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EXPERIMENTAL AND THEORETIC RESEARCH REGARDING OPTIMIZATION EXTRUSION PROCESS FOR POLYMERS REINFORCED FIBER (*PA 6.6 – 30 % GF*)

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Abstract: *Polyamide 6.6 reinforced with 30 % glass fiber (PA 6.6 – 30 % GF) are defined by performant mechanical, thermal, and tribological properties. These properties improvement can be achieved by optimizing manufactured extruded profiles. Extrusion process temperature is important for extruded product quality. In this paper the authors proposed to realize a mathematical model for a tensile stress, resulted with the help of ANOVA (Analysis of variance) for a processing temperature in metering zone and forming zone (die zone). Metering zone and forming zone are the most important for extrusion process. Extruded product temperature optimization increases quality, productivity and productiveness.* ***Key words:*** *extrusion, reinforced polymers, polyamide, glass fiber short fiber, temperature, optimization.*

1. INTRODUCTION

Extrusion is a plastics continuous technological process can lead to infinite profile length such as: tubes, pipes, sheets, electrical cable insulation, molded products, [1], [2], [7], [8], [9], [10].

In this paper the main task is, properties and mechanical characteristics improving for *polyamide 6.6 reinforced with 30% glass fiber* (PA 6.6 - 30 % GF) extruded products. One of this improving solution is typical extrusion process temperatures optimization. From literature we know a fact that a major importance for a polymer extrusion process belongs to its processing temperature. Due to material data sheet fact *PA 6.6 – 30 % GF*, there is no specified processing temperature (this value ranges in a certain gap), we need to set an optimal value. In this case metering zone recommended processing ranges between
270 ÷ 290 °C, and forming zone temperature is between 260 – 280 °C, a certain value being unspecified (unknown). Metering zone and forming zone processing temperature was taken to account because these two areas are most important for an extruded product quality. [3], [4], [7], [8].

With help of ANOVA (Analysis of variance) we will be able to determine optimal temperature pairs for *PA 6.6 – 30 % GF* products, for which tensile value is maximum.

Dispersional analysis *ANOVA* is a part of mathematical and static techniques collection, used for technological process improving, optimizing and developing. This method belongs to (Response Surface Methodology - RSM) and is able to verify if a certain should belong or not, to the model. [5], [6], [8].
Most *RSM* applications are particular situations that may assume that more input variables can influence a performance size or a process quality characteristic. Those, performance size and process quality characteristic can be named response or dependents variables. Input variables are sometimes independent variable. [6].

Any technological process optimization is based on a mathematical model. Mathematical models can be used not only to optimal reveal conditions, they can be used as information for technological process optimal management. Modeling process and computer utility, allows an optimal decision problem approach, with a positive impact over technical and economical efficiency [6].

In our case we used 8.0.5. Design Expert, a software application that shows a group of statistical techniques used for experiment design and interpretation in which many factors can interfere. This software application offers an input factor analysis possibility found in a process, a quick view for critical ones and interactions between them. The program also can make a numeric and graphic optimization, a process factors deeper analysis and a process variable. The software allows a response surface shape graphical view under different forms [6].

2. SETTING THE NUMBER OF EXPERIMENTS. INSTALLATION EXTRUSION

In what follows, tensile stress of extruded products will be called a dependent variable and the two process parameters: metering zone temperature and die zone temperature will be called independent variables. When using response surface method, the first phase of establishing the conditions for optimization occurs, then the response surface is created between process variables and objective function.

Using response surface and a conventional method of optimization, optimal solution can be found. Since it is used a simple function, called the response surface, the procedure is very fast. It should be noted that an optimal solution depends on the precision accuracy of response surface approximation.

In response surface method, the choice of parameters is generally achieved by designing experiments and numerical analysis is repeated as many times as are set out in planning experiments.

Then, based on these results, response surface is constructed. Experiment planning purpose is to establish values of independent variables so their variation in space can be exploit.

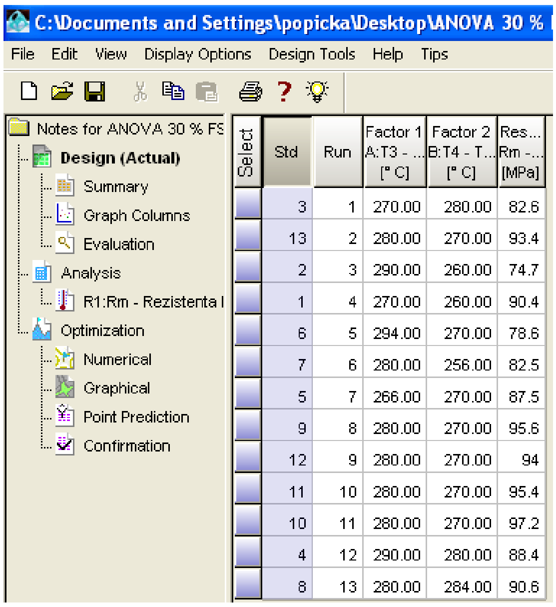
For various techniques that has been developed, such as factorial planning, central planning compositional (CCD), orthogonal planning, etc.

To obtain experimental data representative of the extrusion process, optimizing in *PA 6.6 - 30% GF* material, we adopted a central planning-type second-order compositional 2k where k = 2 (k-number of independent variables).

Following introduction of the minimum values of independent variables that will give maximum number of 13 experiments (virtual experiments, numerical simulation). After completion of the 13 experiments and numerical analysis, we determined the values of the dependent variable (tensile strength) figure 1 [5] [6].

For each experiment in hand we need to carry a minimum of 5 samples (according to SR EN ISO 527 - 2000), they will be tested at the traction.

Data corresponding response variable (tensile stress), last column on the right table in figure 1 was entered manually from the computer keyboard.



Std	Run	Factor 1 A:T3 - ... [° C]	Factor 2 B:T4 - T... [° C]	Res... Rm -... [MPa]
3	1	270.00	280.00	82.6
13	2	280.00	270.00	93.4
2	3	290.00	260.00	74.7
1	4	270.00	260.00	90.4
6	5	294.00	270.00	78.6
7	6	280.00	256.00	82.5
5	7	266.00	270.00	87.5
9	8	280.00	270.00	95.6
12	9	280.00	270.00	94
11	10	280.00	270.00	95.4
10	11	280.00	270.00	97.2
4	12	290.00	280.00	88.4
8	13	280.00	284.00	90.6

Fig.1. Programming matrix of central compositional experiments Design Expert 8.0.5 software in order to *PA 6.6 - 30% GF*

In our research we used a type Cincinnati Monos+45 installation with a single screw extruder (figure 2). Following the extrusion process resulted in a tube with a diameter of 30 mm and a thickness of 4 mm [6].

Processing temperatures were set according to experimental plan established with ANOVA test analysis (figure 1). Values of independent variables can be set using thermocouples T_3 , T_4 respectively.

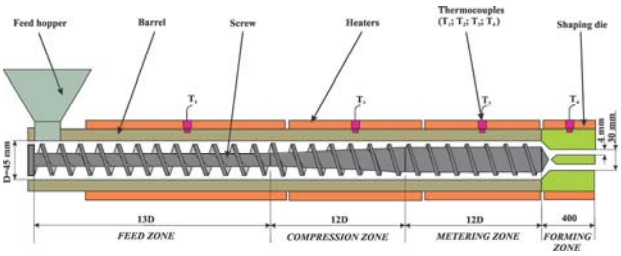


Fig. 2. Schematic single screw extruder type Cincinnati Monos + 45

From extrusion resulted tubes we cut required specimens according to SR EN ISO 527 – 2000 and they were choused for an experimental proposed program, figure 3. The researched specimen dimensions are: L_3= 150 mm, L_1= 60 mm, R= 60 mm, b_1= 10 mm, b_2= 20 mm, h= 4 mm, L_0=50 mm, L= 115mm. The samples were cut using a specially designed dies, mounted on a hydraulic press. Tensile tests of samples were carried out on tensile test machine Instron type 3366 shown in figure 4 [6].

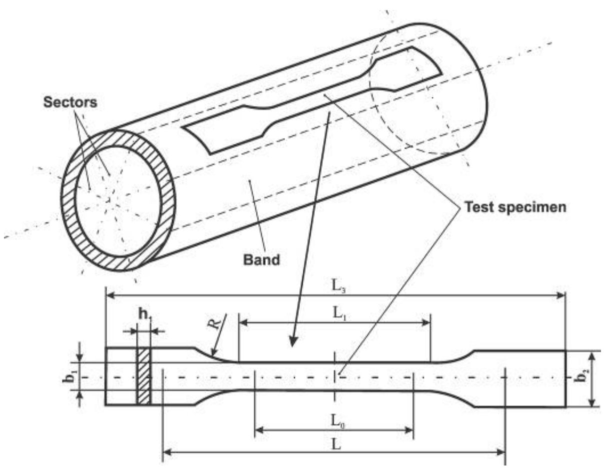


Fig. 3. Tensile test specimens according to SR EN ISO 527-2000

Fig. 4. Instron type 3366 tensile testing machine

After determining the arithmetic average values of tensile stress of each experiment, these values are entered in the Design Expert program, in order to determine the mathematical model of the tensile strength R_m according to the temperatures T_3 and T_4 for the chosen material [6].

3. TENSILE STRESS MATHEMATICAL MODEL FOR POLYMERIC MATERIAL *PA 6.6 - 30% GF*

To determine which model best describes the mathematical relationship between dependent variable and independent variables were used F statistic (Fischer), coefficient of determination R^2 and adjusted factor of determination R^2adj.

ANOVA test is presented in table 1, in which mathematical models are compared for *PA 6.6 - 30% GF*.

For each source we examine probability ("Prob> F") in order to see if it is below 0.05, a value that represents maximum permissible level of statistical significance.

As can be seen (see table 1), where polymeric materials in *PA 6.6 - 30% GF*, in addition 2FI vs. Quadratic model, all other models is more than statistical significance level of 0.05, which implies that Quadratic model vs. only 2FI is significant, other models are excluded [6].

Table1

ANOVA test table for the choice of model mathematical analysis dispersion tensile of material due *PA 6.6 - 30% GF* ("df" IS degrees of freedom)

Mathematical model	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Mean vs Total	1.019E+005	1	1.019E+005		
Linear vs Mean	100.65	2	50.33	1.02	0.3967
2FI vs Linear	115.56	1	115.56	2.74	0.1324
Quadratic vs 2FI	**365.60**	**2**	**182.80**	**88.66**	**<0.0001**
Cubic vs Quadratic	4.96	2	2.48	1.31	0.3490
Residual	9.47	5	1.89		
Total	1.025E+005	13	7883.56		

To ensure effectiveness of selected model we will consider coefficient of determination R^2 and predicted residual error sum of squares (PRESS - Predicted Residual Error Sum of Squares) (see table 2). PRESS is a measure of capacity model for approximates each point in space of independent variables.

R^2 is a measure of dispersion from the average value.

As "R Squared" value is closer to the unit value, the better model approximates the response function. Based on the quadratic model we chose for *PA 6.6 - 30% GF* material.

Table 2

The coefficient of determination R^2 for Rolling *PA 6.6 - 30% GF*

Mathematical model	Std. Dev.	R-squar	Adjusted R-Squared	Pred. R-squared	PRESS
Linear	7.04	0.1688	0.0026	-0.4466	862.54
2FI	6.50	0.3626	0.1502	-0.4969	892.50
Quadratic	**1.44**	**0.9758**	**0.9585**	**0.9102**	**53.55**
Cubic	1.38	0.9841	0.9619	0.9098	53.79

After establishing the type of mathematical model for approximating the dependent variables, we check whether a term should or should not be part in model [6].

The most common criterion in order to adding or removing a variable is based on partial F test.

In Table 3 *ANOVA* test is presented for a quadratic model adequately *PA 6.6 - 30% GF* material.

ANOVA test method confirm adequacy quadratic model, the term "sample F is smaller than 0.0001 (see table 3) [6].

Fischer 56.44 Value of Statistics shows that model is significant. There is only a 0.01% probability that average model lie outside the confidence interval (see table 3). There is only a 0.01% probability that average model lies outside the confidence interval (see table 3).

The value of "Prob> F is less than 0.05 and indicates that model terms are significant.

In our case all the terms A, B, AB, A^2 and B^2 are significant terms of the model (see table 3).

"Prob> F values are greater than 0.1000 and indicates that insignificant terms can be excluded in the regression equation [6].

Table 3

ANOVA test table for response surface design material due tensile *PA 6.6 - 30% GF.*

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Model	**581.82**	**5**	**116.36**	**56.44**	**< 0.0001**
A-T_3-Temp.in metering zone (°C)	63.13	1	63.13	30.62	0.0009
B-T_4-Temp. in forming zone (°C)	37.53	1	37.53	18.20	0.0037
AB	115.56	1	115.56	56.05	0.0001
A^2	268.63	1	268.63	130.29	< 0.0001
B^2	138.00	1	138.00	66.94	< 0.0001

In the following regression equation results for a tensile stress mathematical model of the corresponding *PA 6.6-30% GF* material:

$$R_m(T_3, T_4) = -4057.85352 + 20.50976\, T_3 + 9.56852\, T_4 + 0.053750\, T_3 T_4 - 0.063044\, T_3^2 - 0.045187\, T_4^2. \quad (1)$$

where: R_m – tensile stress [MPa]
T_3 – temperature in metering zone [°C],
T_4 – temperature in forming zone [°C]

4. GRAPHICAL REPRESENTATION OF A RESPONSE SURFACE

In figure number 5 a response surface is graphically presented and corresponds to regression equation for tensile stress mathematical model. "(1),".

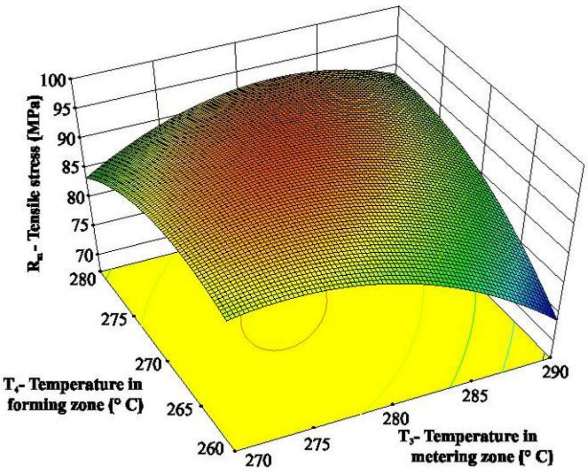


Fig. 5. Variation of tensile stress according to and temperature in a metering and forming zone (T_3 , T_4)

5. CONCLUSIONS

In order to achieve *PA 6.6 – 30 % GF* optimum melt homogeneity and maximum tensile stress, process temperature in those two areas must be properly set.

We may see that tensile stress minimal value is only obtained when difference between these two areas is maximum like: T_3= 290 °C and T_4= 260 °C. That result from forming zone is bigger than metering zone extrusion installation danger can appear and extruded profile calibration becomes hard to handle. Maximum tensile stress value is obtain when metering zone temperature is 280 °C and forming zone temperature is 270 °C. Besides a good material homogenizing because of a decreased temperature in profile zone we create an extrusion optimal pressure and an extruded profile good calibration. In conclusion, we may say that metering zone temperature must be 280 °C and chain zone temperature must be 270 °C. In order to obtain a better tensile stress for a reinforced fiber glass extruded polymer (*PA 6.6 – 30 % GF*), the required, forming zone temperature must be smaller than metering zone temperature. Between those two a big difference must not exist because an extrusion installation binding can appear and the technological can be compromised process. Researches will be extended for other degrees of reinforcement for a PA 6. 6 polymer [6].

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Cercetări teoretice şi experimentale privind optimizarea procesului de extrudare a materialelor polimerice armate cu fibre scurte (*PA 6.6 – 30 % FS*)

Produsele din poliamidă 6.6 armată cu 30 % fibre de sticlă (*PA 6.6 – 30 % GS*) sunt caracterizate prin proprietăți mecanice, termice şi tribologice performante. Îmbunătățirea acestor proprietăți se poate realiza prin optimizarea procesului de fabricație prin extrudare a produselor profilate. Temperatura de pre-lucrare în cazul procesului de extrudare are un rol determinant asupra calității produsului extrudat. În cadrul acestei lucrări este prezentat un model matematic al rezistenței la tracțiune, rezultat în urma optimizării, cu ajutorul analizei *ANOVA* (Analysis of variance) a temperaturilor de prelucrare în zona de omogenizare respectiv zona filierei. Zona de omogenizare şi zona de profilare (zona filierei), sunt două dintre zonele cele mai importante ale procesului de extrudare. Optimizarea temperaturilor în aceste zone duce la creşterea calității, productivității şi competitivității produselor extrudate.

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