

Determination of The Best Injection Stretch Blow Molding Process Parameters in Polyethylene Terephthalate Bottle Service Performance

Bilal DEMIREL¹, Fatih AKKURT^{2,*}, Ipek Adeviye UYSAL¹, Ercan SENYIGIT ³

¹ Erciyes University, Department of Material Science and Engineering, 38039 Kayseri, Turkey.
 ² Gazi University, Department of Chemical Engineering, 06570 Ankara, Turkey
 ³Erciyes University, Department of Industrial Engineering, 38039 Kayseri, Turkey.

Highlights

• The top load strength of the final bottle was associated with the stretch rod movement

• The panel (body) weight is directly and seriously affected by the preform surface temperature.

• The final pressure and stretch rod also affected the body weight by bilateral interaction.

• GRA results (except for Stress Crack Resistance) are greater than the general results of Taguchi.

• The general result of Stress Crack Resistance with Taguchi is greater than GRA.

Article Info Abstract Polyethylene terephthalate (PET) bottles which are thermoplastic materials are used very commonly for the storage and transport of carbonated beverages. The most used production Received: 16 Mar 2020 method for PET bottles is the Injection Stretch Blow Molding (ISBM) process. There is a variety Accepted: 28 Dec 2021 of parameters affecting the produced PET bottles' performances. Amongst these parameters, stretch rod movement, blowing pressure and preform surface temperature are the most important Keywords ones. Assignation of the optimal design parameters in PET bottles is taken into account. The effects of the parameters such as Preform Temperature (°C), Stretch Rod Position (mm) and Final PET Pressure (Bar) were analysed with the Taguchi method (TM), Grey relational analysis (GRA) and ISBM Taguchi ECHIP. Body-weight (gr) (R1), Top Load (Pa) (R2), Burst Pressure (Bar) (R3), Stress Crack Grev relational Resistance (Min.) (R4) and Tg (oC) (R5) were considered as performance parameters. The analysis experimental design proposed by Taguchi involves using orthogonal arrays. An L9 orthogonal array was chosen for the procedure. Primarily, the performance parameters were optimized with the ECHIP Design of the Experiment (DOE). Thereafter, all of the factors were optimized together with TM, GRA and ECHIP.

1. INTRODUCTION

The bottles made of polymeric materials have some superior advantages over packaging materials made of glass and metal [1]. Polyethylene terephthalate (PET) bottles are among the most utilized polymeric material for Carbonated Soft Drinks (CSD) packaging because of its surpassing physical, mechanical and barrier properties [2]. Furthermore, that the densities of metals are higher than the PET material is a basis for the preference by bottle producers.

Bottles produced with PET usually are fabricated with the ISBM process. In this process, the pre-formed PET tubes are blown into the desired bottle-shaped mold and the preformed tubes are distorted in two directions axially by the stretch rod and radially by the internal pressure. There are two types of blow pressure in the ISBM process. The first type, which has a much lower pressure value than the second type (known as final pressure), is called pre-blow pressure. While pre-blow with low pressure forms a large part of the bottle, the final blow forms complex specification such as the petaloid base with the application of a higher pressure [3].

In the ISBM parameters, the preform surface temperature, timing of the mold, relationship between stretching and blowing according to time, stretch rod movement, blowing pressures and characteristics of PET are among the parameters that affect the final product quality. However, in a literature search, there is not much work on process parameters affecting the final bottle properties [4-8].

The preform surface temperature, which can be defined as the final performance of the bottle, is one of the basic process parameters [9]. In order to produce a quality bottle, the surface temperature profile, blowing pressure and the stretch rod movement should be kept under check during the stretching and inflation process [5].

Crystallinity is a very important physical property of PET and usually is induced in two ways. One is called thermal crystallization and the other is stress or strain-induced crystallization. If PET is heated above the Tg and not kept satisfactorily at those conditions, then thermally induced crystallization occurs, and the colour of PET turns opaque because of the spherulitic structure formation caused by the thermal crystallization behaviour of the undirected PET bonds [10]. In the stress-induced crystallization process, orientation and stretching are enforced to the heated PET preform, and the chains are reformed in parallel and are in nearly packed form [11]. When the preform surface temperature rises above the Tg and falls below the Tm, spherulitic crystallization and nucleation occur and some hazy regions form on the bottle surface [12]. Conversely, if melt quenching is applied quickly, PET chains are aligned in an entirely amorphous phase [13].

During stretching and blowing, the temperature control of the surface, the pre and final blow pressures and the sequence of the stretch rod movement are crucial [5]. Preform reheating is ensured by infrared radiation provided by a series of lamps. The preform surface temperature generally ranges between 90-115°C; this is the preferred range in trade and prevents potential haziness on the final bottle surface. The axial temperature profile of the pattern surface should be hold under check since the upper and lower region should be colder than the chassis.

In the ISBM process, the bottom of the preform may be deformed or ruptured by the stretch rod due to overheating. On the other hand, the body temperature profile of the pattern can change due to a decrease in wall thickness. Furthermore, when the preform surface temperature is incorrectly defined, too much or too little material may reside in the body or bottom of the bottle [14]. Furthermore, the temperature profile of the preform is of significant importance for the last form of the petaloid ground [7].

Based on their experiments with PET, under or near glassy transition temperature, Lebaudy and Grenet aimed to find the preform surface temperature and crystal structure of the preform to obtain a principally oriented glassy structure (Tg) [15].

It has been found that lowering the reheating temperature increases the top-load capacity for light preforms as well heavy preforms. However, the highest base clearance is obtained at low reheat temperatures for lightweight preforms and at higher reheat temperatures for heavyweight preforms [16].

Before stretching, the temperature interval for the region between the neck and bottom of the preform is 15-20°C [4]. It is also very substantial in terms of transparency and pattern on the bottle. Higher blasting may be required for lower temperatures and vice versa.

Preform blowing strongly depends on design and temperature distribution. The hot zones of the sample are blown faster and the bottle becomes slimmer. However, the stiffer and colder parts of the preform are blown slower and the bottle then becomes thicker [5]. Monteix et. al. compared the results of simulation and experimental study of a preform that was subjected to a heating process by infrared radiation [9]. As a result, it was found that the final thickness of a bottle depends on the primal temperature distribution of the preform in the reheating step.

The necessary stress hardening of the side walls of the bottles is provided by a linear stretch rod, thus providing a constant wall thickness, as well. Comparing the flow of the stretch rod and the pre-blowing

pressure, the pre-blow pressure must be very small; otherwise, the stretch rod movement does not occur correctly. In addition, the preform end cap can move away from the center of the bottle mold and cause an uneven wall thickness [4]. The reason why the bottle base is stretched slightly is that the temperature range in this section is higher than that of the upper region. At this stage, the stretch rod may adhere to the bottom of the bottle. Therefore, the bottom of the bottle is weaker with regard to tensile crack resistance. When the stretch rod approaches the bottom of the bottle mold, the preform initially is stretched by the pre-blow pressure and then by the ultimate-blow pressure.

As a result, compression might take place at the bottom of the bottle [6]. Given typical graphs showing the time-displacement of the stretch rod, preform blowing time can be simulated because it is efficient for ISBM [4].

In existing work, it was aimed to investigate the influence of preform surface temperature and stretching rod movement on the performance of the final product by using different experimental design methods such as ECHIP-7 and TM. In the design of the experimental section, the response surface method, which is a module in the statistical analysis software of ECHIP-7, was used. First, the output and input variables of the method were assigned, and then the partial cubic design method was used in the ECHIP software. Design outputs were selected from mechanical performance and morphological values. The mechanical performance criterions are top-load, burst pressure and environmental stress cracking while the morphological values are Tg and crystallinity. A 34 g preform with various surface temperatures was vertically stretched with the stretch rod with different positions and blown into a 1 litre CSD bottle mold using a custom made ISBM machine. Finally, to evaluate the changes occurring on the final bottle, the produced 1 litre bottles were tested both mechanically and physically. The data obtained was analysed by both ECHIP and TM experimental methods in order to determine the best process parameters.

The Taguchi (TM) is an experimental design (ED) procedure which is attributed to the analysis of variances, which starts an enterprise to uncover the main causes of variations experiments (Es) [17]. TM was developed by Taguchi [18-28]. TM, prevalently operated in engineering analysis, is a robust facility to lay out system parameters [20-23]. TM parameter design stage was carried out and the details of TM stages can be read in study 4. TM is based on an analysis of variances (ANOVA). TM was fulfilled with Minitab Version 16.0. TM exploits the orthogonal arrays (OAs) for ED, which may bring down the number of Es, and signal-to-noise ratios (SNRs). SNR is a performance criterion. Nonetheless, in TM the parameter levels chosen to generate the OA are separate and therefore the true optimum can only be gained from the present parameter levels [24]. As SNR increases, the grade of product developes. The main goal is to maximize the SNR [25, 26, 28]. Three categories of performance characteristics come to the fore in SNR analysis. These are "higher is the better", "nominal is the best", and "lower is the better". The equations of these characteristics can be found in studies 17-25. There are six steps in the TM. In the initial stage, the objectives are determined. In the latter stage, the design parameters and their levels are specified. In the next phase, the performance characteristics of parameters are chosen. The fourth range includes the choosing of convenient OA. The subsequent stage is conducting the experiments and calculation of SNR values. Confirmation studies are conducted in the last stage [25]. This completes the implementation of the TM. Finally, we get optimum design parameter values. It can be said that the Taguchi is very effective in engineering at the point of production. [26, 27, 28].

2. EXPERIMENTAL

2.1. Material

In this study, K-084 grade PET bottle resin was bought and used from the company (Köksan Plastik) with an intrinsic viscosity of 0.84 dl/g and a melt density of 1.2 g/cm³.

2.2. Design of Bottle Mold and Preform

In this study, a preform of 34 gr and a PET bottle mold of 1 litre were used and the corresponding schematic representations are presented in Figures 1 and 2, seriatim.



Figure 1. Bottle design used in the study



Figure 2. Preform lay out

2.3. Injection Molding Process

An ISBM process with two stages was utilized for the production of the bottles used in this study. The preforms of 34 g were manufactured by using an ISBM machine the details of which are seen in Table 1.

2.4. ISBM Process Parameters and Reheating Unit

The ISBM machine with two stage manufactured by a local company was used in this work. The process settings are presented in Table 1. The heating process of the preforms in the oven with 8 IR lamps was operated from a 0 to 10 range, relating with the IR lamps' location. The temperature of the samples were between 30 and 35 $^{\circ}$ C along their length. The preform surface temperature, final blowing pressure and stretch rod movement were determined based on the Design of Experiment (DOE) program. A thermal camera image obtained from the preforms removed from the oven is given in Figure 3. Other process parameters were kept constant. In addition, the ISBM process parameters applied during the production are

given in Figure 4. The pre-blow pressure was pegged at 0.9 bar for 2.5 seconds before the final-blow pressure was gradually increased to the last pressure defined by the DOE program at 2 second intervals. The stretch rod speed was adjusted to 0.75 m/s, so it reached the bottle groung in 0.375 seconds.

Table 1. Working details

	SCREW	
Diameter (mm)	55	
Screw speed (rpm)	0-1	65
Nozzle Diameter (mm)	8	

INJECTION PRESSURE

Primary (MPa)	140.7
Injection rate (g/s)	192
Shot volume (m ³)	5x10-4

STRETCH BLOW MOLDING

Cold preform temperature (°C)	20
Pre-blow (MPa)	0.09
Stretch Rod Speed (m/s)	0.75
Stretch rod outside diameter (mm)	12

	HOT RUNNER BLOCK (°C)		
Sprue	275		
Block	275		
Nozzle	280		

	BARREL TEMPERATURE (°C)			
Front	270			
Middle	280			
Rear	280			
Nozzle	275			



Figure 3. Preform surface temperature profile



Figure 4. Process conditions of stretch blow molding

In the study, the preforms were subjected to ISBM processing immediately after reheating. The mold temperature was kept constant at about 10 °C during the blowing process. The limit values given in Table 2 are described for process limits. The experimental design and details of the DOE program are presented in Table 3.

Design Parameters	Minimum	Maximum
Preform Temperature (°C)	100	115
Stretch Rod Position (mm)	0	250
Final Pressure (Bar)	15	25

Trial	Preform Surface	Final Pressure	Stretch Rod
Number	Temperature	(Bar)	Position
	(°C)		(mm)
11	105-110	20	0
6	105-110	15	250
9	105-110	15	0
12	110-115	20	250
5	110-115	15	0
3	110-115	25	250
28	100-105	15	250
30	110-115	25	150
18	105-110	25	250
21	100-105	20	250
12	110-115	20	250
29	100-105	20	0
15	105-110	15	150
16	105-110	20	150
10	110-115	25	0
22	100-105	25	250
1	100-105	15	0
19	105-110	20	250
24	110-115	20	150

17	110-115	15	250
8	100-105	15	150
23	110-115	15	150
1	100-105	15	0
7	100-105	25	0
13	100-105	20	150
2	105-110	25	0
20	105-110	25	150
14	110-115	20	0
15	105-110	15	150
4	100-105	25	150
20	105-110	25	150
2	105-110	25	0

2.5. Material Distribution

To weigh the material distribution in the bottle, the produced bottle samples were divided into three parts (base, body and top) via a particular hot wire cutter. Weighing of the parts of the bottle was done on a precision scale and the outputs were recorded.

2.6. Burst Strength

Samples were filled with CSD having a pressure of about 7 bar. It is very important to pack the carbonated beverages in the filling stage to prevent the explosion of the bottles and over-expansion of the filling bottles along storage and/or along heating of the bottle for pasteurization. There are several components affecting burst strength. Thin wall thickness based on poor material distribution, loss of mold compensation originating from wide mold parting lines and mold damage are some. The burst strength test was performed by means of a custom-made Plastics Burst Tester. For samples identified by the DOE program, at least three bottles were tested to procure an average value.

2.7. Top-Load Strength

This parameter is an indicator of the whole endurance required to perform and impute bottles during manufacture, storage and distribution. The tests were performed with a Shimadzu tensile tester equipped with a top-loading unit. The above tests were carried out at thereabout 23 ° C and atmospheric pressure. At least three bottles were tested to obtain an average value. To perform the test, the bottle was placed in the device, and then a compression load of 2.5 N / sec was practiced to the upper region of the bottle. The load was set as the highest when the bottle reached the first buckling point [29].

2.8. Glass Transition Temperature (Tg)

The crystallinity and Tg values of the patternswere measured by Differential Scanning Calorimetry (DSC) to interpret the reasons for the differences in mechanical analysis. The crystallinity degrees were calculated using Eq. (1) regarding the heat of fusion of 100% crystalline PET (ΔH_f^0) to be 135 J/g [30]. ΔH_m and ΔH_c are the enthalpy of melting and crystallization, respectively.

$$X(\%) = \frac{(\Delta H_m - \Delta H_c)}{\Delta H_f}.$$
(1)

2.9. Environmental Stress Crack Resistance

In this step, an accelerated stress-crack test unit (ASCRU) with a solution of sodium hydroxide (NaOH) 0.2 %) was used. Test results were recorded. Information on the standard test method can be acquired from the ASTM D 2561 [29].

2.10. Experimental Design and Statistical Analysis

An experimental design was set up to specify parameters that considerably affect the performance of the final product's service by using a statistical analysis program called ECHIP-7 (ECHIP Inc. USA). The preform surface temperature, stretch rod movement and final-blow pressure were assigned as design variables. The limit of each parameter was determined according to the processing capability of the bottles (Table 2). Based on the partial cubic method, maximum top load resistance, burst pressure, environmental stress cracking, body weight and Tg values were taken according to DOE. 32 experiments were identified by ECHIP-7 based on standard deviations of 0.1 sec. and at least a significant difference of 0.3 sec. All output variables were loaded as response variables to the program and modelled with a partial cubic model. Therefore, the experimental values as a whole were set as a function of the process parameters to evaluate the dependence of the output variables on the input variables. In addition, burst pressure, top load, body weight, environmental stress cracking resistance and Tg values were considered as output variables.

3. RESULTS AND DISCUSSION

The results obtained according to the ECHIP-7 DOE program are given in Table 4.

After entering the obtained numerical values into ECHIP-7 as input variables, statistical analysis done and the obtained results are summarised as follows.

- It was found that the top load strength of the final bottle was associated with the stretch rod movement. However, as can be seen from Table 5, this is not just about stretch rod movement. Parameters such as preform and bottle mold design, mold cooling temperature and stretch rod acceleration also affect the top load strength [31]. In the experimental study, buckling usually occurred in the body part of the bottle. Therefore, body weight directly affected the top-load resistance.

Trial No	Body Weight (gr)	Top Load (Pa)	Burst Pressure (Bar)	Stress Crack Resistance (min)	Tg (°C)
11	12.21	11.9	6.84	25	76.26
6	12.36	203.75	10.99	0	92
9	15.58	73.66	10.68	180	92.3
12	11.51	174.92	9.28	60	102.62
5	11.62	95.1	9.09	15	72.66
3	12	222.97	9.04	0	85.88
28	13.8	196.7	5.74	0	33.5
30	12.22	85.7	10.04	80	67.4
18	11.69	203.58	5.73	180	88.19
21	13.41	116.8	4.41	0	88.9
12	11.51	174.92	9.28	60	102.62
29	13.56	98.91	12.06	5	92.9
15	12.42	77	11.02	30	62.3
16	12.98	96.4	11.11	885	70.3
10	12.26	86.26	9.24	15	116.18

Table 4. Numerical values of the output variables

22	12.97	206.5	6.24	0	84.52
1	14.38	37.76	12.27	105	65.96
19	13.03	241.6	4.34	0	57.3
24	12.35	97.98	10.16	180	94.9
17	11.13	163.87	9.03	25	84.18
8	13.36	73.74	12.23	0	88.75
23	11.61	84.64	9.49	21	84.6
1	14.38	37.76	12.27	105	65.96
7	14	70.9	11.8	10	76.2
13	13.68	97.74	9.27	100	78.13
2	11.97	154.6	9.66	61	87.13
20	12.31	120	10.48	12	83.25
14	12.02	104.66	9.69	60	92.2
15	12.42	77	11.02	30	62.3
4	13.31	87.75	10.16	122	84.78
20	12.31	120	10.48	12	83.25
2	11.97	154.6	9.66	61	87.13

Table 5. Summary results of the experimental data

Project Summa	Project Name: Definition of ISBM Process Parameters Summary Results by sources									
TOP LOA	BURST PRESSUR	GLASS TEMPERATUR	PANEL WEIGH	STRESS CRACKIN						
D	Ε	\mathbf{E}	Т	G						
			XX		TEMPERATURE					
Х	Х			XX	STRETCH ROD					
			X		TEMPERATURE*FINA					
					L PRESSURE					
			Х	Х	STRETCH ROD* FINAL PRESSURE					

1) It was found that the burst pressure strength also depends on the stretch rod movement. However, this property is not only related to the stretch rod movement. In experimental studies, the bottles subjected to burst pressure testing were found to break at the intersection of the bottom and panel sections of the bottle [19]. Since the movement of the stretch rod is also effective in weight distribution, it has also been effective like preform surface temperature and preform design in this test as well.

2) In this study, it was not possible to establish a clear relationship affecting the glass transition temperature. A more detailed study is required to clarify this phenomenon, as the glass transition temperature is influenced by parameters such as stretching rod movement, preform surface temperature, mold temperature, application pressure and preform storage time after molding [6, 32].

3) It was seen that the panel (body) weight is directly and seriously affected by the preform surface temperature. It was also found that the final pressure and stretch rod also affected the body weight by bilateral interaction. When looking at Table 4 carefully, it can be seen that body weight decreases as preform surface temperature increases. However, the body weight decreased with increasing stretch ratio, but increased with increasing final-blow pressure (Table 6).

		a		
Trial No	Preform Surface	Final Pressure	Stretch Rod	Body Weight
	Temperature	(Bar)	Position	(gr)
	(°C)		(mm)	-
21	100-105	20	250	13.41
19	105-110	20	250	13.03
12	110-115	20	250	11.51
		b		
Trial No	Preform Surface	Final Pressure	Stretch Rod	Body Weight
	Temperature	(Bar)	Position	(gr)
	(°C)		(mm)	
17	110-115	15	250	11.13
12	110-115	20	250	11.51
3	110-115	25	250	12.00
		c		
Trial No	Preform Surface	Final Pressure	Stretch Rod	Body Weight
	Temperature	(Bar)	Position	(gr)
	(°C)		(mm)	
10	110-115	25	0	12.26
30	110-115	25	150	12.22
3	110-115	25	250	12.00

Table 6. Variation of the bottle body weight by the preform surface temperature (a), final pressure (b) and stretch rod position (c)

4) As can be seen from Table 7, the results (R2=0.85, P=0.0001) from the ECHIP-7 statistical analysis program for body weight are meaningful and within the acceptable limits. These values indicate that the output from the program for the final bottle body weight is reliable. Since all input variables used in the study were categorical, no equation could be given in this study.

Centered continuous variables								
Coefficients	Coefficients SD P Condition Term							
127.31				0	Constant			
-0.31859	0.233793	0.1908	0.975	1	Final-Pressure			
-0.018663	0.0830939	0.8250-	0.982	2	Final-Pressure ²			
0.0238095	2.4748	0.9924-	0.462	3	Temperature (105-110)			
-8.38095	2.33326	0.0022	0.512	4	Temperature (110-115)			
-1.71429	2.33326	0.4725-	0.500	5	Stretch-Rod (Bottom)			
3.02381	2.4748	0.2384	0.461	6	Stretch-Rod (Middle)			
-1.03513	0.316893	0.0045	0.847	7	Temperature (105-110)^^Final- Pressure			
1.05192	0.343764	0.0071	0.864	8	Temperature (110-115)^^Final- Pressure			
0.0852564	0.343764	0.8071-	0.831	9	Stretch-Rod (Bottom) ^^Final- Pressure			
0.652372	0.321526	0.0584	0.831	10	Stretch-Rod (Middle) ^^Final- Pressure			
-0.0139267	0.117539	0.9071-	0.457	11	Temperature (105-110)^^Final- Pressure^2			
-0.0184799	0.11592	0.8752-	0.512	12	Temperature (110-115)^^Final- Pressure ²			

 Table 7. Statistical analysis results for the bottle body (panel) weight

 Coefficients for response "PANEL-WEIGHT"

-0.0784799	0.11592	0.5075-	0.495	13	Stretch-Rod (Bottom) ^^Final- Pressure^2			
-0 196427	0.118045	0 1 1 4 4	0.453	14	Stretch-Rod (Middle) ^^Final-			
-0.190427	0.110045	0.1144	0.433	17	Pressure ²			
N trials	= 32							
N terms=15								
Residual SD	=5.346174							
Residual DF	=17							
Residual SD u	sed for tests							
Replicate SD	=0.000000							
Replicate DF	=5							
Cross val RMS	5 =8.130272							
R Squared	=0.852,	p=0.000	1 ^^^					
Adj R Squared =0.731								
Maximum Cook-Weisberg LD influence (scaled 0-1) =1.000								
Maximum absolute Studentized residual $=3.516 p = 0.0002$ ^^^								
- This te	erm may be eli	minated						

5) It was observed that the top-load strength value is related to the final-blow pressure and increased with increasing final-blow pressure. For the top-load resistance, there is no direct univariate relationship with the input variables. Therefore, for the top-load resistance, it can be said that the statistical analysis results having R and P values of about 0.77 and 0.0043, respectively, are reliable (Table 8).

 Table 8. Statistical analysis results for the bottle top-load strength

Coefficients for response "TOP-LOAD" Centered continuous variables

Coefficients	SD	Р	Condition	Ter	m
114.833				0	Constant
3.62019	1.79934	0.0603	0.975	1	Final-Pressure
0.514295	0.639515	0.4324-	0.982	2	Final-Pressure ²
1.83333	19.0468	0.9244-	0.462	3	Temperature (105-110)
8.33333	17.9575	0.6485-	.512	4	Temperature (110-115)
60.3333	17.9575	0.0037	.500	5	Stretch-Rod (Bottom)
-17.5	19.0468	0.3711-	0.461	6	Stretch-Rod (Middle)
3.15885	2.43867	0.2125	0.847	7	Temperature (105-110)^^Final-Pressure
-1.92019	2.64571	0.4778-	0.864	8	Temperature (110-115)^^Final-Pressure
-1.32019	2.64571	0.6242-	0.831	9	Stretch-Rod (Bottom) ^^Final-Pressure
-1.8849	2.47456	0.4567-	0.831	10	Stretch-Rod (Middle) ^^Final-Pressure
0.824897	0.904617	0.3746-	0.457	11	Temperature (105-110)^^Final-Pressure^2
-0.514295	0.892157	0.5719-	0.512	12	Temperature (110-115)^^Final-Pressure^2
0.459038	0.892157	0.6135-	0.495	13	Stretch-Rod (Bottom) ^^Final-Pressure^2
-0.997186	0.908507	0.2877	0.453	14	Stretch-Rod (Middle) ^^Final-Pressure^2
N trials	= 32				
N terms=15					
Residual SD	=41.145727				
Residual DF	=17				
Residual SD u	sed for tests				
Replicate SD	=31.306549				
Replicate DF	=5				
Cross val RMS	5 = 59.590627				
R Squared	=0.765,	p=0.0043			
Adj R Squared	1 =0.572				

Maximum Cook-Weisberg LD influence (scaled 0-1) =0.639 This term may be eliminated

6) Finally, Environmental Stress Crack Resistance (ESCR) was affected by the stretch rod movement. However, it was observed that final-blow pressure also affects ESCR. In this study, ESCR tests were applied only to the base section of the bottles because stress cracks occur only in this region. Therefore, the base weight, which means the bottle base thickness, was a determinant. There are some studies in the literature showing that crystallization also has an effect on ESCR [32]. The factors affecting thickness must also affect the ESCR. However, there are other factors affecting the ESCR besides thickness. For example, crystallisation and biaxial orientation are factors affecting ESCR and there are some studies in literature related to this phenomenon [16].

In the study, the process parameters affecting the bottle performance were determined by means of the ECHIP program but since some of the process parameters were categorical parameters, the optimum process parameters could not be determined. For this reason, the Taguchi and Grey relational analysis (GRA) methods were used.

There are three parameters which have three stages in this work. The degrees of freedom (DOF) are the sum of one less of the number of each factor levels [25,28]. Moreover, six DOF were designated to three parameters (3×2) . When the required DOF was known, an convenient OA was specified to suit the specific task. The number of experiments for OA should be equal to or greater than the DOF, L9 was appropriate for this study. For 3 parameters at 3 levels each, It would need to traditional full factorial design 3³ (27) experiments. This plan cut down on 27 experiments to 9 assessments. This is significant benefit of TM [28]. Table 9 shows the parameters and their values related to their levels. All of the experiments were carried out with the experimental plan in Table 10. The contribution ratios of all factors of the performance benchmark were described depending on the SNR, as is seen in Tables 11-15. The ideal values of the factors were identified by scaling up the Body weight, Top Load, Burst Pressure, Tg and Stress Crack Resistance as shown in Table 16. $A_1B_2C_2$ was specified as the optimum state of parameters in compliance with the "the higher is the better" situation for Bodyweight. A2B3C3 was determined as the best state of design parameters according to "the higher is the better" situation for Top Load. A₃B₃C₁ was described as the optimum condition of design parameters according to "the higher is the better" situation for Burst Pressure. A₃B₁C₁ was defined as the optimum condition of design parameters according to "the higher is the better" situation for Stress Crack Resistance. $A_2B_1C_3$ was defined as the optimum condition according to "the higher is the better" situation for Tg. The Final Pressure parameter is the most important parameter for Top Load, Burst Pressure, Stress Crack Resistance. Preform surface temperature is the most important parameter for Body weight and Tg [28].

Parameters	Levels			
	1	2	3	
A: Preform Temperature (°C)	100-105	105-110	110-115	
B: Stretch Rod Position (mm)	15	20	25	
C: Final Pressure (Bar)	0	150	250	

Table 9. The design parameters and their values relating to their levels

Experiment No	А	В	С	R 1	R2	R3	R4	R5
1	1	1	1	14.38	37.76	12.27	105	65.96
2	1	2	2	13.68	97.74	9.27	100	78.13
3	1	3	3	12.97	206.5	6.24	0	84.52
4	2	1	2	12.42	77	11.02	30	62.3
5	2	2	3	13.03	241.6	4.34	0	57.3
6	2	3	1	11.97	154.6	9.66	61	87.13
7	3	1	3	11.13	163.87	9.03	25	84.18
8	3	2	1	12.02	104.66	9.69	60	92.2
9	3	3	2	12.22	85.7	10.04	80	67.4

Table 10. Experimental plan for L9

 Table 11. Factorial effect and contribution ratio for Body-Weight

	Level	А	В	С
SNR	1	22.71	21.99	22.11
	2	21.91	22.21	22.12
	3	21.42	21.85	21.83
R (Max-Min)		1.29	0.35	0.29
Rank		1	2	3
Contribution ratio (%)		66.84	18.13	15.03

 Table 12. Factorial effect and contribution ratio for Top-Load

	Level	А	В	С
SNR	1	39.21	37.85	38.57
	2	43.06	42.62	38.73
	3	41.12	42.91	46.08
R (Max-Min)		3.85	5.06	7.51
Rank		3	2	1
Contribution ratio				
(%)		23.44	30.82	45.74

Table 13. Factoric	ıl effect an	d contribution	ratio for	·Burst-Pressure
---------------------------	--------------	----------------	-----------	-----------------

	Level	А	В	С
SNR	1	19.01	20.58	20.4
	2	17.76	17.27	20.07
	3	19.62	18.55	15.92
R (Max-Min)		1.86	3.31	4.48
Rank		3	2	1
Contribution ratio				
(%)		19.27	34.31	46.42

Table 14. Factorial effect and contribution ratio for Stress Crack Resistance

	Level	А	В	С
SNR	1	-6.525	32.642	37.231
	2	-11.584	-8.146	-35.868
	3	33.861	-8.744	-57.347

R (Max-Min)	45.445	41.386	94.578
Rank	2	3	1
Contribution ratio			
(%)	25.05	22.81	52.14

	Level	А	В	С
SNR	1	37.59	36.93	38.16
	2	36.62	37.44	36.77
	3	38.12	37.97	37.4
R (Max-Min)		1.51	1.05	1.39
Rank		1	3	2
Contribution ratio				
(%)		38.23	26.58	35.19

Table 15. Factorial effect and contribution ratio for Tg

|--|

		Parameter	rs	
		А	В	С
Bodyweight	Optimum Level	1	2	2
	Optimum Value	100-105	20	150
Top Load	Optimum Level	2	3	3
	Optimum Value	105-110	25	250
Burst Pressure	Optimum Level	3	3	1
	Optimum Value	110-115	25	0
Stress Crack Resistance	Optimum Level	3	1	1
	Optimum Value	100-105	25	0
Tg	Optimum Level	2	1	3
	Optimum Value	105-110	15	250
General	Optimum Level	2	2	2
	Optimum Value	105-110	20	150

The most valuable step is to reunit these factors (maximizing of the Bodyweight, Top Load, Burst Pressure, Tg and Stress Crack Resistance, simultaneously) for gaining an idea over the whole optimization by making a reasonable comparison of each target. The optimization of the multiple performances of the bottle, which is the subject of this study, can be investigated in two ways [28]. Firstly, the separate effects of each goal for general optimization, the importance levels of each parameter were defined. The importance level of each parameter was determined. The optimums of multi-performance levels were specified in accordance with these ranks [28, 33]. The optimum conditions of the design parameters were found to be $A_2B_2C_2$.

The second way for the optimization of multi-performance is Grey Relational Analysis. The details and results are given in the next section.

3.1. Grey Relational Analysis

The Grey Relational Analysis (GRA) depends on the grey system theory. It is used to qualify complex relationships in cases where there are multiple performance characteristics [26]. GRA was offered by Deng as a relatively accurate method for multiple attribute decision making, which is predicated on the minimization of maximum distance from the ideal referential alternative. Several response variables are converted into single response function and the final response function is maximized in GRA [27, 28]. Our

aim in this study was to achieve the highest value of Bodyweight, Top Load, Burst Pressure, Tg and Stress Crack Resistance (higher-the-better) simultaneously [26, 28, 33].

The experimental results (from TM OA) were normalized ranging from 0 to 1 via using the equation in the initial stage that is known as grey relational generation [27, 28]. e_{rp} shows the rth response variable among p experiments. mine_{rp} and max e_{rp} are the smallest and the largest value. N_{rp} shows the normalized value rth response variable in the pth experiment. Higher-the-better and lower-the-better criteria are stated by equations:

$$N_{rp} = \frac{\left[e_{rp} - mine_{rp}\right]}{\left[maxe_{rp} - mine_{rp}\right]} , \qquad (2)$$

$$N_{rp} = \frac{[maxe_{rp} - e_{rp}]}{[maxe_{rp} - mine_{rp}]}.$$
(3)

Grey relational coefficients (C_{rp}) of the responses were evaluated using Eq 4. These values were calculated from the normalized data (N_{rp}) to exemplify a correlation between the desired and the actual experimental data [28]. N_r is the ideal normalized value i.e. the maximum of the normalized SNR for the rth response variable. W is the identification co-efficient whose value is generally 0.5 [28, 33].

$$C_{rp} = \frac{\min_{r}^{|N_{r} - N_{rp}|} + \max_{r}^{|N_{r} - N_{rp}|}}{|N_{r} - N_{rp}| + \max_{r}^{|N_{r} - N_{rp}|}}.$$
(4)

Grey relational grade (G_r) was estimated by averaging C_{rp} with Equation (5) as below:

$$G_r = \frac{1}{n} \sum C_{rp} \,. \tag{5}$$

 G_r is the total response of the process instead of the multiple responses. A higher value of G_r indicates that the corresponding factors combination is closer to the optimum condition. In addition, the optimum process condition is evaluated from the main effect plot of C_{rp}. Thus the optimization of the multiple process response is converted into optimization of a single Gr [27, 28, and 33]. Finally, the optimal combination of the parameters was procured in care of grey relational analysis [26]. The GRA results are shown in Tables 17, 18 and 19. The highest grey relational degree was acquired in experiment 1 (see Table 19 and Figure 5). That is to say that the highest value for the Body Weight, Top Load, Burst Pressure, Tg and Stress Crack Resistance were got through in the 1st experiment in the OA. Table 20 and Figure 6 point out average grey relational levels (GRLs) of the design parameters. The highest value gives the best result here as well [26]. Red values represent the highest grey relational value for each parameter. The effect of design parameters on multiple performances was attained on $A_1B_2C_1$ levels. These levels are the optimum parameter levels which permit for achieving maximum Bodyweight, Top Load, Burst Pressure, Tg and Stress Crack Resistance factor values. The differences between maximum and minimum values of grey relational degrees are shown in Table 20. The benchmarking of these differences gives the significance levels of parameters affecting the performance characteristic [26, 28]. The most significant parameter is the one with the highest difference value. In other words, among the 3 design parameters in this study, the most important parameter affecting the multi-performance properties is the Preform Temperature (parameter A) [26]. However, Final Pressure (parameter C) is very close to Parameter A. Validation of experiments was performed in the final step. The general results for TM and GRA are seen in Table 21. According to these results, all the general results of GRA (except for Stress Crack Resistance) are greater than the general results of TM [28, 33]. The general result of TM for Stress Crack Resistance is greater than the general result of GRA for Stress Crack Resistance.

Table 17. Normalized data

	R1	R2	R3	R4	R5
References Series	1	1	1	1	1
Comparability series					
1	1	0	1	1	0.248138

3 0.566154 0.827806 0.239596 0 0.779943 4 0.396923 0.192504 0.842371 0.285714 0.143266 5 0.584615 1 0 0 0 6 0.258462 0.573195 0.67087 0.580952 0.854728 7 0 0.618672 0.591425 0.238095 0.770201 8 0.273846 0.328199 0.674653 0.571429 1 9 0.335385 0.235184 0.718789 0.761905 0.289398	2 3 4 5 6 7 8 9	$\begin{array}{c} 0.784615\\ 0.566154\\ 0.396923\\ 0.584615\\ 0.258462\\ 0\\ 0.273846\\ 0.335385\end{array}$	$\begin{array}{c} 0.29425\\ 0.827806\\ 0.192504\\ 1\\ 0.573195\\ 0.618672\\ 0.328199\\ 0.235184 \end{array}$	0.62169 0.239596 0.842371 0 0.67087 0.591425 0.674653 0.718789	$\begin{array}{c} 0.952381\\ 0\\ 0.285714\\ 0\\ 0.580952\\ 0.238095\\ 0.571429\\ 0.761905 \end{array}$	$\begin{array}{c} 0.596848\\ 0.779943\\ 0.143266\\ 0\\ 0.854728\\ 0.770201\\ 1\\ 0.289398 \end{array}$
--	--------------------------------------	--	--	---	--	--

Table 18. Difference between the reference value and normalized value

	R 1	R2	R3	R4	R5
1	0	1	0	0	0.751862
2	0.215385	0.70575	0.37831	0.047619	0.403152
3	0.433846	0.172194	0.760404	1	0.220057
4	0.603077	0.807496	0.157629	0.714286	0.856734
5	0.415385	0	1	1	1
6	0.741538	0.426805	0.32913	0.419048	0.145272
7	1	0.381328	0.408575	0.761905	0.229799
8	0.726154	0.671801	0.325347	0.428571	0
9	0.664615	0.764816	0.281211	0.238095	0.710602

Table 19. GRA results

			Crp			_	
Test No	R1	R2	R3	R4	R5	$\mathbf{G}_{\mathbf{r}}$	Order
1	1	0.333333	1	1	0.399405	<u>0.746548</u>	1
2	0.698925	0.41468	0.569275	0.913043	0.553617	0.629908	2
3	0.53542	0.743833	0.396698	0.333333	0.694389	0.540735	5
4	0.453278	0.38241	0.760307	0.411765	0.368532	0.475258	9
5	0.546218	1	0.333333	0.333333	0.333333	0.509244	7
6	0.402726	0.539488	0.603042	0.544041	0.774867	0.572833	4
7	0.333333	0.567325	0.550312	0.396226	0.68512	0.506463	8
8	0.407779	0.426693	0.605806	0.538462	1	0.595748	3
9	0.429326	0.395315	0.640032	0.677419	0.413018	0.511022	6



Figure 5. Grey relational levels

Levels	Α	В	С
1	<u>0.639063</u>	0.57609	<u>0.638376</u>
2	0.519112	<u>0.5783</u>	0.538729
3	0.537745	0.54153	0.518814
Max	0.639063	0.5783	0.638376
Min	0.519112	0.54153	0.518814
Deviation	0.119952	0.03677	0.119562

Table 20. Average GRL values and parameters

Table 21. General results for TM and GRA

	General results	А	В	С	R1	R2	R3	R4	R5
ТМ	Optimum Level	2	2	2	12.98	96.4	11.11	<u>885</u>	70.3
GRA	Optimum Level	1	2	1	<u>13.56</u>	<u>98.91</u>	<u>12.06</u>	5	<u>92.9</u>

4. CONCLUSION

The applications of TM and GRA methods were carried out to specify the best values of design parameters in PET bottles. The impacts of the design parameters on results and the performance were controlled with TM and GRA. In this study, it was cleared up that the top load strength of the final bottle was associated with the stretch rod movement. In addition, top load strength was effected by the preform and bottle mold design, mold cooling temperature and stretch rod acceleration. It was not possible to establish a clear relationship affecting the glass transition temperature. Moreover, it was seen that the panel (body) weight is directly and seriously affected by the preform surface temperature. It was also found that the final pressure and stretch rod also affected the body weight by bilateral interaction. As can be seen from the ECHIP-7 statistical analysis program for body weight are meaningful and within the acceptable limits. These values indicate that the output from the program for the final bottle body weight is reliable. Environmental Stress Crack Resistance (ESCR) was affected by the stretch rod movement. Finally, the process parameters affecting the bottle performance were designated by means of the ECHIP program in this work. However, Taguchi and Grey relational analysis methods were used since the optimum process parameters could not be specified. According to these results, all the general results of GRA (except for Stress Crack Resistance) are greater than the general results of Taguchi. The general result of Taguchi for Stress Crack Resistance is greater than the general result of GRA for Stress Crack Resistance. Other multicriteria decision making methods can be used in future studies instead of the GRA method.



Figure 6. Effects of parameter levels on multi-performance

5. ACKNOWLEDGEMENTS

This study was advanced by Erciyes University under the project number FYL-2016-6999. The authors would like to thank undergraduate students Mert Öztürk and Orhan Peşan, and Dear Mehmet Durusoy owner of Durusoy Makina for their contribution.

CONFLICTS OF INTEREST

No conflict of interest was declared by the authors.

REFERENCES

- Demirel, B., Daver, F., Yaraş, A., "Simulation of stress distribution in the base of pet bottles under different processing conditions", 6th International Advanced Technologies Symposium, Elazığ, 1-5, (2011).
- [2] Demirel, B., Daver, F., Kosior, E., "Petaloid bases of PET bottles: Design and process optimisation against environmental stress cracking", SPE ANTEC, Cincinnati, 2159-2164, (2007).
- [3] McEvoy, J. P., Armstrong, C. G., Crawford, R. J., "Simulation of the stretch blow molding process of PET bottles", Advances in Polymer Technology, 17: 339-352, (1998).
- [4] Martin, L., Stracovsky, D., Laroche, D., Bardetti, A., Ben-Yedder, R., Di Raddo, R., "Modelling and experimental validation of the stretch blow molding of PET", SPE ANTEC, New York, 982-987, (1999).
- [5] Yang, Z. J., Jones, E. H., Menary, G. H., Armstrong, C. G., "A non-isothermal finite element model for injection stretch-blow molding of PET bottles with parametric studies", Polymer Engineering and Science, 44: 1379-1390, (2004).
- [6] Hanley, T., Sutton, D., Cookson, D., Koisor, E., Knott, R., "Molecular morphology of petaloid bases of PET bottles: a small-angle X-ray scattering study", Journal of Applied Polymer Science, 99: 3328-3335, (2006).

- [7] Demirel, B., "Optimization of mold surface temperature and bottle residence time in mold for carbonated soft drink PET containers", Polymer Testing, 60: 220-228, (2017).
- [8] Monteix, S., Schmidt, F., Le Maoult, Y., Ben Yedder, R., Diraddo, R. W., Laroche, D., "Experimental study and numerical simulation of preform or sheet exposed to infrared radiative heating", Journal of Materials Processing Technology, 119: 90-97, (2011).
- [9] Jabarin, S. A., "Optical properties of thermally crystallized poly (ethylene terephthalate)", Polymer Engineering and Science, 22: 815-820, (1982).
- [10] Salem, D. R., "Microstructure development during constant-force drawing of poly (ethylene terephthalate) film", Polymer, 39: 7067-7077, (1998).
- [11] Miller, M. L., "The Structure of Polymers", Reinhold Publishing Corp., New York, (1966).
- [12] Collins, E. A., Bares, J., Billmeyer, F. W., "Experiments in Polymer Science", John Wiley and Sons, New York, (1973).
- [13] Wang, S., Makinouchi, A., Nakagawa, T., "Three-Dimensional viscoplastic FEM simulation of a stretch blow molding process", Advances in Polymer Technology, 17: 189-202, (1998).
- [14] Lebaudy, P., Grenet, J., "Heating simulation of multilayer preforms", Journal of Applied Polymer Science, 80: 2683-2689, (2001).
- [15] Zagarola, S. W., "Designing PET preform injection molding process for the lightest practical weight offers opportunities for improved productivity and quality", SPE ANTEC, Atlanta, 606-610, (1998).
- [16] Senyigit, E., Düğenci, M., Aydin, M. E., Zeydan, M., Heuristic-based neural networks for stochastic dynamic lot sizing problem", Applied Soft Computing, 13(3): 1332–1339, (2013).
- [17] Babayigit, B., Senyigit, E., "Design optimization of circular antenna arrays using Taguchi method", Neural Computing and Applications, 28(6): 1443-1452, (2017).
- [18] Babayiğit, B., Şenyiğit, E., "Application of the Taguchi Method to the Design of Circular Antenna Arrays", 9th International Conference on Electrical and Electronics Engineering (ELECO), Bursa, Türkiye, 342-345, (2015).
- [19] Ball, A. K., Das, R., Roy, S. S., Kishu, D. R., Murmu, N. C., "Experimentation modelling and optimization of electrohydrodynamic inkjet microfibration approach: a Taguchi regression analysis", Sadhana, 44: 167, (2019).
- [20] Nguyen, T.T., Duong, Q.D., "Optimization of WEDM process of mould material using Kriging model to improve technological performances", Sadhana, 44: 154, (2019).
- [21] Adhikary, S., Sekhar, H., Thakur, D. G., "Optimisation of density of infra-red decoy flare pellets by Taguchi method", Sadhana, 44: 160, (2019).
- [22] Gunes, S., Manay, E., Senyigit, E., Ozceyhan, V., "Taguchi approach for optimization of design parameters in a tube with coiled wire inserts", Applied Thermal Engineering, 31(14-15): 2568–2577, (2011).
- [23] Yildiz, Y. Ş., Şenyiğit, E., İrdemez, Ş., "Optimization of Specific Energy Consumption for Bomaplex Red Cr-L Dye Removal from Aqueous Solution by Electrocoagulation Using Taguchi-Neural Method", Neural Computing and Applications, 23(3-4): 1061-1069, (2013).

- [24] Babayiğit, B., Şenyiğit, E., Mumcu, G., "Optimum broadband E-patch antenna design with Taguchi method", Journal of Electromagnetic Waves and Applications, 30: 915-927, (2016).
- [25] Celik, N., Pusat, G., Turgut, E., "Application of Taguchi method and grey relational analysis on a turbulated heat exchanger", International Journal of Thermal Sciences, 124: 85-97, (2018).
- [26] Nelabhotla, D. M., Jayaraman, T. V., Asghar, K., Das, D., "The optimization of chemical mechanical planarization process-parameters of c-plane gallium-nitride using Taguchi method and grey relational analysis", Materials and Design, 104: 392-403, (2016).
- [27] Gunes, S., Senyigit, E., Karakaya, E., Ozceyhan, V., "Optimization of heat transfer and pressure drop in a tube with loose-fit perforated twisted tapes by Taguchi method and grey relational analysis", Journal of Thermal Analysis and Calorimetry, 136(4): 1795-1806, (2019).
- [28] Demirel, B., Daver, F., "The effects on the properties of PET bottles of changes to bottle-base geometry", Journal of Applied Polymer Science, 114: 3811-3818, (2009).
- [29] Mehta, A., Gaur, U., Wunderlich, B., "Equilibrium melting parameters of poly (ethylene terephthalate", Journal of Polymer Science: Polymer Physics Edition, 16: 289-296, (1978).
- [30] Demirel, B., "Optimisation of Petaloid Base Dimensions and Process Operating Conditions to Minimise Environmental Stress Cracking in Injection Stretch Blow Molded Pet Bottles", Ph.D. Thesis, RMIT University, Australia, (2008).
- [31] Brocka, Z., Schmactenberg, E., Ehrenstein, G. W., "Radiation Cross Linking Engineering Thermoplastic for Tribological Applications", SPE ANTEC, Cincinnati, 1690-1694, (2007).
- [32] Üstüntağ, S., Şenyiğit, E., Mezarcıöz, S., Türksoy, H. G., "Optimization of coating process conditions for denim fabrics by taguchi method and grey relational analysis", Journal of Natural Fibers, 1–15, (2020). DOI:10.1080/15440478.2020.1758866
- [33] Soutis, C., "Fibre reinforced composites in aircraft construction", Progress in Aerospace Sciences, 41(2): 143–51, (2005).