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DEVELOPMENT OF SHORT TERM VALIDATION TEST FOR THERMO-FORMED NYLON TUBES

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ABSTRACT

There are few or none investigations on short-term validation test to verify whether a thermoforming process of nylon tubes is robust or not. Analysis of variance allows us to identify significant factors in the validation of thermoforming process to establish or develop a short term validation test. Parametric study t-student analysis was used to compare a long term testing established within the automotive industry in thermoformed plastic with a proposal short term test to be used on a production environment. These experiments guide us to understand that the temperature and time factors are not significant on a validation test for Nylon 6 thermoforming process when working in a lower range of yield temperature, and the testing period can be reduced significantly.

Keywords: Analysis of variance, parametric study t-student, Thermoforming process, Validation Test.

I. INTRODUCTION

During the last decades the application of plastic components in the automotive industry has been increasing. Following this evolution, specifically talking about thermo-forming nylon tubes, the designs have become very complex geometries, and this requires having more robust manufacturing processes to ensure that the tubes will maintain their shape under operating and different environmental conditions; as a result the product validation tests become an indispensable tool to ensure the quality of the parts. Unfortunately the testing for nylon parts has not been improving according with this evolution, the automotive industries have only long –term validation tests that are not optimal for use in a production environment, this situation led us to the need of developing short-term tests that are equivalent to those already defined and validated by the automotive customers. Some polymers or plastics such as Nylon 6 have a property called shape memory, this feature is the ability of the material to remember its original shape when is affected by external stimulus that alter some of its properties [1], as long as these do not reach the temperature of formation (yield temperature) the plastic will maintain the form [2], this feature of the material will provide the basics for the definition of the parameters on the proposed testing.

1.1 Thermoforming process

When a thermoplastic polymer is heated and it will show a variation in his hardness, these characteristic is used in thermoforming process to change the form of a plastic tube [4]. The following describes in general terms the thermoforming process of a plastic tube. The process starts with nylon tube extrusion, that consist in a plastic deformation in which the material is forced to flow through one die orifices under high pressure to produce tubes of the desired diameter configuration. The next process is the thermoforming of tubes, also known as bending process, in which the tube is routed into a mold that will give the required form, the first step is the heating cycle is carried out by injecting compressed hot air at high temperature (700^{0}F) and at a considerable speed [1], so that the necessary heat energy is transferred to modify the modulus of elasticity and hardness of the plastic tube and reach the yield temperature [2], after reaching this temperature the material tends to become a rubberized state. Once the yield temperature is reached to form the tube, hot air injection is stopped and stability cycle starts. Follow by a coolant period using injection of air at room temperature, until gets the original rigidity and retain the shape and geometry set by the mold. The yield temperature, determined that it became the most critical parameter during the process of thermoforming, when this temperature is not achieved the tube will not be able to overcome the memory of the material and will tend to recover its original shape over time, this feature of the material is called relaxation. There are several internal and external factors that could affect the thermoforming process to not reach the yield temperature, some of them can be controlled and some not, due to the nature of this process the implementation of a verification test becomes an important part to ensure the tube has been bent correctly and will maintain the form under any environmental condition or temperature changes. When the tube does not maintain its geometry or specific form and presents relaxation, it could cause installation problems to the end customer. The present work has as its central theme the definition of a short test for thermo-formed nylon tubes adapted to a production environment.

1.2 Long Term Validation Test

Long term formed bend tube- relaxation test procedure consist in expose the tube to a hot air soak of 105°C - 110°C for one hour minimum. Measure bend angle within 5 minutes of removal from the hot air exposure. Requirements of acceptability: the angle of the bend when measured in Free State must not exceed the tolerance specified before or after heat soak. Normal tolerance specified +/- 10 grades of relaxation [5]

This is the specification test required by some automotive customers and will be used as a base line to develop an equivalent testing reducing the testing time.

II. MATERIAL AND EXPERIMENTAL METHODS

2.1 Materials

GCA Precision Mechanical Convection Oven will be used to perform the long term Validation test. And metal angle meter will be used to measure the tube angles. The relaxation angle will be calculated measuring the tube angle before testing with metal angle meter and subtracting the value of the measurement of the same tube angle after testing. Two different geometric tubes were used to perform several experiments detailed below.

2.2 Experimental Methods

The first step was to evaluate the significant effect on the two main factors identified on the validation test: temperature and time. Factorial designs are widely used in experiments involving several factors where it is necessary to study the joint effect of the factors on a response [3]. A factorial design 2^2 was used to evaluate the effect of these factors on the variable response defined as the relaxation angle. Once these experiments were run the second step was define the parameters to be used on the proposed test method (short test) called Hydro test, the suggested short test consist in immersing the tubes in high temperature water for a period of time. Final step

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was used the Student t-test for testing a hypothesis on the basis of an equal sample mean between two populations for the long term test and suggested hydro test as an optional short testing method.

III. RESULTS

Table (1) shows the Factorial Design 2^2 used on tube A (complex geometry Tube A) to determine if the factors temperatures and time have significant effect on the variable response relaxation angle using the long term validation test. To perform the experiment it was confirmed that the data values of relaxation angle follow a normal distribution; the decision rule for normality is P-value greater than 0.05 with a confidence level of 95%. In this case the P-value was 0.229, so we conclude that the data was normal an ANOVA assessment was determined feasible.

Table 1. Factorial Design- Long Test Tube A

RunOrder	Temp	Time	Piece Num	Temp	*Time*	Relaxation angle
1	-1	-1	21	85°C	*8 min*	4
2	1	-1	29	135°C	*8 min*	18
3	-1	1	17	85°C	*60 min*	3
4	1	1	25	135°C	*60 min*	12
5	-1	-1	22	85°C	*8 min*	8
6	1	-1	30	135°C	*8 min*	17
7	-1	1	18	85°C	*60 min*	18
8	1	1	26	135°C	*60 min*	15

Table (2) shows the ANOVA results for factorial Design-Long Test tube A using software Minitab 16. Analyzing this results of full factorial experiment, it is conclude that the levels identified for both time and temperature factors for the long test suing tube A were not significant (P value>0.05). When the interaction is significant, this takes precedence over the individual effects, in this case no significant interaction resulted therefore optimal levels would be selected based only on the graphs of the main effects.

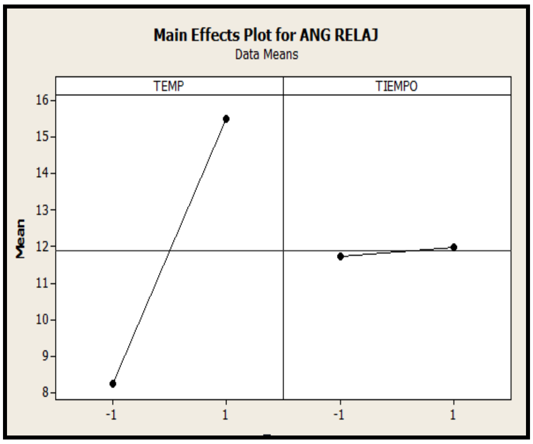
Table 2: Results of Factorial Design – Long Test Tube A

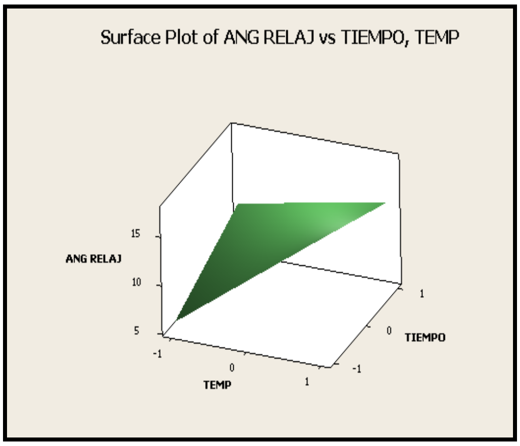
Estimated Effects and Coefficients for Relaxation Angle					
Term	Effect	Coef	SE Coef	T	P
Constant		11.875	1.98	6.00	0.004
Temp	7.250	3.625	1.98	1.830	0.141
Time	0.250	0.125	1.98	0.060	0.953
Temp*Time	-4.250	-2.125	1.98	-1.070	0.344
S =5.60134	PRESS = 502				
R-Sq = 52.97%	R-Sq (pred) = 0.00%		R-Sq (adj) = 17.70%		

Analysis of Variance for ANG RELAJ (coded units)						
Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	2	105.250	105.250	52.625	1.68	0.296
Temp	1	105.125	105.125	105.125	3.350	0.141
Time	1	0.125	0.125	0.125	0.000	0.953
2-Way Interact	1	36.125	36.125	36.125	1.150	0.344
Temp*Time	1	36.125	36.125	36.125	1.150	0.344
Residual Error	4	125.500	125.500	31.375		
Pure Error	4	125.500	125.500	31.375		
Total	7	266.875				

Last Squares Means		
TEMP	Mean	SE Mean
-1	8.250	2.801
1	15.500	2.801
TIME		
-1	11.750	2.801
1	12.000	2.801

Figure 1: Shows the main effects and Surface plot: Temperature and time factors long test Tube A





Same experiment was developed to the same Tube A, the Table (3) shows the Factorial Design 2^2 used on tube A to determine if the factors temperatures and time have significant effect on the variable response relaxation angle using the short term validation test (proposed Hydro test as an alternative to the long test). To perform the experiment it was confirmed that the data values of relaxation angle follow a normal distribution, the decision rule for normality is P-value greater than 0.05 with a confidence level of 95%, and then the data was normal. In this case the P-value was 0.077, so we conclude that the data was normal an ANOVA assessment was determined feasible.

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Table 3. Factorial Design- Short Test Tube A

RunOrder	Temp	Time	Piece Num	TEMP	TIME	Relaxation angle
1	-1	-1	1	85°C	8 min	20
2	1	-1	3	100°C	8 min	18
3	-1	1	13	85°C	60 min	37
4	1	1	15	100°C	60 min	51
5	-1	-1	2	85°C	8 min	23
6	1	-1	4	100°C	8 min	17
7	-1	1	14	85°C	60 min	33
8	1	1	16	100°C	60 min	19

Analyzing the results of full factorial experiment, it is determined that the levels identified for both time factor and temperature factor were not significant (P value>0.05). It was concluded that both experiments with long and short test for the factor level selected the temperature and time were not significant to the variable response for tube A,

Estimated Effects and Coefficients for Relaxation Angle

Term	Effect	Coef	SE Coef	T	P
Constant		27.250	4.05	6.73	0.003
Temp	-2.000	-1.000	4.05	-0.025	0.817
Time	15.500	7.750	4.05	1.910	0.128
Temp*Time	2.000	1.000	4.05	0.250	0.817

S =11.4564 PRESS = 2100
R-Sq = 48.60% R-Sq (pred) = 0.00% R-Sq (adj) = 10.06%

Analysis of Variance for ANG RELAJ (coded units)

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	2	488.500	488.500	244.250	1.86	0.268
Temp	1	8.000	8.000	8.000	0.060	0.817
Time	1	480.500	480.500	480.500	3.660	0.128
2-Way Interact	1	8.000	8.000	8.000	0.060	0.817
Temp*Time	1	8.000	8.000	8.000	0.060	0.817
Residual Error	4	525.000	525.000	131.250		
Pure Error	4	525.000	525.000	131.250		
Total	7	1021.5				

Last Squares Means

TEMP	Mean	SE Mean
-1	28.250	5.728
1	26.250	5.728
TIME		
-1	19.500	5.728
1	35.000	5.728

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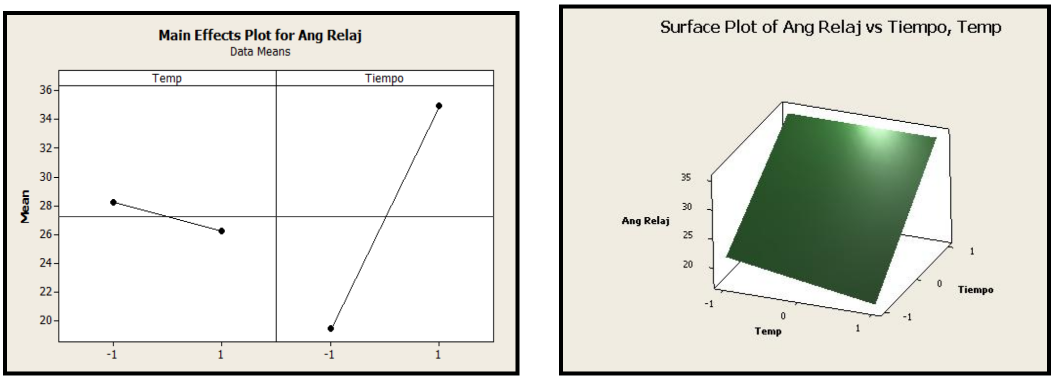
Figure 2: Shows the main effects and Surface plot: Temperature and time factors



One of the main issues observed during these two experiments was that the relaxation angle data shows that the tube exceed the acceptance criteria defined on the long term test, therefore the bending process was not adjusted properly. The tube A used on these experiments it is a tube made of nylon 6 with a complex geometric and a length over 600 mm, it was considered necessary to perform both experiments to a different tube with simple complexity in order to compare results.

Same process was performed to a second tube B, nylon 6 with one simple bend of 90° angle, the Table (5) shows the Factorial Design 2^2 used to evaluate the same factors temperatures and time and see if they have significant effect on the variable response relaxation angle using first the long term validation test. To perform the experiment it was confirmed that the data values of relaxation angle follow a normal distribution; In this case the P-value was 0.466, so we conclude that the data was normal an ANOVA assessment was determined feasible.

Table 5. Factorial Design- Long Test Tube B

RunOrder	Temp	Time	Piece Num	Temp	Time	Relaxation angle
1	-1	-1	1	85°C	8 min	1
2	1	-1	5	135°C	8 min	1
3	-1	1	3	85°C	60 min	4
4	1	1	7	135°C	60 min	2
5	-1	-1	2	85°C	8 min	0
6	1	-1	6	135°C	8 min	4
7	-1	1	4	85°C	60 min	3
8	1	1	8	135°C	60 min	3

Analyzing the results of full factorial experiment, it was determined that at the levels identified for both time and temperature factors were not significant (P value>0.05), this show same results than the previous experiments done on Tube A, with the difference that the relaxation angle comply with the acceptance criteria (+/-10°). Time and temperature are no significant factors affecting the relaxation angle by the levels identified.

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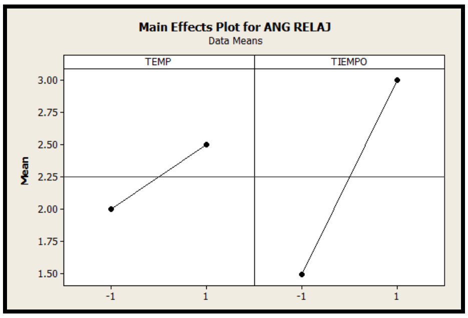
Table 6: ANOVA Results

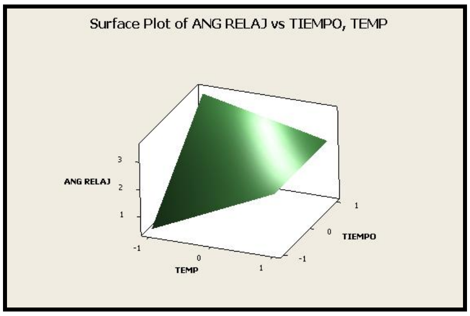
Estimated Effects and Coefficients for Relaxation Angle					
Term	Effect	Coef	SE Coef	T	P
Constant		2.250	0.433	5.200	0.007
Temp	0.500	0.250	0.433	0.580	0.595
Time	1.500	0.750	0.433	1.730	0.158
Temp*Time	-1.500	-0.75	0.433	-1.73	0.158
S =1.22474	PRESS = 24		R-Sq (adj) = 32.26%		
R-Sq = 61.29%	R-Sq (pred) = 0.00%				

Analysis of Variance for ANG RELAJ (coded units)						
Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	2	5.000	5.000	2.500	1.67	0.298
Temp	1	0.500	0.500	0.500	0.330	0.595
Time	1	4.500	4.500	4.500	3.000	0.158
2-Way Interact	1	4.500	4.500	4.500	3.000	0.158
Temp*Time	1	4.500	4.500	4.500	3.000	0.158
Residual Error	4	6.000	6.000	1.500		
Pure Error	4	6.000	6.000	1.500		
Total	7	15.5				

Last Squares Means		
TEMP	Mean	SE Mean
-1	2.000	0.612
1	2.500	0.612
TIME		
-1	1.500	0.612
1	3.000	0.612

Figure 3: Shows the main effects and Surface plot: Temperature and time factors for Tube B Long Test





The Table (6) shows the last Factorial Design 2^2 performed on tube B to determine if the factors temperatures and time have significant effect on the variable response relaxation angle using the short term validation test (proposed Hydro test as an alternative to the long test). To perform the experiment it was confirmed that the data values of relaxation angle follow a normal distribution, the decision rule for normality is P-value greater than 0.05 with a confidence level of 95%, and then the data was normal. In this case the P-value was 0.683, so we conclude that the data was normal an ANOVA assessment was determined feasible. Analyzing the results of full factorial experiment, it was determined that at the levels identified for both time and temperature factors were not significant (P value>0.05) as well for Tube B running with the short test.

The relaxation angle data for tube B showed acceptable results based on acceptance criteria, nevertheless for both tubes, complex and simple geometry tubes were determined that the temperature and time factors were no significant effect when is applied a temperature below the yield point. This is very important item to consider on the parameters selected for the proposed short test. We can concluded based on this data that we can develop a testing minimizing the timing until 8 minutes with a minimum *temperature* of 85° without affecting drastically the relaxation angle compared with the long test used on automotive industry.

Table 6. Factorial Design- Long Test Tube B

RunOrder	Temp	Time	Piece Num	Temp	Time	Relaxation Angle
1	-1	-1	1	85°C	8 min	3
2	1	-1	3	100°C	8 min	1
3	-1	1	13	85°C	60 min	2
4	1	1	15	100°C	60 min	2
5	-1	-1	2	85°C	8 min	0
6	1	-1	4	100°C	8 min	5
7	-1	1	14	85°C	60 min	5
8	1	1	16	100°C	60 min	4

Table 7: ANOVA Results

Estimated Effects and Coefficients for Relaxation Angle

Term	Effect	Coef	SE Coef	T	P
Constant		2.750	0.7706	3.570	0.023
Temp	0.500	0.250	0.7706	0.320	0.762
Time	1.000	0.500	0.7706	0.650	0.552
Temp*Time	-1.000	-0.500	0.7706	-0.65	0.552

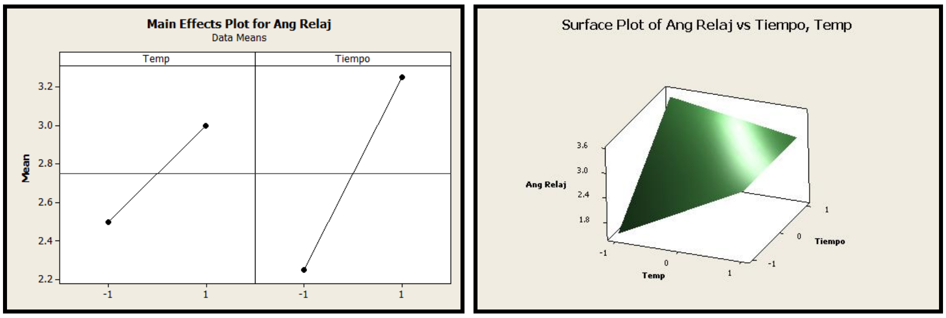
S =1.22474 PRESS = 24 R-Sq (adj) = 32.26%

R-Sq = 61.29% R-Sq (pred) = 0.00%

Analysis of Variance for ANG RELAJ (coded units)

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	2	2.500	2.500	1.250	0.26	0.781
Temp	1	0.500	0.500	0.500	0.110	0.762
Time	1	2.000	2.000	2.000	0.420	0.552
2-Way Interact	1	2.000	2.000	2.000	0.420	0.552
Temp*Time	1	2.000	2.000	2.000	0.420	0.552
Residual Error	4	19.000	19.000	19.000		
Pure Error	4	19.000	19.000	19.000		
Total	7	23.500				

Figure 4: Shows the main effects and Surface plot: Temperature and time factors for Tube B Short Test



In order to finalize the development of the short term test validation, we need to probe that the relaxation angle under the long term validation test has similar or equal results than the proposed short test. To develop this experiment it was decided to use the Tube B due to the bending process was considered stable to produce good parts. 60 samples were taken to run the long term validation test and the short test with a significant sample size of 30 samples used on each test to determine if both of them have same effect on the relaxation angle.

See Table (8) below with the relaxation angle data results for both tests. Applying a parametric test requires normality of the observations for each of the groups. So the data was analyzed using Minitab. The following Fig. 5 show the test results and both of them are normal due to P-value>0.05.

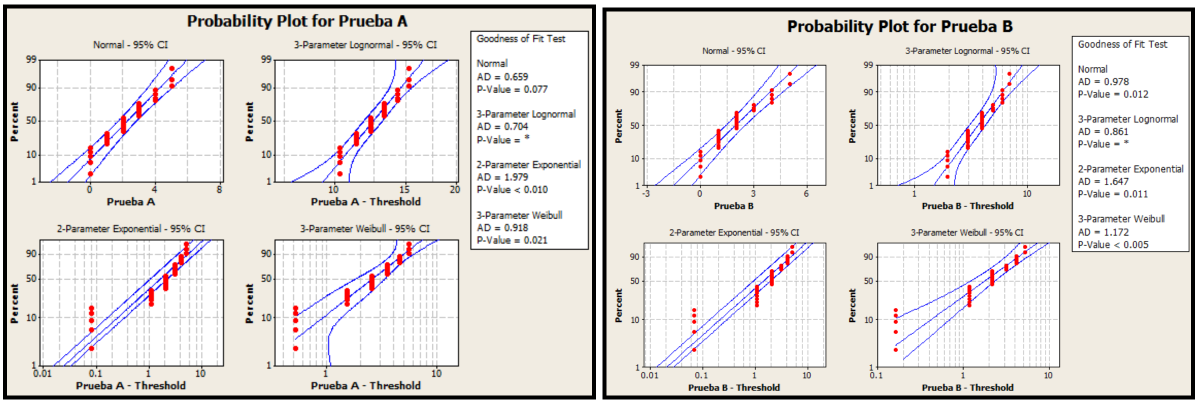
Table 8. Relaxation angle applying Long and Short term validation test to tube B

LONG TERM VALIDATION TEST				SHORT TERM VALIDATION TEST			
Piece No.	Temp	Time	Relax Angle	Piece No.	Temp	Time	Relax Angle
1	110°C	60 min	1	1	100°C	8 min	5
2	110°C	60 min	0	2	100°C	8 min	1
3	110°C	60 min	1	3	100°C	8 min	5
4	110°C	60 min	0	4	100°C	8 min	3
5	110°C	60 min	4	5	100°C	8 min	1
6	110°C	60 min	3	6	100°C	8 min	2
7	110°C	60 min	2	7	100°C	8 min	1
8	110°C	60 min	0	8	100°C	8 min	3
9	110°C	60 min	2	9	100°C	8 min	2
10	110°C	60 min	4	10	100°C	8 min	4
11	110°C	60 min	3	11	100°C	8 min	2
12	110°C	60 min	1	12	100°C	8 min	1
13	110°C	60 min	5	13	100°C	8 min	3
14	110°C	60 min	4	14	100°C	8 min	1
15	110°C	60 min	2	15	100°C	8 min	1

16	110°C	60 min	4
17	110°C	60 min	0
18	110°C	60 min	2
19	110°C	60 min	5
20	110°C	60 min	1
21	110°C	60 min	2
22	110°C	60 min	3
23	110°C	60 min	3
24	110°C	60 min	2
25	110°C	60 min	0
26	110°C	60 min	3
27	110°C	60 min	1
28	110°C	60 min	2
29	110°C	60 min	3
30	110°C	60 min	5

16	100°C	8 min	4
17	100°C	8 min	2
18	100°C	8 min	0
19	100°C	8 min	2
20	100°C	8 min	0
21	100°C	8 min	3
22	100°C	8 min	2
23	100°C	8 min	3
24	100°C	8 min	0
25	100°C	8 min	3
26	100°C	8 min	2
27	100°C	8 min	4
28	100°C	8 min	1
29	100°C	8 min	0
30	100°C	8 min	4

Figure 5. Probability Plot: Long test (A) and Short Test B



After determining the normal distribution of both tests and similar variance, we proceeded to perform statistical analysis t-Student. Minitab 16 was used and the test results are shown in the following Table:

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Table 9: Two-Sample T-Test and CI: Long and Short Test

Two -Sample T-Test and CI: Long Term, Short Term test

Source	N	Mean	St Dev	SE Mean
Long Term test	30	2.27	1.57	0.29
Short Term test	30	1.97	1.5	0.27

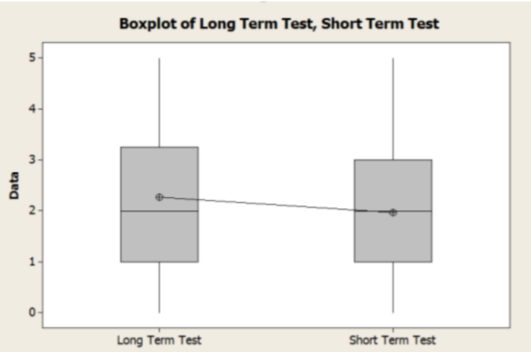
Difference = mu (Prueba A) - mu (Prueba B)

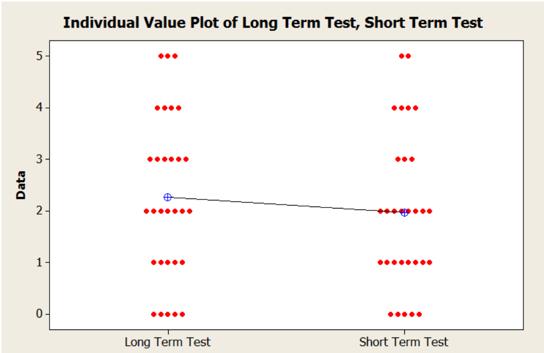
Estimate for difference: 0.300 0.3

95% CI for difference: (-0.494, 1.094)

T-Test of difference = 0 (vs not =): T-Value = 0.76 P-Value = 0.452 DF = 58

Both use Pooled StDev = 1.5360





Based on the P-Value that it is greater than 0.05, we can conclude that there is evidence that there is no difference in mean Relaxation angle using Long term and short term Validation Test, therefore the test method of tubes Immersed in water at high temperature with a defined time of 8 minutes have equal results to the long test defined by the automotive customer using 60 minutes to a temperature of 110 ° C.

IV. CONCLUSION

The main conclusions are based on the analysis of all experiments results. We can have two important results summarized as follows:

The temperature and time are two critical factors in the processes of thermo-formed plastic, but these are not significant factors for the development of a verification test for thermoforming process, when working in a lower range to yield temperature point.

It is further concluded that the test method immersed product in water at a temperature of 100 ° C with a time of 8 minutes has the same results as the test method defined by customers at a temperature of 110 ° C and a time of 60 minutes.

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