Dependence of Polypropylene Yarn Mechanical Properties on Manufacturing Parameters

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Polypropylene (PP) multifilament structure is a valuable way to prepare yarns for medical application. In this study iPP multifilament yarns were prepared from iPP granules Moplen 462R by melt spinning technique COLLIN® CMF-100. The aim of investigation was to study and forecast mechanics indicators of threads being produced through the creation of mathematical model that define the association between yarn indicator and technological parameters of production. The samples were made using circular spinnerets with 24 holes. It has been shown that analysed technological parameters have a significant impact on the tensile characteristics of the PP multifilament yarns. The tensile properties of PP filaments are strongly influenced by their physical structure, which is controlled by the choice of the starting material and the fibre formation conditions.

Keywords: D – optimal designs, polypropylene, multifilament yarns, mechanical properties.

1. INTRODUCTION

Isotactic polypropylene (iPP) is among the most important commodity polymers. This polymer is a popular material for fibre manufacture due to its properties like low density, easy processability, excellent orientation characteristics, superior tensile properties, good chemical resistance, hydrophobicity, resistance to micro-organisms and the relatively inexpensive cost of production [1 – 5].

Textile fibres have to fulfill certain requirements that are primarily determined by the end use. Melt spinning process is an efficient way to produce multifilament yarns to many potential applications (filtration devices, automotive industry, agriculture, light industry, medicine, electronics industries, etc) [6 – 8]. The primary process variables of melt spinning process are: extrusion temperature, mass throughput per spinneret hole, cooling conditions along the spin line, the size and shape of the spinneret holes, spin line length, and take – up velocity of the filaments or filament drawing ratio. These process variables interact with the resin characteristics to control the processability and the structure and properties of the as – spun filaments. The important resin variables are those affecting the rheological and crystallization behaviour of the polymer. These include such basic issues as chain stiffness and mobility, and the sensitivity of the crystallization kinetics to the presence of molecular orientation [3, 9].

In common with the other synthetic fibers, the mechanical properties of polypropylene fibers are strongly influenced by their physical structures, which are controlled by both the choice of the starting material and the fiber formation conditions. The different conditions of the fiber formation during melt spinning and drawing processes cause different arrangements of the supermolecular structural elements, resulting in different fiber properties [10]. Some parameters, especially two throughput rate and take up velocity are typical characterized by researchers [11 – 13]. Melt spinning is a very complex process, with many variables; we will first describe a simple engineering analysis leading to a model of the melt spinning process, which will help in understanding the interrelationship of some variables. An understanding of how the effect of processing parameters on the morphology is crucial in optimizing the melt spinning process.

The fabrication stage was necessary to determine the effective technological spinning parameters, which allow obtaining thin PP yarns. Due to that influence of extrusion temperatures and melt pump speed on the resulting multifilament yarns thickness and on mechanical characteristics of multifilament yarns have been investigated. The aim of investigation was to study and forecast mechanics indicators of threads being produced through the creation of mathematical model that define the association between yarn indicator and technological parameters of production.

2. EXPERIMENTAL DETAILS

2.1. Experimental

Materials. The samples were spun from granules of iPP Moplen 462R (Basell Service Company B. V.). This type of iPP granules can be used for producing multifilament yarns. It is very narrow molecular weight distribution homopolymer, suitable for extrusion applications. Typical properties of this type of PP polymer are: melt flow rate (MFR) (230 °C/2.16 kg) – 25 g/10 min, melt volume flow rate (230 °C/2.16 kg) – 34 cm³/10 min, tensile stress at yield σ_y = 34 MPa, tensile strain at break ε_B > 50 %, tensile strain at yield ε_y = 10 %, flexural modulus FM = 1450 MPa. Glass transition temperature of PP is T_g = 15 °C ÷ –20 °C, dielectric constant at 10^6 Hz ε_r = 2.0 ÷ 2.4.

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Table 1. Constant processing parameter

Parameter	Extrusion				Spinning				
	Hole diameter, mm	Hole length, mm	Extruder pressure, MPa	Screw speed, rpm	Stretching godets temperature, ºC / speed, rpm				Drawing ratio
					No. 1	No. 2	No. 3	No. 4	
Unit	0.45	1.3	5	20	81 / 316	97 / 632	107 / 727	120 / 799	2.53

The iPP multifilament yarns were prepared on a COLLIN® CMF 100 laboratory melt spinning equipment. Single screw extruder (L/D = 25:1) includes seven heating zones in which the temperature can be independently and gradually fixed from 10 ºC up to 300 ºC. Four godets is driven separately for a speed range of (50 ÷ 800) m/min.

Experiments were carried out at three profile of extruder heating zones temperatures: T = 195, 233, 271 ºC and three extruder melt pomp speeds: n = 10, 18.7, 27.5 rpm. The samples were extruded using circular spinnerets with 24 holes diameter of them was 0.45 mm. Cooling of the extruded filaments was achieved with cross – flow air quenching at the temperature of 12 ºC. The as-spun filaments were four stages drawn with overall draw ratio of 2.53, in a continuous spin drawing process. All other processing parameters were kept constant (Table 1).

For the tests iPP multifilament yarns were conditioned not less than 48 hours at the standard atmosphere according to the standard i. e. at relative humidity of φ = 65 % ±5 % and temperature of T = 20 °C ±2 ºC. All the tests were made under standard conditions (ISO 139). The yarn linear density was determined using Zweigle L232 reeling machine. Linear density of the samples was determined according to ISO 2060:1994. Mass of iPP multifilament yarn in 200 m in length was determined. The test result was assumed as average of five measurements.

The yarn tensile tests were determined on the Zwick / Z005 universal testing machine using *testXpert*® software (ISO 2060:1993). The length between sinks was fixed at 250 mm and the crossbar speed at 500 mm/s for all tested yarns and at pretension of 0.5 cN/tex was used. The test result was average of 25 measurements.

2.2. Mathematical simulations

Yarn properties and application opportunities are affected by various controlled and uncontrolled factors: it is very difficult to assess the influence of all the parameters. It is well known that optimization technological parameters performing experimental testing is time consuming process and usually unsuitable for selection more effective processing parameters. One of possible way to eliminate this disadvantage is mathematical experiment design. This method facilitates the simulation of the process by choosing a technologically modes in order to obtain materials with the required properties. During this process more significant technological parameters can be selected and irrelevant eliminated. As a result of this design is mathematical model, which represents relationship between the criteria and selected parameters [14]. Initially, function simplified function can be presented as:

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

where y – is the optimization criterion (size controlled process); $x_1, x_2, ...x_k$ – are the factors linked with (y) criterion.

From the many factors influencing the yarn structure, its mechanical and other properties the main important are these two [7]:

1. extruder heating zones temperature (X_1);
2. melt pump speed (X_2).

In this study the investigation is described using D – optimal design for second – order model 2^k (k – number of variables, $k = 2$), a 2^2 factorial with one central point, because this plan enables to explore the maximum latitude of factors and enough to accurately assess the impact of selected factors. Plan choice was determined by two criteria: optimality criteria of plan for maximum efficiency; the appropriate number of rows in the plan.

The model matrix consisting $N = 9$ ($N = 2^k + 2 \cdot 2^{k-1} + 1$) lines used for process design is presented in Table 2. The both extrusion temperature at zero level was 233 ºC and for levels –1 and +1 step of 38 ºC (accordingly 195 ºC and 271 ºC) was applied. The zero level for melt pump speed was 18 rpm and step was 8.7 rpm (accordingly 10 rpm and 27.4 rpm).

Commonly a second – order model with k variables is represented by the equation [14]:

$$\hat{y} = b_0 + \sum_{i=1}^{k} b_i x_i + \sum_{i=1}^{k} b_{ij} x_i x_j + \sum_{i=1}^{k} b_{ii} x_{ii}^2 ; \quad (2)$$

where k – is the number of variables; b_0 – is the free member, b_i – is the linear coefficients, b_{ii}, b_{ij} – are the coefficient dual (pair) interaction; x_i, x_j – are the process parameters (controllable factor) of coded values during the experiment.

According to equation (2), second – order model for $k = 2$ is represented as square regression equation [16]:

$$\hat{y} = b_0 + b_1 x_1 + b_2 x_2 + b_{12} x_1 x_2 + b_{11} x_1^2 + b_{22} x_2^2 ; \quad (3)$$

where x_1, x_2 are controllable factor of coded values during the experiment.

After solution of the equation (3) defines a surface and allows determining the optimal design area.

Table 2. D – optimal design parameters

N	Coded values		Fair values	
	X_1	X_2	Extruder heating zones temperature T, ºC	Melt pump speed n, rpm/min
A1	+1	+1	271	27.4
A2	+1	0	271	18.7
A3	+1	–1	271	10.0
A4	0	+1	233	27.4
A5	0	0	233	18.7
A6	0	–1	233	10.0
A7	–1	+1	195	27.4
A8	–1	0	195	18.7
A9	–1	–1	195	10.0

The regression coefficients of mathematical model are calculated with the help of matrix method, using the software program EKSPLA created at the Department of Textile Technology, Kaunas University of Technology. Mathematical regression models to assess the homogeneity of variances and according to informativeness criteria (F_i). For new multifilament yarns design was use of regression models, which inequalities in the system have been met [14]:

$$C_j \langle C_t ; \quad (4)$$

where C_j, C_t – are the calculated and tabular Cochrain's criteria.

Insignificant regression coefficients are determined by calculating a confidence interval (Δb) for all regression coefficients [15]:

$$\Delta b = t_{0.95}(f_E)\sqrt{S^2(b_i)} ; \quad (5)$$

where $t_{0.95}(f_y)$ – is the Student's coefficient; $S^2(b_i)$ – is the variance of regression coefficients.

Student's criteria depends on the number of degree of freedom (f_y) [14]:

$$f_E = N \cdot (n-1); \quad (6)$$

where N – is the number of plan lines; n – is the number of measurements per test (n = 25).

Regression equation coefficients are considered significant if:

$$b_0, b_i, b_{ii}, b_{ji} \geq \Delta b ; \quad (7)$$

Mathematical model is considered informative if [14]:

$$F_i \rangle F_{it} ; \quad (8)$$

where F_i, F_{it} – are the calculated and tabular (at a specified probability level α = 0.95) Fisher's criteria. Otherwise, the model is no informative.

Prediction made by the creation and interpretation of the dependence of the two multifilament yarn production parameters. For this purpose, create second – order three – dimensional surfaces describing these dependencies. Dependence presented graphically with fair X_1, X_2 factor values.

3. RESULTS AND DISCUSSION

The linear density and other characteristics of the extruded samples are given in the Table 3.

Analyzing the data of Table 3, it was found that using a higher polymer extrusion temperature smaller linear density of the yarn can be achieved. It was observed that increasing the extrusion temperature of polymer from 195 ºC to 271 ºC degrees, the linear density of iPP yarns decrease linearly. Difference between samples No A1 and A7 is 11.76 %, between samples No A2 and A8 – 13.04 % and between samples No A3 – A9 is 18.46 %.

The effect of extruder melt pomp speed on deformation behaviour of iPP multifilament' is also seen from the Table 3 data. If the melting speed is high, the linear density of the fiber will also be high. However, the increase the melting pomp speed of the polymer results in an increasing elongation at break and linear density. Maximum relative elongation at break, work of break has yarns produced at the highest parameter values of temperature (271 ºC) and extruder melt pump speed (27.4 rpm): its multifilament's No A1, A4 and A7. The ratio of these samples varied from 3.0 % to 18.8 %.

Breaking tenacity is important indicator in terms of yarns for many purposes. Breaking tenacity of yarns should be the higher; it makes possible to use thinner yarns with the same strength characteristics. The highest specific breaking force values showed the samples No A9, A8, A6 and A3. Three of these samples (No A3, A6 and A9) were produced using the optimum polymer extrusion temperature 195 ºC and melt pump speed 10 rpm.

Changes of temperature effect viscosity and shear effects due to relaxation of molecular link. Analysis showed that by decreasing strength of yarns the strain of yarns increased a reason for that can be structural changes of iPP yarns. Subjected to high temperature of polymer molecule decrease degree of polymerization, changing the orientation of molecules. [11, 13] The reduction in the degree of polimerization of macromolecules decreases the breaking force. Thus, the extrusion temperature of polymer affects the linear density, breaking force and other mechanical characteristics.

In general, fibres had the highest tensile module. Multifilament yarn tensile modulus varied within a wide range from 52.18 cN/tex (A7) to 264.08 cN/tex (A9). It was found that this index is the largest of yarns, which produced at the polymer temperature 195 ºC and the minimum pump speed 10 rpm. If the sample has a low tensile modulus, it means it is easily deformed and conversely if the sample has a high tensile modulus, it means it resists deformation. The yarn samples No A1, A2, A4, A5 and A7 are not are not sufficiently strong and tough compared with other samples, but are very tensile. Variation range of tensile modulus varied from 2.3 % to 5.7 %. Area underneath its curve is a lot larger than the area under the all other sample's curve. So it can absorb a lot more energy than the other sample can.

The regression coefficients of equations, describing the multifilament yarn mechanical characteristics dependence on yarn production parameters are given in the Table 4.

From the data presented (Table 4) it can be noticed that multifilament yarn production technology parameters (X_1 – extrusion temperature, X_2 – extruder melt pump speed) affect the mechanical characteristics of the tested yarns.

Table 3. Mechanical characteristics of iPP yarns

Characteristics	A1	A2	A3	A4	A5	A6	A7	A8	A9
Breaking force, cN/tex	10.43 ±2.8	10.72 ±5.9	14.05 ±6.8	10.67 ±4.8	11.04 ±5.3	14.58 ±8.2	12.26 ±5.8	17.95 ±6.6	20.59 ±5.4
Elongation at break, %	86.62 ±1.7	78.77 ±2.1	63.22 ±1.8	83.96 ±1.8	68.20 ±2.0	52.38 ±2.1	75.50 ±2.2	72.49 ±1.9	41.88 ±2.0
Work of break, J	1.74 ±6.9	1.06 ±8.8	0.72 ±6.9	1.47 ±3.4	0.96± 5.6	0.72 ±8.3	1.81 ±7.8	1.63 ±6.2	0.65 ±3.1
Tensile modulus, cN/tex	65.23 ±4.5	115.14 ±4.0	262.29 ±1.4	69.82 ±5.7	95.73 ±3.1	231.57 ±2.0	52.18 ±4.1	99.32 ±2.3	264.08 ±1.7
Linear density, tex	30.00 ±2.5	20.00 ±2.0	10.6 ±2.0	32.00 ±1.7	21.00 ±3.9	11.6 ±6.3	34.00 ±2.3	23.00 ±1.9	13.00 ±5.2

Table 4. The regression coefficients of iPP multifilament yarns

Coefficients	*RB*, cN/tex	*εB*, %	*WH*, J	*E*, cN/tex	*T*, tex
b_0	11.57	73.11	1.08	96.29	21.18
b_1	(–2.51)	6.46	(–0.09)	4.52	(–1.57)
b_2	(–2.64)	14.27	0.49	(–95.1)	10.13
b_{12}	(1.05)	(–1.80)	(–0.04)	(3.71)	(–0.40)
b_{11}	2.50	(1.57)	0.22	10.67	(0.23)
b_{22}	(0.53)	(–7.40)	(–0.03)	54.14	(0.53)

* – insignificant regression coefficients in parentheses.

The regression coefficients of statistical approach, in many cases are significant, except linear density and breaking force parameters, where four coefficients of expressing linear density and breaking force dependence are insignificant. Investigating the breaking force of multifilament yarns dependencies it was found, that a significant is 5 of the 6 regression coefficients. Tensile modulus, work of break and technological parameters dependence of the describing equation three regression coefficients are significant.

Another important phase of the study is to set informativeness of iPP yarns mechanical characteristics. Comparison of iPP multifilament yarns mechanical characteristics of mathematical models informativeness criterion F_i with criterion F_{it} is presented in Table 5.

As it can be seen from the data presented in Table 5, mathematical models of four mechanical characteristics: breaking force, elongation at break, tensile modulus and linear density are informative.

Table 5. Comparison of mechanical properties of yarns of informativeness criterion F_i with criterion F_{it}

Characteristics	F_i	F_{it}
Breaking force *RB*, cN/tex	5.57	4.15
Elongation at break *εB*, %	4.95	4.82
Work of break *WH*, J	3.77	4.82
Linear density *T*, tex	2424.81	4.15
Tensile modulus *E*, cN/tex	35.65	4.82

Graphic dependences of iPP multifilament yarns technological production parameter values are presented below in Figures 1 – 4. Until now no studies were performed that determine the optimal value for extrusion parameters such as melt pump speed in producing PP multifilament fibers. The study showed that the use of lower extruder pump speed in multifilament's production it makes possible to shrink PP yarns elongation at break.

iPP multifilament yarns having a minimum breaking strength at such encoded factors values: $X_1 = 271$, $X_2 = 27.4$. The maximum breaking force of iPP yarns fixed at coded values: $X_1 = 195$, $X_2 = 10$ (Fig. 1).

Breaking force of iPP multifilament yarns varies, as increasing the extruder pump speed breaking force decreases up to a certain minimum value in the field and it reached starts to decline.

The elongation at break increases by increasing the extruder pump speed and temperature (Fig. 2). Maximum elongation at break is at the maximum pump speed and temperature parameter values.

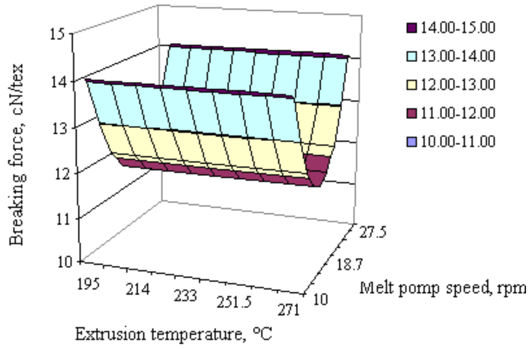

Fig. 1. Dependence between iPP multifilament yarns breaking force and manufacturing parameters

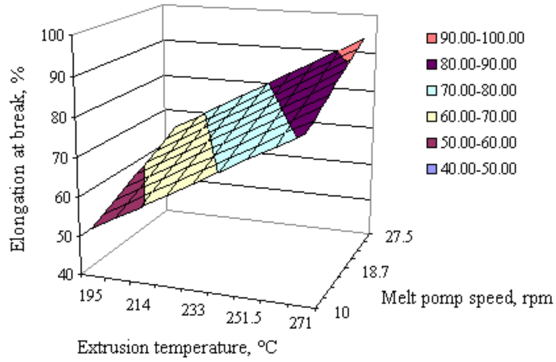

Fig. 2. Dependence between iPP multifilament yarns elongation at break and manufacturing parameters

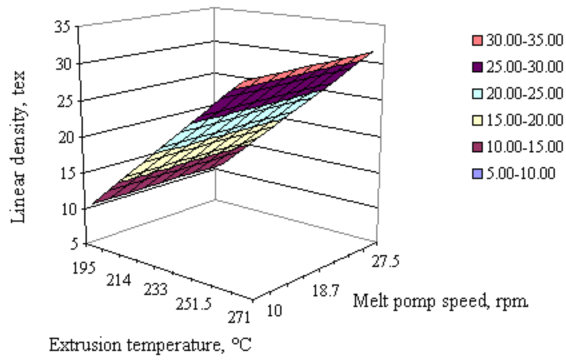

Fig. 3. Dependence between iPP multifilament yarns linear density and manufacturing parameters

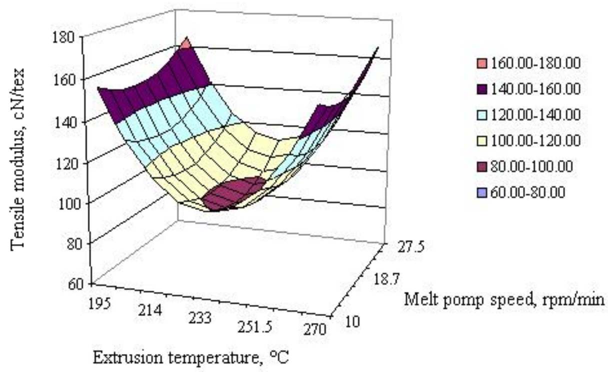

Fig. 4. Dependence between iPP multifilament yarns tensile modulus and manufacturing parameters

Processing temperature has a great importance on the fiber spinning process since it affects not only the polymer viscosity but also the heat transfer between the polymer and air. For comparison [12, 16], the simulations were conducted with three different processing temperatures, 180 ºC, 230 ºC and 260 ºC. With the increase of processing temperature, the final fiber diameter becomes smaller. The higher temperature results in a lower melt viscosity, which makes polymer melt more easily to be attenuated under the air drag force. Meanwhile, the higher processing temperature also provides longer time for attenuation before the polymer melt is solidified.

Other studies [17, 18] have also revealed that the temperature has a considerable effect on the stability of extrusion and quality of fibres, and that fiber diameter decreases with the increase of processing temperature (up to 210 ºC – 290 ºC).

The model describing work of break and X_1, X_2 factors dependence was noninformative. Particularly high informativeness of mathematical models were between X_1, X_2 factors and characteristics of tensile modulus ($F_i = 2424.81$) and linear density ($F_i = 35.65$).

Elongation at break of iPP multifilament yarns is minimal when the coded factor values were $X_1 = 195$, $X_2 = 10$. The maximum value of elongation at break of iPP yarns fixed at coded values $X_1 = 271$, $X_2 = 27.4$.

Linear density directly depends on the production parameters (Fig. 3). iPP multifilament yarn linear density decreases increasing the extruder pomp speed and reducing the extrusion temperature. The minimum value of the yarn linear density, when the coded factor values were $X_1 = 271$, $X_2 = 10$. The maximum value of linear density of iPP yarns fixed at coded values $X_1 = 195$, $X_2 = 27.4$.

It can be argued that the tensile modulus of iPP multifilament yarns varies consistently; i. e. with decreasing temperature tensile modulus decreases to a certain minimum value in the field and it reaches begins to rise.

The model of multifilament yarn formation can be a useful tool not only in yarn research but also in the production process. The simulations can never replace the experimental work and the empirically collected experiences, but they can powerfully support the laboratory and industrial research in order to avoid time consuming and / or expensive investigations regarding process modification, extension, or optimisation, respectively. Especially the last mentioned task of optimisation is typical for engineering procedures in melt spinning, the prediction of resulting multifilament yarn properties after changing the process parameters.

4. CONCLUSIONS

The influence on the mechanical properties of iPP multifilament yarns has been investigated. iPP was chosen for its valuable properties such as chemical and biological resistance, low density, good resistance for extension, good tear and abrasion resistance, and low price.

Using mathematical experiment design, regression equations were derived that allow predicting and exploring dependencies of technological manufacturing parameters, linear density and complex mechanical properties of polypropylene multifilament yarns. In this study these mechanical characteristics: breaking force, elongation at break, work of break, linear density and tensile modulus were analyzed and forecasted.

Analysis of the design methods and forecasting results showed that analysed technological parameters (extrusion temperature – X_1 and extruder melt pump speed – X_2) have a significant impact on the tensile characteristics of the iPP multifilament yarns. It has been found that consisting of five mathematical models, generalized yarn characteristics and technological parameters, four of them are informative. Breaking force and tensile modulus increases with decreasing of extrusion temperature and extruder melt pump speed, meantime elongation at break, work at break and linear density decreasing.

The effect of extrusion parameters on yarn characteristics shown that a low linear density of iPP yarns could be obtained at higher polymer melting temperature (271 ºC) combined with low extruder pump speed (10 rpm/min). To get even thinner iPP yarns should try to change drawing ratio between godets. However, there is still room for further optimisation of the melt – spinning process in order to produce the required structure and mechanical properties of the yarns.

The tensile properties of iPP filaments are strongly influenced by their physical structure which is controlled by the choice of the starting material and the fibre formation conditions. It is important to note that produced iPP yarns are strong enough, that to withstand pressure for example of the abdominal cavity in laparoscopic abdominal surgery. Mechanical properties of iPP yarn allows to select a more appropriate regime for spinning yarns intended to be used for specific purpose in medicine.

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