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IMPACT OF IMPURITIES OF POLYPROPYLENE AND SILICONE INCLUSIONS ON THE PROPERTIES OF POLYAMIDE 6.6 REGRANULATES DERIVED FROM THE RE-PROCESSING OF AIRBAGS

The following article presents the results of selected properties of regranulates of polyamide 6.6, regranules of polyamide contaminated with polypropylene and regranules of polyamide contaminated with silicone. The tested materials came from the reprocessing of polyamides 6.6 originally derived from production of airbags from renowned world producers (material for the research came from production waste). The results of examination were referred to regranulates of uncontaminated polyamide but also obtained from waste from the production of these airbags.

The influence of impurities on properties of regranulates such as their density and melt flow index was assessed. The tests allowed to show a significant impact of impurities on the density but above all on the mass and volume flow rate index which ranged from 47 to 116 g/10 min.

In the case of standardized test specimens selected thermal and mechanical properties were analyzed. Differential scanning calorimetry was used to assess the impact of impurities on the thermal properties of polyamides, allowing primarily identification of materials and impurities (especially polypropylene) as well as characteristic temperatures and the enthalpy of melting of the materials being analyzed. The mechanical properties were assessed using a DMA device. DMA research allowed to determine changes in mechanical properties in a wide temperature range of tested materials. It allowed to obtain full characteristics of changes in material stiffness under the influence of two factors, i.e. temperature and content of impurities, like polypropylene or silicone.

Keywords: recycling; composites; polyamide 6.6; MFR; mechanical properties

1. Introduction

Currently, there are many polymer materials on the market of different properties and applications. In modern applications, composites based on thermoplastics, and thermoplastics blends are increasingly used [1-11]. The problem of recycling these materials is becoming a challenge for the current industry and the natural environment (due to the presence of many materials and their blends) [10-12]. Composites based on thermoplastic polymers and their blends pose increasing difficulties during recycling [13] and re-use [14,15] (increasing degree of filling and reduction of length of fibrous fillers or impact of different pollution like silicone particles). the presence of so many material groups with different rheological properties (differences in the melt flow rate, viscosity, etc.) and containing different fillers or impurities, leads to difficult mixing in the case of recycling. In many cases they are substances that do not mix and dissolve

in one another. These materials differ not only in supermolecular structure but also in thermal properties. All these factors affect not only the recycling process (making it difficult or in some cases impossible to obtain a homogeneous material) but also the properties of the finished product (chemical incompatibility of individual components, uneven dispersion of fillers or impurities).

Apart from the recycling process itself, attention should be paid to a significant change in various properties of recycled material and products made of regranulates [16-18]. Unfortunately, the recycling process causes deterioration of polymeric materials mechanical properties, which makes it impossible to use them again in the same application [19]. In particular, the group of materials to which attention should be paid are polyamides [20], which are used in the automotive industry for the production of technical products [21,22] incl. airbags, the article describes the recycling of waste from the production of airbags (in the form of scraps).

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The change of mechanical properties, thermal and rheological properties of polymers, including viscosity, significantly affect their processing [21-25]. The analysis of changes in the flow characteristics of the processed polymers (especially those highly filled with glass fibers or other fillers) is crucial for the proper course of the technological process and thus for obtaining the correct product. Failure to adjust technological parameters to the new properties of regranulates may result in deficiencies and defects in finished products [8-12,32]. It also influences the acceleration of the degradation process, which limits their further use and leads to an increase in energy expenditure.

The following article analyzes the properties of polyamide 6.6 with various types of impurities resulting from the recycling process [21-30].

Three types of polymeric materials belonging to the polyamide group 6.6 were analyzed. All tested materials come from the reprocessing of technological waste from the airbag manufacturing process [12]. Regranulates were divided into three groups: pure material (PA6.6) from which polypropylene film was mechanically separated before processing, the second group are PA 6.6 regranulates which were processed together with polypropylene film and the third group of materials are PA6.6 silicone contaminated (airbag material was coated with a layer of silicone that cannot be removed by mechanical methods) [31-32].

2. Materials tested

Three types of material derived from technological waste obtained in the process of manufacturing airbags were analyzed. First group was clean material subjected to the process of sorting and physical separation of impurities marked as polyamide 6.6. The second group was polyamide 6.6 contaminated with polypropylene (there was no physical separation of the polyamide

covered with polypropylene film – the amount of polypropylene was 10%). The third group was polyamide 6.6 covered with silicone which could not be removed (according to data and tests carried out by an independent laboratory with the use of chemical technology, it has been shown that the amount of silicone is 16%) (Fig. 1).

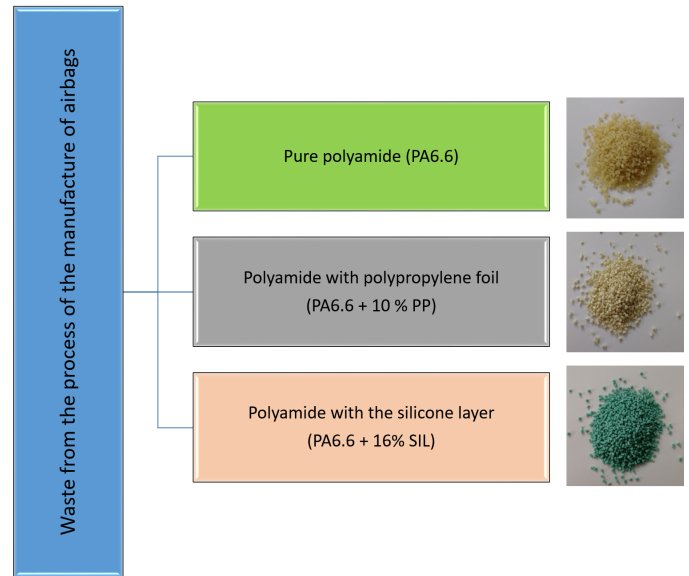


Fig. 1. Classification of materials used for research

The obtained material was reprocessing using advanced extrusion and filtration technologies to minimize the impact of impurities on the properties of the granulate, Pietroluongo et al., also Singh et al. as well as Schmid et al. described in a complex way the process of recycling (regranulation and extrusion) of the tested materials [11-16,21-23,31,32]. The regranulation process is shown in Fig. 2.

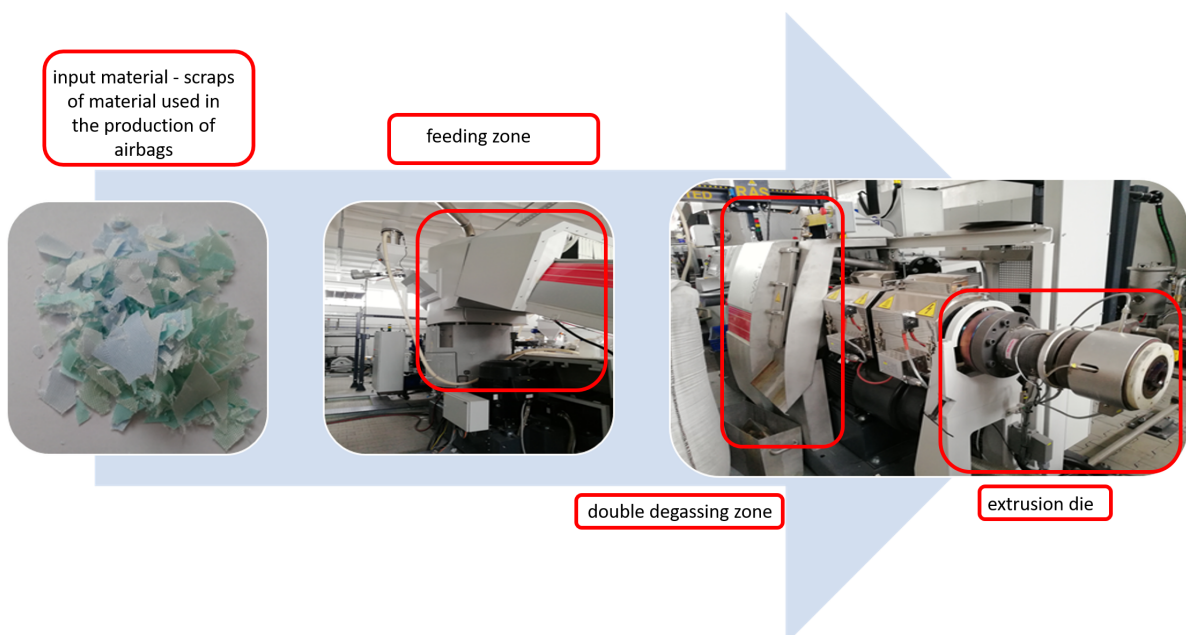


Fig. 2. Diagram of the regranulation process (own based study)

TABLE 1

Injection moulding parameters

Injection molding parameters	Value
Injection pressure	100 [MPa]
Injection speed	55 [cm ³ /s]
Holding pressure	50 [MPa]
Holding pressure time	10 [s]
Cooling time	20 [s]
Mold temperature	30 [°C]

The material in the form of scraps of polyamide fabric was automatically transported to the shredding system (the shredding system is built of a roller equipped with a number of blades enabling shredding of the fed material). Then the pre-shredded material was moved into compaction system shown in Fig. 2. After compression, the material was transported to the plasticizing system of STARLINGER single-screw extruder (Fig. 2). Device is equipped with a double degassing zone and a die used for wet granulating (elements also visible in the picture). The next step was to dry the regranulate and pack it. In the above described technological cycle, the regranulates used for these researches were produced.

The standardized test specimens (ISO 294, ISO 527) were manufactured from the obtained regranulate (regranulate was obtained from waste from the production of airbags, the color of the material depended on the origin of the waste and the type of impurities or their absence, green samples were made of a green polyamide material covered with silicone, samples without impurities have a deep amber color, samples containing polypropylene are brighter – this may be due to the foil content). For injection of the samples, a KraussMaffei KM65/160/C4 hydraulic injection molding machine was used. The same injection conditions were used for all processed materials and they were as follows – TABLE 1.

The samples were manufactured using a double-cavity injection mold made in accordance with PN-EN ISO 294-1 [26]. The molded parts obtained in the injection process are shown in Figure 3 and they show respectively the molded parts obtained from uncontaminated polyamide, polyamide with polypropylene and polyamide with silicone.

3. Results

The tests were carried out in accordance with the standards. Description of the measurement procedure has been placed in the following sections.

3.1. Density measurement

The density measurement was carried out for the obtained regranulates and for standard molded parts obtained in the injection process, in accordance with PN-EN ISO 1183-1 [27]. Further analysis included the immersion method. Before the measurement, the samples were conditioned for 24 hours at a temperature of 23°C with an ambient humidity of 50%. Then, a measurement was carried out for prepared weighing regranulate.

The density test was carried out by the immersion method using an analytical balance with an accuracy of ± 0.1 mg using an immersion vessel and stationary support. The stand has been

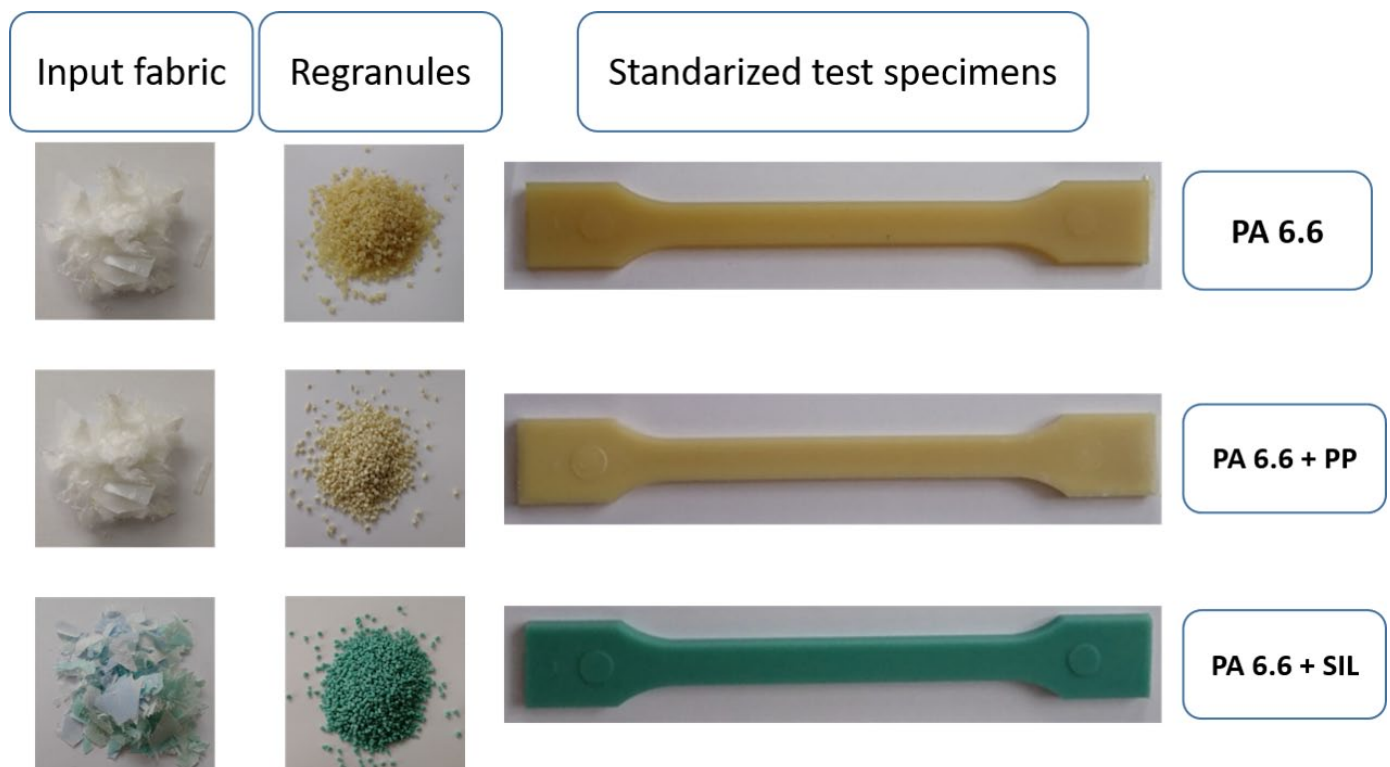


Fig. 3. Standardized test specimens obtained in accordance with ISO 294

equipped with a thermometer. Distilled water was used as the immersion liquid. To determine the sample density ρ_s at 23°C in grams per cubic centimeter, the following equation was used:

$$\rho = \frac{m_{S,A} * \rho_{IL}}{m_{S,A} - m_{S,IL}} \quad (1)$$

where: $m_{S,A}$ – apparent mass in the air [in grams], $m_{S,IL}$ – apparent mass of the sample in the immersion liquid [in grams], ρ_{IL} – density of the immersion liquid at 23°C given by the supplier.

The obtained density measurement results are presented in TABLE 2.

TABLE 2
Results of density measurement

Lp.	Density of tested materials [g/cm ³]		
	PA 6.6	PA 6.6 + PP	PA 6.6 + SIL
1	1,11744	1,10682	1,10533
2	1,11842	1,10677	1,10366
3	1,116985	1,10498	1,10293
Average value	1,117615	1,10619	1,103973
Standard deviation	0,000733	0,001048	0,00123

The test was carried out for regranulates obtained in the extrusion process. The samples were sequentially placed in a measuring instrument. The highest density value was obtained for the granulate marked as PA6.6 (without presence of other polymers – pure PA6.6). However, the introduction of impurities in the form of polypropylene or silicone causes changes in the density of the material – its reduction, it can be seen that impurities introduced into polyamide 6.6 reduce its density. Due to the presence of materials with a lower density (for polypropylene it is 0.91 g/cm³, and for silicone, depending on the type, from

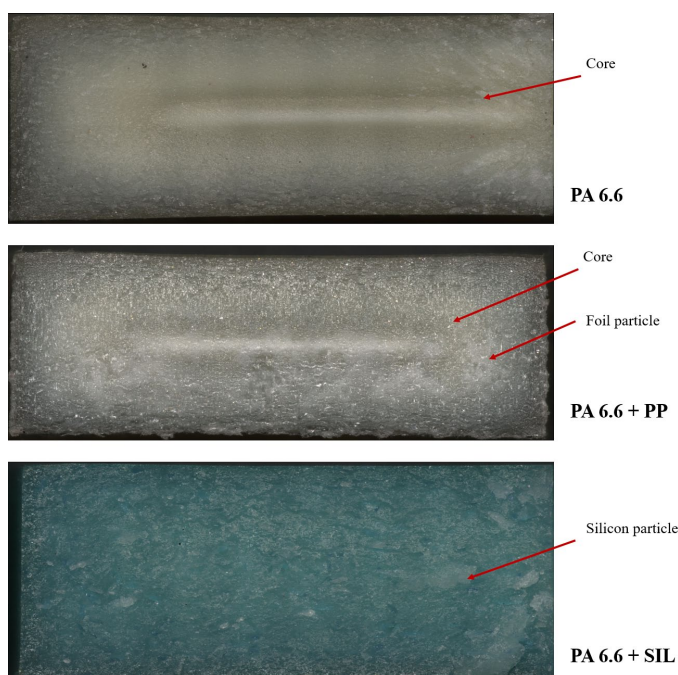


Fig. 4. The cross-sections of standardized test specimens

0.9 to 1.26 g/cm³), this leads to a reduction in the density of the finished product. Figure 4 shows the cross-sections of standardized test specimens (magnification x200).

Microscopic observations of the sample cross-sections (standard samples were conditioned for 24 hours at minus 35 degrees and then broken in the center of the sample, then fragments were taken to observe the cross section) were performed using the Keyence VHX 7000 electronic optical microscope.

For the sample marked as PA6.6 in Figure 4, a characteristic core is visible, but no inclusions or presence of other materials were observed. For the sample marked as PA6.6 + PP, the core can also be distinguished, on the surface of the entire cross-section, small shining particles are visible, the foil contained in the processed polyamide is responsible for their formation (for polyamide contaminated with polypropylene, despite higher processing temperatures, the homogenization of both polymers did not take place, as shown in the figure). For the sample marked as PA6.6 + SIL, no core was observed, while the cross-section shows large silicone agglomerates that have not been homogenized with polyamide 6.6.

3.2. Measurement of melt flow rate

Melt flow rate analysis of PA6.6, PA6.6 + PP and PA6.6 + SIL was carried out in accordance with PN-EN ISO 1133 [28]. A plastometer Dynisco D4003DE was used for the test.

To calculate the obtained indicators, the following equation was used:

$$MFR(T, m_{nom}) = \frac{600m}{t} \quad (2)$$

where: T – test temperature (expressed in Celsius degrees), m_{nom} – nominal load (in kilograms), m – average weight of extrudates (in grams), t – cut-off time interval (in seconds), 600 – factor for calculating grams per second per gram for 10 min (600 s).

$$MVR(T, m_{nom}) = \frac{A600l}{t} \quad (3)$$

where: T – test temperature (expressed in Celsius degrees), m_{nom} – nominal load (in kilograms), A – arithmetic average of the cross-sectional area of the cylinder and piston head (in square centimeters), previously determined measurement time or arithmetic average of individual time measurements (in seconds), l – previously determined piston displacement distance or arithmetic mean of the results of individual distance measurements (in centimeters).

Before starting the measurement, all material groups were properly prepared for the process, each of the materials was dried, using a SHINI CD-60 shelf dryer, the drying process was carried out for 4 hours and the drying temperature was 80°C. The measurement was carried out at 280°C with a nominal load of 5 kg.

The obtained results of the MFR and MVR measurement are presented in TABLE 3.

TABLE 3
Results of mass and volume flow rate measurement

Polymer tested	MFR [g/10 min]	MVR [cm ³ /10 min]
	Average	Average
PA6.6	96,64	91,20
PA6.6 + PP	115,80	110,15
Pa6.6 + SIL	46,87	44,60

The obtained results show the diverse influence of impurities on the obtained values of melt flow rate of the material. The uncontaminated polyamide 6.6 regranulate has a relatively high flow rate, which indicates low melt viscosity at elevated temperatures and may affect the way the mould cavity is filled. In the case of polypropylene-contaminated regranulate, the MFI index and also polymer viscosity is changed greatly. A significant increase in the melt flow rate and a decrease in viscosity may be due to the presence of polypropylene, which during the measurement acted as a lubricant, reducing the flow resistance and increasing the fluidity of the mixture (for polypropylene, this test is carried out at 230°C, therefore its fluidity increased significantly at 280°C). The opposite effect can be observed in the case of polyamide 6.6 contaminated with silicone, for this material a significant decrease in the melt flow index was obtained and, consequently, an increase in the viscosity of the tested material. A sufficient reduction in the melt flow rate of a silicone contaminated polyamide 6.6 may be associated with the formation of silicone clusters, hindering polymer flow.

3.3. Differential scanning calorimetry

The tests of thermal properties were aimed at determining the enthalpy of melting of the examined materials. Also, it was meant to determine whether the introduction of polypropylene or silicone impurities into the polyamide would significantly affect the value of melting point and the degree of crystallinity obtained. The test was performed in accordance with PN-EN ISO 11357-1 [29].

The obtained diagrams of the thermograms are presented in Figure 5 (for analysis on one chart the curves of the second heating are compared, while on the second diagram the curves from the cooling of samples). The test was carried out in the heating – cooling (H-C-H) thermal program with a temperature step of 20 K heating and 20 K cooling per minute, where each sample was heated up to 300°C and then cooled to ambient temperature and then reheated to 300°C.

The total quantity of heat transferred, Q , corresponds to the time integral to the heat flow rate:

$$Q = \int \frac{dQ}{dt} dt \quad (4)$$

Heat flow rate is a quantity of heat transferred per unit time (dQ/dt), expressed in watts (W) or milliwatts (mW).

ΔQ is a quantity of heat absorbed (endothermic, ΔQ positive) or released (exothermic, ΔQ negative) within a specified time t , and temperature T , range by a specimen undergoing a chemical or physical change or temperature change:

$$\Delta Q = \int_{t_1}^{t_2} \frac{dQ}{dt} dt \quad (5)$$

ΔQ – is expressed in joules (J)

Specific heat capacity at constant pressure is a quantity of heat necessary to rise the temperature of unit mass of material by 1 K at constant pressure:

$$C_p = \frac{1}{m} * \left(\frac{dQ}{dT} \right)_p \quad (6)$$

ΔQ – is a quantity of heat, expressed in joules (J) necessary to rise the temperature of material of mass m , expressed in grams (g), by dT kelvins at constant pressure, C_p – is expressed in joules per gram per kelvin ($Jg^{-1}K^{-1}$)

The DSC thermograms show the variation in thermal properties of the tested materials. For polyamide processed together with polypropylene film, two distinct transformations have been demonstrated, the first of which occurs at the temperature of 163.8°C and is characteristic for polypropylene, while the second at 262.7°C corresponds to the melting temperature of polyamide 6.6. The other two DSC thermograms obtained for pure polyamide and polyamide with a layer of silicone have only one transformation occurring at 262°C. DSC did not show the effect of silicone applied to the surface of the polyamide fabric on the course of silicon presented on the thermogram (no thermal changes related to the presence of silicone, it may be related to its insufficient amount of the silicone in the analyzed sample the temperature range of measurement).

From a technological and processing point of view, the presence of other polymeric materials may have a negative impact on the expected properties of the recyclate and the finished product. The analysis of the melt flow index showed a significant influence of the addition of polypropylene and silicone on the flow characteristics and the viscosity of the polymer mixture. This will affect its processing. The presence of 10% polypropylene or 16% silicone significantly affects the properties of the finished recyclate. In the case of obtaining this type of mixtures, particular attention should be paid to the difficulty of their homogenization. Difficulties with homogenization result from different processing temperatures of the analyzed materials, their belonging to a completely different material group (in the case of silicone) as well as different values of the melt flow index (in the case of polyamide and polypropylene). These differences causes technological difficulties during the processing of such doped regranulates. They can also lead to defects in the finished product in the form of delamination [26,27].

However, DSC tests did not show the presence of silicone in the sample, so it is difficult to determine its influence on thermal properties. In order to broaden the knowledge in this field, it would be necessary to perform thermogravimetric tests and determine the mechanical properties.

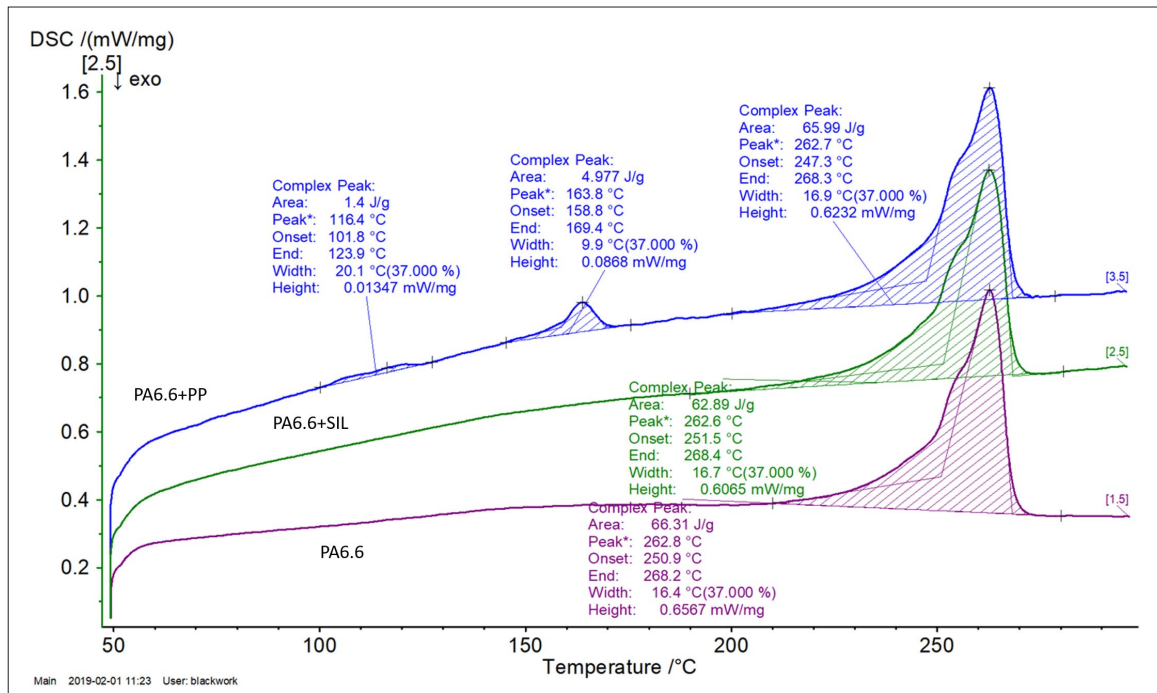


Fig. 5. DSC thermograms for test specimens PA6.6, PA6.6 + PP and PA6.6 + SIL, heating curves

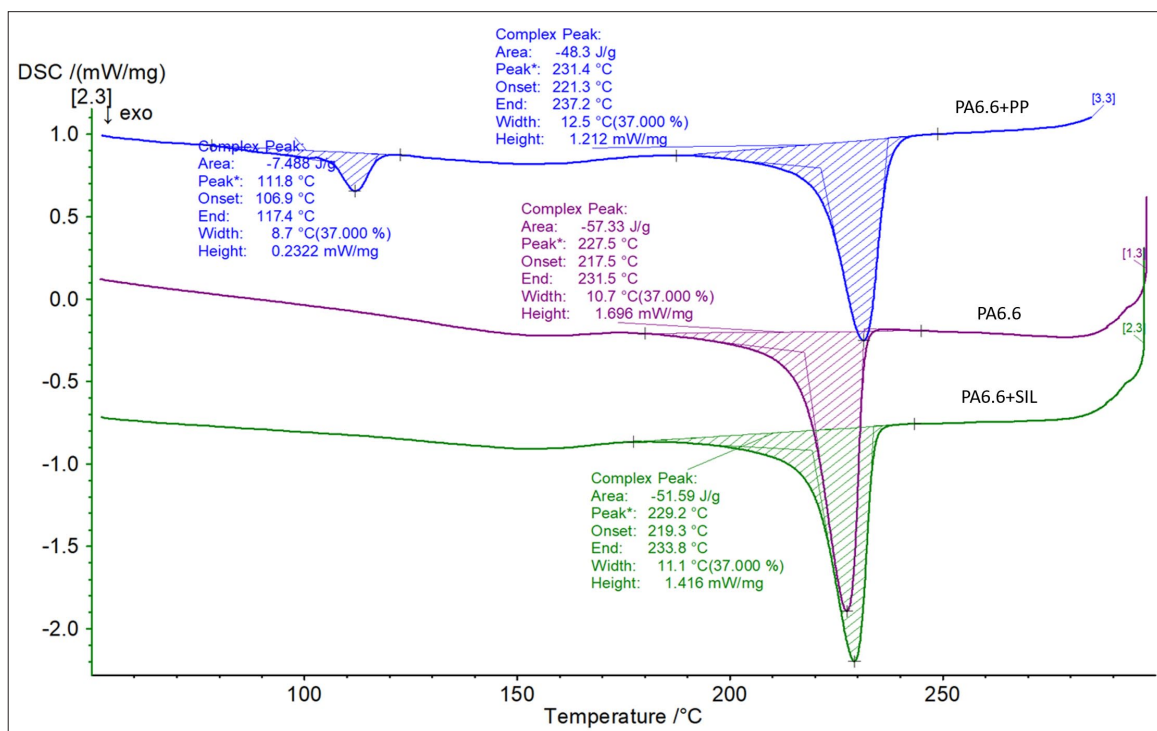


Fig. 6. DSC thermograms for test specimens PA6.6, PA6.6 + PP and PA6.6 + SIL, cooling curves

3.4. Dynamic mechanical properties

The analysis also included testing of dynamic mechanical properties in accordance with PN-EN ISO 6721-1 [30].

The curves obtained are presented in the figures 7-9. The analysis included testing using fixed supports (three-point bending were used in examinations.). Samples with dimensions of

55 mm × 10 mm × 4 mm were used in examination. The test was conducted in the temperature range from -60°C to +130°C, with an amplitude of 120 μm and a frequency of 1 and 10 Hz, and a force of 6 N.

The obtained results clearly indicate that the processing of polyamide even with a low silicone content affects the deterioration of its mechanical properties and reduction of stiffness

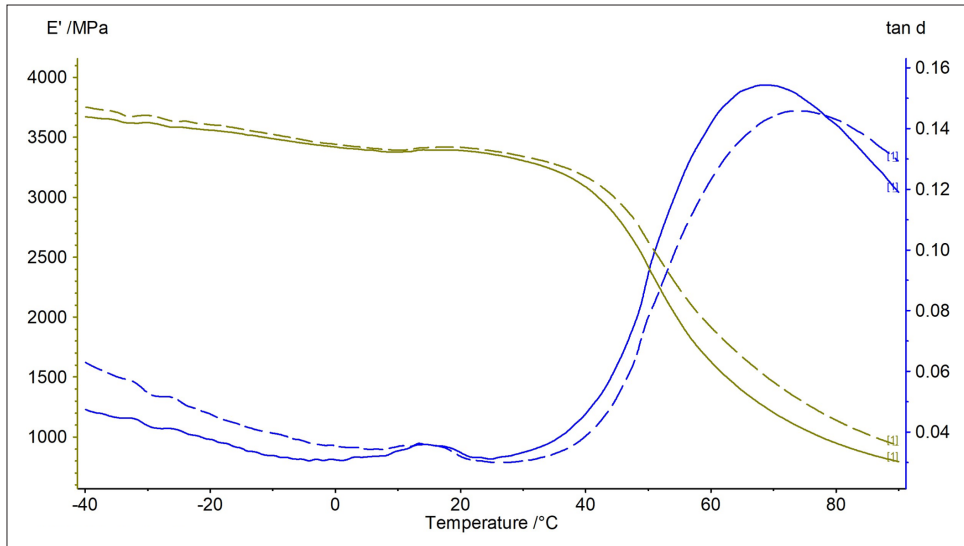


Fig. 7. DMA curves obtained for PA6.6 (pure polyamide)

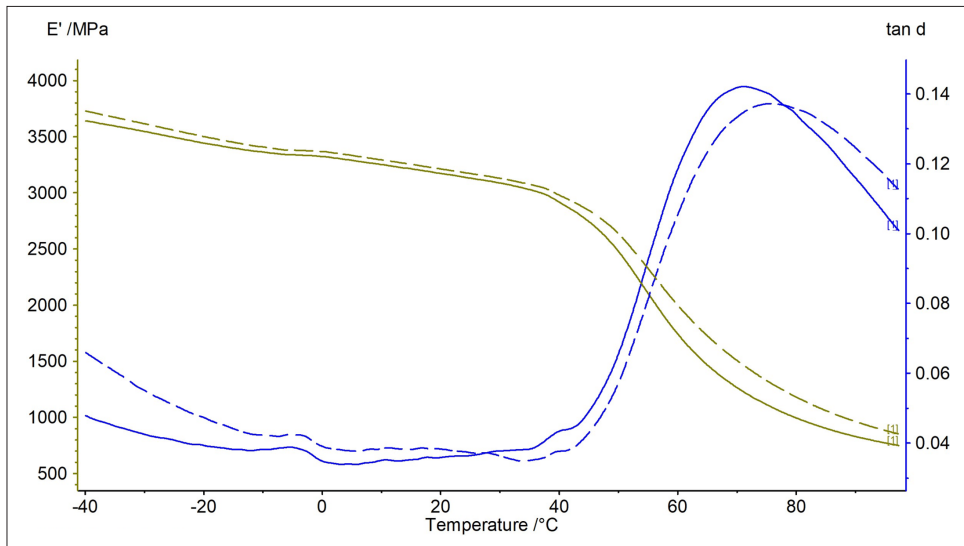


Fig. 8. DMA curves obtained for PA6.6 + PP (polyamide processed with polypropylene film)

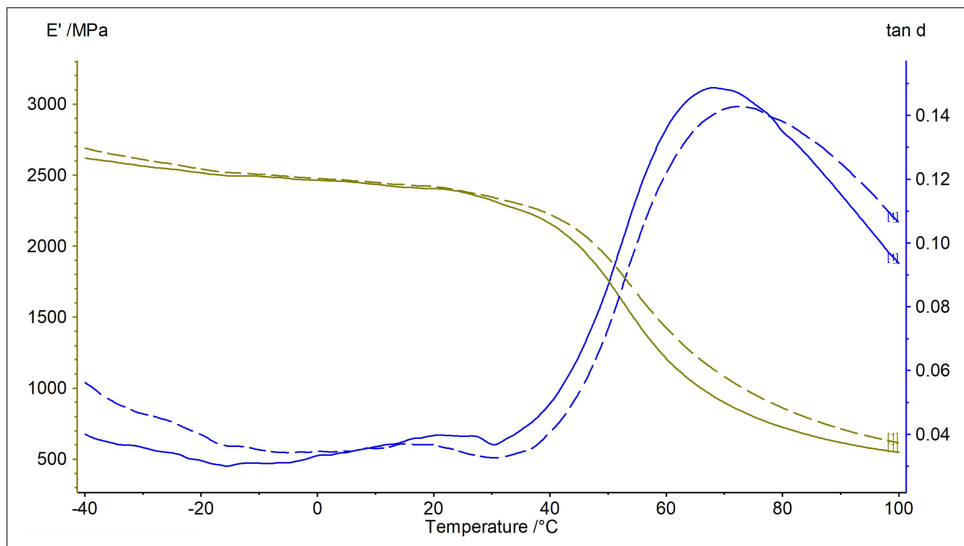


Fig. 9. DMA curves obtained for PA6.6 + SIL (polyamide with a layer of silicone)

(Fig. 7). Similar deterioration is not observed for polyamide processed with polypropylene film and for polyamide without impurities (Figs. 8 and 9). In the case of samples contaminated with polypropylene, no significant drop in stiffness was observed in reference to pure polyamide. The presence of silicone, which is a soft material, significantly reduces the stiffness of the material, it can lead to increased impact strength (thus the functional properties of the finished product may be improved).

4. Conclusions

The research showed that the introduction of impurities in the form of polypropylene and silicon affects selected properties of regranulate. In the case of density measurement in both cases (contaminated with polypropylene and silicone) leads to a decrease in the density of the regranulate in relation to the uncontaminated material (the density of impurities contained in the tested materials is lower than that of polyamide).

An introduction of these admixtures significantly affects the viscosity (conclusion based on the changes in the melt flow rate) and hence the processability of the tested regranulates. When polypropylene is added to the polyamide, there is a sharp decrease in viscosity and an increase in both mass and volume flow rate. In the case of regranulate, polyamide with silicone, there is a significant increase in viscosity and a two-fold decrease in the mass flow rate. From a technological point of view, this knowledge allows for a better process control and thus improves the quality of blow molded products.

The study with the use of a differential scanning calorimetry showed the occurrence of additional thermal changes in the temperature range corresponding to those occurring in polypropylene. The tests showed no significant differences in the course of thermograms as well as in the values of the characteristic temperatures obtained (in this case, melting points). In contrast, DMA tests have shown that the addition of silicone significantly reduces the mechanical properties of the resulting regranulate.

The obtained results allow us to conclude that the plastics processing industry is a growing branch of industry, requiring support and broadening the knowledge in the field of analysis of the properties of recycled polymer materials. As the above research has shown, in the field of processing thermoplastic materials there is no obviousness and the results obtained can be surprising.

On the basis of the conducted research, it was shown that the obtained melt flow rate for all tested materials is relatively high (above 40 g/10 min), therefore the tested regranulates can be used in the production of technical details with a long flow path. However, due to the fact that these are regranulates, the melt flow rate should be controlled in terms of introducing the necessary changes in the technological settings of processing devices. Fluctuations in the melt flow rate may affect the stability of the technological process and the quality of the finished product.

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