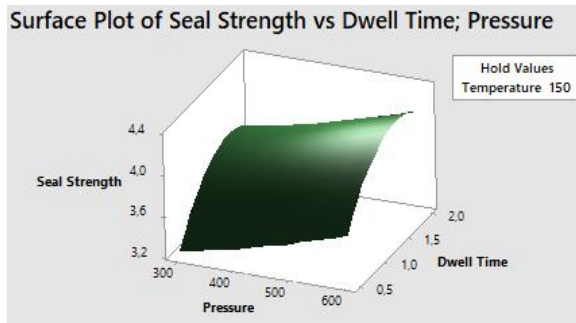
When looking at the Pareto chart to determine the magnitude and significance of the effects, the bars crossing the baseline are statistically significant. The bar representing the temperature factors in the Pareto chart in Figure 6 crosses the reference line. Temperature, pressure, dwell time factor is statistically significant at the 0.05 level with current model terms. In the main effect graph, it is seen that the effect of the temperature factor is more than the other factors.

Box-Behnken design and response surface method not only ensured model for heat seal strength in conditions of heat sealing temperature, jaw dwell time, and jaw pressuring, but also several choices of three variable combinations yielding high seal strength 4.0-4.5 N/15mm. This is not possible with traditional factors at time experiments.

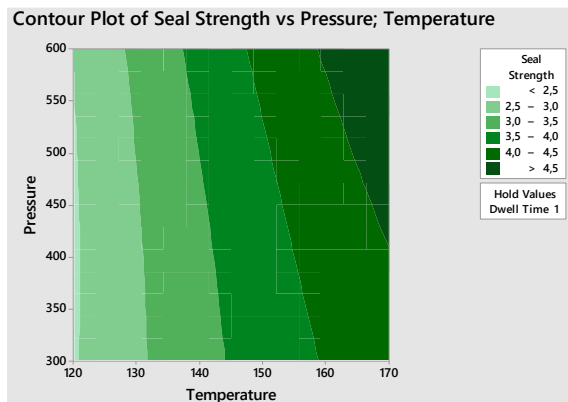
Contour plot (Figure 8) of dwell time and temperature at fixed pressure, 500N, shows that we get high seal strength in the range of 4-5 N/15 mm at higher temperature and dwell time. At a fixed pressure, 500N, a high seal strength of 4.5 N/15mm is available alternatives; at lower than 1s dwell time with temperature above 160°C as well as at 2s high dwell time with temperature between 150°C and 160°C. At a very low dwell time and low temperature seal performance is not sufficient, hence seal strength varies considerably.

Considering the application of final product, a high thermal adhesion force for the packaged food will provide better sealing and better protection. On the other hand, high thermal adhesion force means that it can tolerate contamination and temperature changes in the heat sealing jaw during filling, which positive in terms of performance.





**Figure 11.** Surface plot of seal strength with varying pressure and dwell time at a temperature of 150°C



**Figure 12.** Contour plot of seal strength at varying temperature and pressure with a dwell time of 1s

In this study according to the model, the highest seal strength 5.2197 N/15mm is obtained at a combination 170°C temperature, 1.7 s dwell time, and 600N pressure.

Figure 10, the plot of seal strength as a function of sealing temperature and pressure with dwell time constant at 1s, exhibits the linear behavior of temperature and pressure variables. It is observed that the seal strength surface attains the maximum height at 170° C temperature.

Figure 11, the plot of seal strength as a function of pressure and dwell time with temperature constant at 150°C, shows that seal strength increases rapidly with increased dwell time, up to 1.5 s and after that there is no major change in the seal strength value. Figure 11 exhibits the quadratic behavior of time and temperature variables.

Contour plot (Figure 12) pressure and temperature at fixed dwell time, 1s, shows that we get higher seal strength at high temperature and pressure. At a fixed pressure, dwell time 1s, high seal strength (higher than 4.5 N/15 mm) is available at higher than 400 N pressure with temperature above 160 °C.

Response surface methodology serves idea of getting higher seal strength with temperature range 150 °C - 170 °C and middle level dwell time 1–2 s.

#### 4. Conclusions

In this study, the parameters that affect bond strength of heat sealable coated BOPET film in food packaging industry were investigated and these parameters were optimized using systematic experimental design methods. In thermal adhesion analysis; the effects of heat seal temperature, dwell time and jaw compression force (pressure) on the seal strength obtained were made into a mathematical model using Box-Behnken design (BBD), a response surface method (RSM). After modeling of thermal adhesion parameters, optimization study and reduced model study were carried out. The experimental results of the model and the reduced model for the seal strength were compared with the estimated values within the 95% confidence interval. The model obtained in the study represents 99.5% of the experiments. It has been determined that the closeness of the experimental results to the estimated values, the Box-Behnken design model is an effective method in modeling the thermal adhesion force parameters, which is a complex process.

The heat seal performance of surface coated heat-sealable BOPET film can be optimized by obtaining a model using experiments involving changes in heat sealing temperature, jaw pressing force, and dwell time. In this study, three-level and two-factor Box-Behnken experimental design was used. The jaw temperature, jaw pressing force and dwell time values that give the

desired heat seal strength of the heat-sealable BOPET film were determined. Suggested optimal process combinations could offer cost improvements in high sealing temperature and dwell time.

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