

Product Innovations in the Manufacturing Processes of Special Composites

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Abstract

At present, in order to maintain a sustainable production process and achieve real business competitiveness, attention needs to be paid focused planning of innovations in production systems. Major innovations are the product innovations where significant improvements can be reflected in technical specifications and new materials, that are significantly different from the original products, thus replacing the products and materials that are absent for the needs of different sectors of industry.

In our research, we have focused on product innovations in the field of modification of existing polymeric materials for the preparation of new special composites for various industries. Based on the study of the rheological behavior of polymer mixtures and the determination of the optimal technological conditions of the manufacturing process (melting of polymer mixtures and subsequent spinning), the complex evaluation of the formed supramolecular structure and the characteristics of the fiber-forming composites we have experimentally prepared new types of special composites.

Keywords: product innovations, manufacturing processes, special composites.

Introduction

The manufacturing process is a creative process and its function is the creation of planned utility values. It represents the main activity of the production company. The nature of the manufacturing process is based on the basic aspects of the production process, the product complexity, the impact of human, the environment and the maturity of the technology, the technology used, the assortment of products, the way and the repeatability of the production in the subsequent context of the economic efficiency

of the production process (Kavan 2002; Šaderková 2015; Polanecký and Lukoszová 2016). The manufacturing process influences decisively the efficiency of the business and the competitive ability of its products. The realization part of the production process is the most important stage in the creation of final products with minimization, respectively optimization of inputs and the expected maximization of outputs (Caha 2017; Vochozka 2011; Lindermann et al. 2013).

At present, in order to maintain a sustainable production process and achieve real business competitiveness, attention needs to be paid focused planning of innovations in production systems. Solving this relatively complex problem is to focus attention on the manufacturing process that delivers market prospective products from new materials with minimal adaptation of technological processes and repeatable economically efficient and environmentally friendly production (Rusnáková et al. 2014).

The aim of the paper is to determine the optimal conditions of technological preparation of new innovative product - polymer mixtures by studying the process of melting rheology in the manufacturing process of subsequently formed fiber-forming bicomponent polymers applied to new special composite materials in the context of economic efficiency of production innovations.

Innovation in management production of composites

At present, it is crucial for companies to remain in the global market and to become more competitive in their position through the implementation of innovation policy. Major innovations are the product innovations where significant improvements can be reflected in technical specifications and new materials, that are significantly different from the original products, thus replacing the products and materials that are absent for the needs of different sectors of industry. Replacement of existing materials in the production of final products by new, forward-looking materials contribute to preserving and increasing market share and gaining new markets (Vochozka et al. 2015; Kampf et al. 2017).

It is expected that, from the point of view of a company's market position, product product innovations should achieve the positive image of the company marketing them, thus enhancing the preservation of the competitiveness of an industry in the given industry sector, or earning early profits by acquiring a temporary monopoly of production costs or realization higher prices. New products that have been prepared on the basis of patents and utility models can provide a longer-term competitive exit compared to marketing mix tools such as pricing or advertising. At the same time, it is necessary to point out the disadvantages of first initiators and product innovation implementers, including higher research costs, market uncertainty induced by changing the needs of product purchasers (Yang 1991; Kavan 2002; Vochozka et al. 2015; Yaping et al. 2017).

At present, the field of manufacturing of special composite materials is considered to be key in the view of new, safer, technically more environmentally friendly and economically more efficient applications for various industries (especially engineering, construction,

automotive, chemical, rubber, textile) (Lindermann et al. 2013; Rusnáková et al. 2014; Ružinská 2014b-d; Badida et al. 2016).

The preparation of new products with a range of performance features that outweigh the existing products and materials can be successfully leveraged from research and transfer research into supported activities, which is a compromise in product innovation policy (Ružinská 2014b).

New types of materials for preparing composite materials with a wide range of functional properties (with fortified thermal insulating, sound-insulating, multiplied by mechanical, thermal properties, reduced flammability, modified utility features, environmentally friendly, economic sustainability) and higher added value are currently being preferred, prepared by modifying and optimizing the manufacturing process from an existing assortment of fiber-forming polymers for automotive, civil engineering, woodworking, chemical and other sectors of industries (Lindermann 2013; Ružinská et al. 2010, 2014a-d; Rusnáková et al. 2014).

New trends in fibers development are going towards widening the range of fibers from the classic types of fiber-forming polymers. In fibers industry now increasing attention is paid to the polymeric system type "polymer-polymer", usable in special type of composite materials. Polymer blends where a large number of mixtures with a wide range of performance can be prepared from a limited number of ingredients are becoming more important (Scholz et al. 1989; Steinmann et al. 2001; Zheng et al. 2007; Ružinská et al. 2014a; Staropoli et al. 2017).

Based on the study of the rheological behavior polymer mixtures and the determination of the optimal technological conditions of the manufacturing process we have experimentally prepared new types of special composites.

Theoretical foundations of rheology polymer blends for preparation new type of composite

Polymer blends are multicomponent polymer systems obtained by technological methods of mixing two or more polymers. They form a group of polymeric materials, the relevance of which is recently growing. This is due to the fact that it is possible to produce a large number of mixtures with a wide range of performance from limited number of components (Cook et al. 2005; Gold et al. 2017).

They display a distinctive phase structure, which is always non-equilibrium (at thermodynamic equilibrium must occur a complete separation of components) and is dependent on the method of preparation. Polymer blends are different from base polymers, from which they have been prepared. Preparation of polymer blends belongs to one of the physical modifications, which result in changes in the supramolecular polymer structure and also a change in the original properties of the base polymers occurs. This can lead to improved fiber properties while retaining its other features (Cook et al. 2005; Gold et al. 2017).

Practically the most important method of preparing polymer mixtures is mixing the components in the melt. If mixing lasts long enough, and the system does not take chemical reactions a dynamic equilibrium between breakup and coalescence of particles is created in the mixture. Each operation of mixing must have a process of distribution and dispersive mixing that is a mixing operation must be carried out such that the individual components (Scholz et al. 1989; Staropoli et al. 2017).

Processes of polymer blends can be separated into operations involving formation of mixtures and processing operations, wherein the resulting mixture is extruded or formed into a desired shape. Mixing the polymers via extrusion is one of the easiest methods for preparing blends. This type of treatment is complicated by limited miscibility of polymers, which among other factors, is dependent also on their low configuration entropy of mixing (Cook et al. 2015; Mazidi et al. 2015).

Polymers suitable for the preparation of fiber-forming mixtures (polypropylene/polyamide - PP/PA₆) are thermodynamically intolerant systems preventing formations of a homogeneous system. Thus they cause the formation of the boundary as a result of the increase in enthalpy of mixing polymers. The stability of the created phase morphology is highly dependent upon the addition of interfacial agent, ensuring improved dispersibility and improved adhesion at the interface, thus improving the physical and mechanical properties of the polymer system (Ružinská 2010, 2014a-d; Mazidi et al. 2015; Gold 2017).

Deformation and flow of polymers is formed under the influence of external stress. The relationship of these parameters is studied and described by the rheology of polymers. Rheological properties are evaluated in terms of the orientation of the macromolecules in the flow deformation, which is related to the formation of the structure of the polymer and filament current and the processing properties of the polymer (Part et al. 1990; Tol et al. 2004; Van Hemelrick et al. 2004).

The rheology of polymer blends depends not only on the molecular structure, but also on the properties of the studied systems. Rheological properties of polymer blends are determined by two groups of factors (Han et al. 2007; Staropoli et al. 2017):

- factors related to the flow conditions (temperature, pressure, fluid friction flow parameters)
- factors related to the material itself and its status (molecular weight, polydispersity, chemical composition, additives).

The aim of the study of rheology is to find a unified theory for all kinds of distortions and relationship of strain to the exerted tension. It is an expression of relation between stress and strain rate. This functional dependence to describe the flow of non-Newtonian fluid is non-linear and generally can be expressed by the equation (Han et al. 2007):

$$\tau = \eta \cdot \dot{\gamma} \quad (1)$$

where τ - represents the shear stress

η – non-Newtonian viscosity

γ - shear rate.

The melts of fiber-forming polymers behave as so called pseudoplastics fluids, the viscosity of which decreases with increasing shear stresses, which is caused by the breaking up of the original structure (due to the orientation of asymmetrical particles and breaking up of agglomerates) (Scholz et al., 1989; Han, 2007).

So-called power law satisfactorily explains flow curves and their behavior in the range of 1 - 2 shear rate:

$$\tau = K \cdot \gamma^n \quad (2)$$

The exponent n in the so-called power law is less than 1 for pseudo-fluids (for a Newtonian fluids $n = 1$) than in the power law passes to Newton's law of flow and the *consistency coefficient* K is equal to the dynamic viscosity η (Han, 2007).

Pseudoplastic liquids are divided into genuine pseudoplastic and structurally viscous liquids. These are fluids with variable viscosity. Nonlinear behavior of non-Newtonian fluids is explained by their structural viscosity. It is the change in viscosity with increasing shear stresses or deformation rate of the structure (Steinmann et al. 2001; Staropoli et al. 2017).

Among the basic characteristics of the rheology of the polymer melts viscosity belongs to one which is affected significantly by the factors such as temperature, polymer molecular weight, molecular weight distribution, shear values, additives in the polymer. Rheological properties of polymer melt mixtures are determined by the nature of the individual components as well as by the process of forming structures in the flow conditions. Rheological characteristics of the mixed melt in comparison to the properties of its components change significantly, and these changes usually are not subject to the rule of additivity (Han 2007; Mazidi et al. 2015).

From several literature sources it is known that for the evaluation of the rheological properties of polymer blends and to study the effect of interfacial agent to enhance the dispersion and reduction of heterogeneity, as well as the impact on the intermediate stages of the structure and characteristics of the mixtures, it is necessary to select the optimum conditions (manufacturing temperature) for preparing the fibers and their further focus (Cook et al. 2005; Zheng et al. 2007; Ružinská et al. 2014a; Gold et al. 2017; Staropoli et al. 2017).

In accordance with the literature (Pötschke and Paul 2003; Tol et al. 2004; Han 2007; Robertson 2007; Zheng et al. 2007; Mazidi et al. 2015; Gold et al. 2017) we can assume that the increasing homogeneity of dispersion was due to increased technological compatibility of the components of the mixture. The degree of dispersity of the mixture increases because more interactions of macromolecular constituents during deformation are created. This confirms the crucial role of interfacial agents in increasing the tolerance of the polymer mutual thermodynamically intolerant system.

Materials and Methods

In experimental part were prepared polymer blends (mixtures) by remelting a mechanical mixture of granules of polypropylene and polyamide, resp. interfacial agent copolymer polypropylene – maleic anhydride using twin screw devices for various experimental determination manufacturing temperatures (from phase structure polymers).

In order to study the impact of the ratio of the components and contents of interfacial agent these series of mixtures were prepared:

Mixtures of various proportions of the components of polypropylene and polyamide 6. Mixtures prepared without interfacial agent were labelled PP/PA₆ and mixtures prepared by mixing polypropylene with polyamide 6, modified by 4% copolymer PP-MAH, were identified as PP/PA_{6M}. The following mixtures were prepared without addition interfacial agent: PP/PA₆ 90/10 and 80/20; PP/PA_{6M} 90/10, 80/20, 70/30, 60/40, 50/50.

Characteristics of used raw materials

For the preparation of polymer blends we used these raw materials:

- polypropylene, granule type TATREN (melt flow index IT = 30g/10 min)
- polyamide 6, silk type (molecular weight $M_w = 29300$)
- interfacial agent: copolymer polypropylene - maleic anhydride (PP-MAH).

Determination of rheological properties of polymers using GÖTTFERT capillary viscosimeter

Rheological properties were studied on semi-automatic discharge plastometer (capillary viscosimeter) from company GÖTTFERT, type MI 3564. The melt is extruded from the plathometer by a piston with weights that are placed in top of the device. A photocell measures the stream-time of a polymer of constant volume that is being extruded.

Conditions of the measurements:

Applied shear stresses: $\tau_1 = 3,997 \cdot 10^3$ Pa, $\tau_2 = 7,368 \cdot 10^3$ Pa, $\tau_3 = 1,224 \cdot 10^4$ Pa, $\tau_4 = 1,746 \cdot 10^4$ Pa, $\tau_5 = 2,103 \cdot 10^4$ Pa, $\tau_6 = 2,499 \cdot 10^4$ Pa, $\tau_7 = 3,025 \cdot 10^4$ Pa

Time point of the polymer blends: 5 minute

Flow conditioning: 1 minute

Temperature : 250, 260, 270 °C

Diameter of capillaries (d_0): $1 \cdot 10^{-3}$ m

Capillary length (l_0): $30 \cdot 10^{-3}$ m

Piston diameter (d_1): $9,95 \cdot 10^{-3}$ m.

Determination of densities of pure polymers and polymer mixtures

Within the rheological measurements, we evaluated the density of pure polymers and polymer mixtures as a parameter reflecting molecular structure using the capillary viscometer type GÖTTFERT, type MI 3564.

We used measures of volumetric flow of extruded melt (in the form of cuttings) and by simple conversion we obtained density values according to the relation:

$$\rho = \frac{m}{V} \quad (3)$$

where ρ - is the density of the polymer, ρ - mass flow of the extruded melt, V - volume flow of the melt.

Results

Evaluation of rheological properties of polymers using a capillary viscosimeter

Flow properties of polypropylene-polyamide 6 polymer system were evaluated using a capillary viscometer GÖTTFERT with automatic subtraction of the flow time of the melt volume unit. Rheology properties of polymers and polymer mixtures (blends) were evaluated under different processing conditions (processing temperature: 250, 260, 270 °C).

Measurement conditions were selected to be as close as possible to resemble conditions of melt flow in the fiber forming process. During rheological measurements, we used a capillary with a ratio $(L/d) = 30$ to reduce the pressure loss resulting from input factors.

Thirty measurements were carried out at each shear stress value from which the arithmetic average was calculated. From the average value of the measured time, we calculated shear velocity according to the relation:

$$\gamma = \frac{32 V}{\pi d^3} = \frac{8 d_1^2 h}{d^3 t} \quad (4)$$

where d_1 - is the diameter of the piston, d - diameter of the capillary, h - height of the cylinder of ejected string, V - volume flow of extruded polymer melt, t - time in (seconds).

Shear stress was calculated according to the equation:

$$\tau = \frac{z g r_0}{2 l r_1^2} = 1051,89 \cdot z \quad (5)$$

where z - is the weight in kg, r_0 - the radius of the piston, l - capillary length, g - gravitational acceleration, r_1 - the radius of the capillary.

For the evaluation of the flow properties on capillary viscometer we used a mixture of PP/PA_{6M} with a changing content of the dispersed component (polyamide containing 4 % wt. of interfacial agent PP-MAH) in the range of 10 to 50 %. To assess the effect of interfacial agent to flow parameters of the mixtures we evaluated PP/PA₆ mixture prepared without interfacial agent containing polyamide 6 in the amount up to 20 % wt.

Mixtures prepared without the interfacial agent, in the abovementioned conditions with more than 20 % wt. of polyamide phase, were very badly workable and practically not forming any fibers. From the results of rheological measurements of mixtures prepared at temperatures of 250 to 270 °C, we compiled flow curves (Ružinská et al. 2014a). Rheological behaviour of pure polypropylene and polymer blends have linear shape corresponding to the power law. Based on the assessment of the rheological behavior of polymer melting, that the flow of the melt has mixed pseudoplastic character similar to pure polypropylene.

The flow properties of mixtures with different organic ingredients can be described by Ostwald de Waele rheological models (Han 2007; Ružinská et al. 2014a, d). Power type parameters of equations for the flow of a mixture containing 10 to 50 % wt. of polyamide component prepared with interfacial agent are shown in Table. 1.

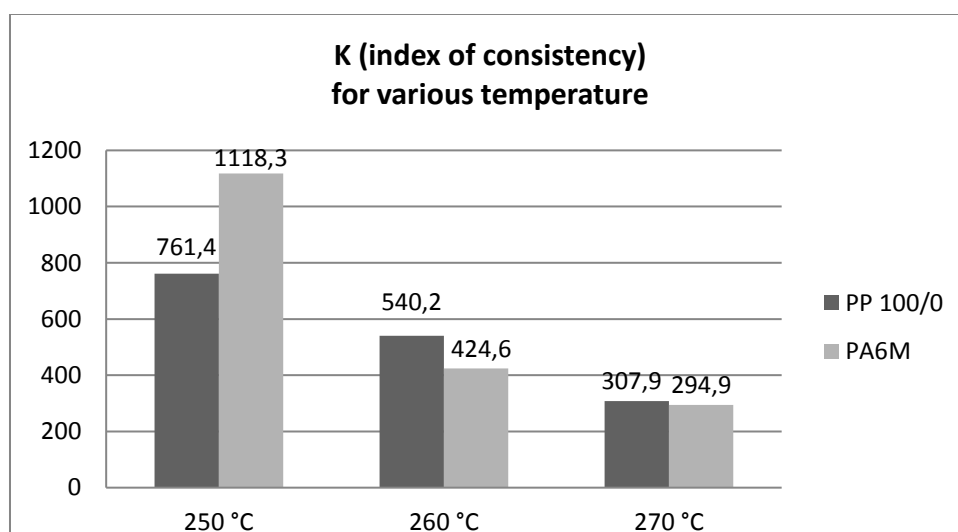
Table 1: Parameters of Ostwald de Waele model (n, K) for pure polymers and mixtures prepared with different content of the polyamide component

SAMPLE	250 °C		260 °C		270 °C	
	n	K	n	K	n	K
PP 100/0	0.64	761.4	0.64	540.2	0.73	307.9
PA _{6M}	0.62	1118.3	0.78	424.6	0.82	294.9
PP/PA _{6M}						
80/20	0.67	974.9	0.64	745.1	0.69	520.9
70/30	0.76	868.9	0.63	877.2	0.67	630.9
60/40	0.79	912.4	0.64	1013.8	0.72	565.9
50/50	0.87	858.6	0.58	1385.9	0.71	682.2

Source: Author

Notes: n – index of pseudoplasticity, K – index of consistency

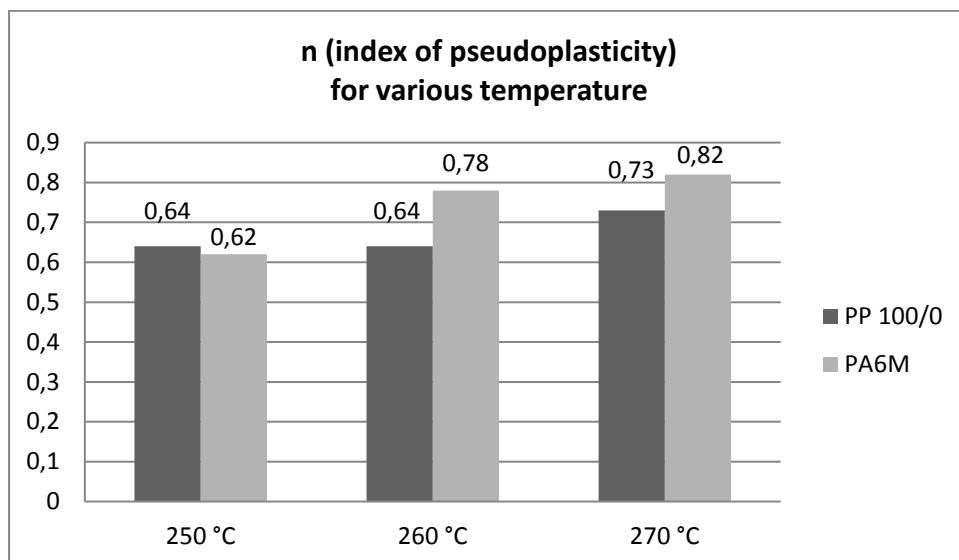
Figure 1: Comparison of indexes of consistency of pure PP and modified PA_{6M} under different conditions of preparation



Source: Author

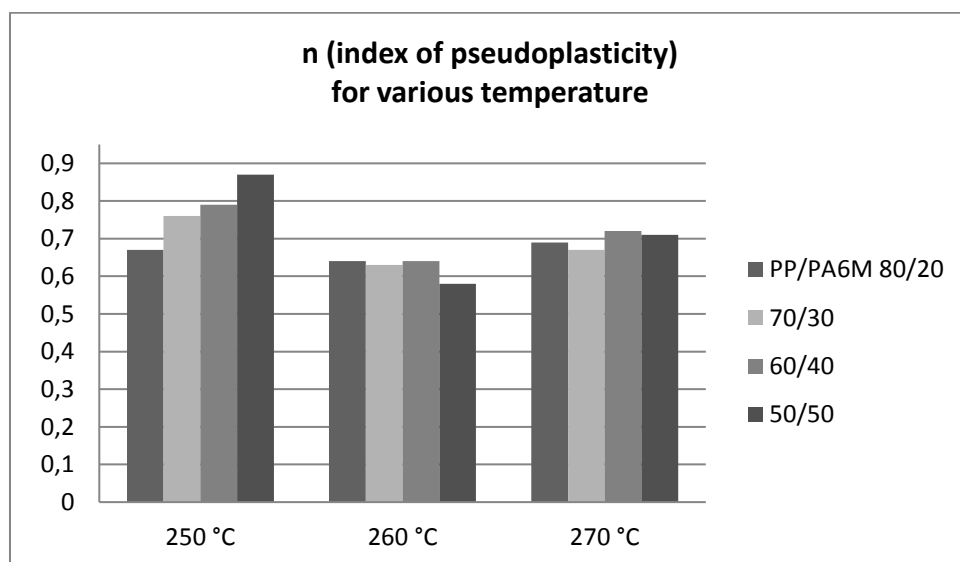
In the Figure 1 and Figure 2 are presented comparison of indexes of consistency and pseudoplasticity of pure PP and modified PA_{6M} under different conditions of preparation – 3 various manufacturing temperatures (results from Table 1).

Figure 2: Comparison of indexes of pseudoplasticity of pure PP and modified PA_{6M} under different temperature of preparation



Source: Author

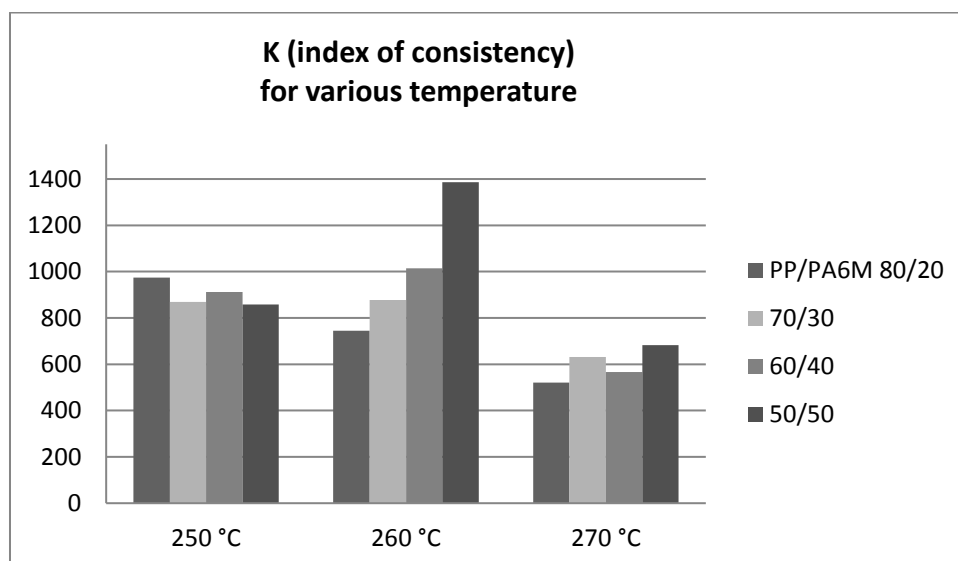
Figure 3: Comparison of indexes of pseudoplasticity of polymer mixtures with variable polyamide portion under different temperature of preparation



Source: Author

In the Figure 3 and Figure 4 are presented comparison of indexes of pseudoplasticity and consistency of polymer mixtures with variable polyamide portion under different temperature of preparation (results from Table 1).

Figure 4: Comparison of indexes of consistency of polymer mixtures with variable polyamide portion under different temperature of preparation



Source: Author

Non-Newtonian flow index values of mixtures do not significantly differ from pure polypropylene and a clear dependence on the content of the dispersed component was not shown. The differences, however, occurred at the comparison of values for mixtures prepared by mixing the pure components respectively using interfacial agent.

Table 2: Comparison of parameters of Ostwald de Waele rheological model for the mixture of polypropylene - polyamide 6 (80/20) with the interfacial agent and without the use of intermediate stage

SAMPLE	250 °C		260 °C		270 °C	
	n	K	n	K	n	K
PP/ PA ₆ 80/20	0.52	717.9	0.57	717.9	0.51	929.4
PP/PA _{6M} 80/20	0.67	974.9	0.64	745.1	0.69	520.9

Source: Author

According to the evaluated rheology values in Table 2 it is clear that the decrease of the parameter n , that is the largest variation from Newtonian flow was in mixtures prepared without the use of interfacial agent.

In the following stage, we monitored the change in apparent viscosity of the mixtures depending on the composition and content of the interfacial agent. The measured and evaluated results are shown in Table 3.

Table 3. Calculated values of the difference of the actual and additive values of apparent viscosities of polymer mixtures

MIXTURE	$\Delta\eta = \eta_{exp} - \eta_{adit}$			
	260 °C	260 °C	270 °C	270 °C
	$\tau_1=1,7 \cdot 10^4$ (Pa)	$\tau_2=2,5 \cdot 10^4$ (Pa)	$\tau_1=1,7 \cdot 10^4$ (Pa)	$\tau_2=2,5 \cdot 10^4$ (Pa)
PP/PA ₆ 90/10	7.2	3.2	7.2	10.3
80/20	0.6	0.9	4.7	7.4
PP/PA _{6M} 90/10	20.3	3.3	23.6	20.9
80/20	40.5	26.3	40.1	40.6
70/30	55.1	33.6	46.1	47.6
60/40	93.5	57.2	57.6	53.5
50/50	123.5	107.8	80.2	82.7

Source: Author

Discussion

Rheological properties of polymer melt are one of the decisive processing properties of the polymer in fiber forming processes including the structures and properties of the final product. From measured and evaluated rheological characteristics the Ostwald de Wael model (n-index of pseudoplasticity and K-coefficient of consistency and parameters of flow curves – shear velocity and shear strengths) of the experimental prepared bicomponent polymer mixtures showed that the temperature of 260 °C is optimal in the melting manufacturing process of the bicomponent polymer blends from the three usual technological temperatures (250, 260, 270 °C).

This finding confirmed the rheological characteristics of PP/PA₆, PP/PA_{6M} 80/20 (Table 1, 2 and Figure 1-4) at the temperature 260 °C, which are almost identical to the original polymers (PP, PA₆), but thermodynamically insensitive and addition of the MAH interfacial reagent (maleic anhydride) is required for compatibility.

The consistency coefficient - K is proportional to the dynamic viscosity of the pseudoplastic polymers and is one of the most important parameters in the technological processing of the polymerization process. This parameter is conditional to processing temperature and consequently influences all other rheological characteristics. From the measured and evaluated results of the consistency coefficient, it emerged that at the temperature of 260 °C the composition of thermodynamically most suitable polymer mixtures was balanced. When comparing the two variants of the blends PP/PA₆ 80/20 and PP/PA_{6M} 80/20 prepared, we prefer a system with the presence of a MAH interfacial agent, which also showed the viscosity values (Table 3).

However, by using the interfacial agent it is possible to prepare PP/PA₆ composite fibers in full concentration ratios. PP-MAH interfacial agent increases the degree of dispersion of the dispersed phase in the matrix of polypropylene, by which we get increased thermodynamic compatibility of the polymer components of the mixed fibers.

The addition of interfacial agent improves the degree of dispersity and adhesion at the interface and thus prevents the formation of pores (Ružinská et al. 2014a).

Viscosity values of the mixtures increase with increasing content of the polyamide components, and their values are significantly higher than the additive values calculated from the pure components. Presumably, this is an indication of increasing tolerance of the system due to the high degree of components dispersion. The role of interfacial agent in the formation of the structure of the mixtures, reflected in the flow properties, was examined from the dependence of the apparent viscosity on the shear stress of the mixtures prepared by using intermediate stage respectively mixtures without the interfacial agent. While the apparent viscosity values of mixtures of PP/PA₆ (without intermediate stages) are close to the additive values, the apparent viscosities of the mixed melt prepared by using intermediate stage have significantly higher values. (Table 3).

Conclusion

The aim of our research was to determine the optimum conditions for the technological preparation of new innovative product mixtures - polymers by studying the process of rheological melting in the production process of subsequently formed bicomponent fiber-forming polymers applied to new special composite materials in the context of the economic efficiency of manufacturing innovations.

By evaluating the viscosities and rheological characteristics of the prepared variants of the bicomponent polymer mixtures, the conclusions were verified regarding the determination of the optimal technological conditions for the prediction of the manufacturing process conditions of the special composites.

Furthermore, based on the results of our experimental research, thermodynamically the most suitable variants of the material composition of new PP/PA_{6M} polymer blend variants were made, and variants were compared without and with the addition of the interfacial agent – maleic anhydride (MAH). The effect of the interfacial reagent on the rheological characteristics as well as the reduction of the thermodynamic incompatibility of the prepared polymer mixtures on the phase interface was studied to improve the dispersibility of the individual components of the polymer mixtures to predict optimal melting conditions and subsequent spinning in the process of making special composites.

In the previous author's work (Ružinská 2010; 2014a-d) the supramolecular and morphological structure, mechanical, physical and utility properties of bicomponent polymeric fibrous mixtures have been studied and compared with the original polymers with regard to their subsequent use as the special composites themselves, resp. as an addition to the non-polymeric matrix of the other composites.

Responding to the current need to focus on societal interest in targeted innovation planning in manufacturing processes to maintain the competitiveness of manufacturing businesses, we have focused our attention on designing product innovations by preparing special composites for the needs of different sectors industries.

We believe that product innovations of new, forward-looking materials are a way of promoting strategic production management, and can significantly boost the unique position of a manufacturing company in a competitive environment. Transfer of research results and its subsequent implementation into the manufacturing process will allow for a relatively short time to implement product innovations with the expected results.

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