

Characterization of Blend PA6 +EPDM (60/40) by Tensile Tests

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Abstract: *This paper presents a characterization of PA6 and the blend PA6+EPDM (60/40) by tensile tests in order to evaluate several mechanical properties for impact resistance applications. Results were found to be dependend on test rate (10 mm/min, 250 mm/min and 1000 mm/min). SEM investigation point out a homogenous structure. The blend has better value of energy at break, for the higher test speed: for $v=250$ mm/min this characteristic has the value of energy at break 29.7 J and the blend has 76.3 J. At 1000 mm/min, PA6 has this characteristic of 20 J, but for the blend, it is almost insensitive for the two higher test speeds (76.3 J at 250 mm/min and 72.4 J at 1000 mm/min, respectively) as compared to the neat polymer that decreases this feature when the test speed increases. At the lowest test speed, the values of energy at break for the materials in this study are close (90.2 J for PA6 and 87.7 J for the blend). The results from tensile tests pointed out that the formulated blend is recommended for impact resistance applications.*

Keywords: PA6, EPDM, blend, tensile test, strength at break, strain at break, energy at break, Young modulus

1. Introduction

The journal only publishes novel, high-quality original research papers. Special issues of selected, peer-reviewed papers from scientific meetings, workshops, conferences on the science, technology and, engineering and emergent subjects will be also published.

The introduction will contain, clearly and concisely, the selective presentation of the scientific information concerning the subject as a support for the original elements of the paper.

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Polymer blends, simple (two components) and complex are of interest for industry, thus, research in this domain has been advancing rapidly, as, for instance, the boost given by Toyota research group for PA6. Another advantage of introducing polymer blends on the market is that their processing technologies are the same or very similar to those for neat polymer, the mixing in the melting state being the most commonly used [1-5]. The properties of the polymeric blends are strongly influenced by the developed morphology and the behavior of the interfaces among constituents, but also by processing parameters and treatments [6, 7].

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Polyamides (PA) are thermoplastic polymers, with very diversificate applications (as fibre, films, fabrics and machine components obtained by injection molding) due to their thermal and mechanical properties. But the engineer has to take into account several disadvantages like liquid swelling, impact sensitivity when the component has high stress concentrators and a poor dimensional stability. The solution offer by researchers is to add improvers in polyamides related to the application [8-11].

EPDM (also named ethylene propylene diene monomer rubber) is a mono layer membrane of synthetic rubber, which could have particular adding materials as black carbon, fibers, oils, curing agents etc., being chemically, UV and ozon. It is compatible with polar substances, as fire-resistant fluids, hot and cold water, ketones and alkalis. It is incompatible with most hydrocarbons. EPDM has outstanding resistance to heat, ozone, steam and weather. It is an electrical insulator [12, 13]. A high strain at break (over 300%) is an advantage of EPDM for bearing mechanical load. At -40°C , it keeps its properties, but other polymers loose thier elasticityand became brittle. At $+150^{\circ}\text{C}$, EPDM are still providing good mechanical characteristics but many polymers are already softened. It allow for repeated elongations, but thermoplastics could fracture or not regain partially their shape [14].

Banerjee et al. [15] considered that mixing melt polymer components is an efficient and economical method to obtained high performance polymeric materials, but the researchers have to evaluate the influence of compatibilizing agents as without them blends could have poor mecanical properties and an ijnstable morphology [7, 16].

Properties of polymeric blends depend on their morphology, the adhesion quality among constituents, the surface tension and the package of properties of each constituents [17-19].

Polyamides could have impact characteristics improved by adding an elastomer or elastomer modified polymers. Frequently, in polyamides and in their blends, there were added SEBS-g-MA with clay [20-24], SEBS-g-MA+montmorillonit [25], (SEBS)+nanosilica [26], SEBS-g-MA+nano particule de carbură de siliciu [3]; NBR+clay [27], ABS [28, 29], EPDM+clay [30-32], EPDM+sepiolit [33] mEPR)+clay [34]; silicon elastomer+clay [35]; PA+(EOR)+clay [36]; elastomer etilen-co-butil acrilat/nanotalc [37], PA+PP+EPDM [38, 39], PA+EPDM [40].

Caramitu et al. [41] report on preparation and characterization of PA6/EPDM blends, for four mass concentrations: 100/0, 90/10, 80/20 and 70/30. The mechanical behavior is characterized by tensile strength and compression resistance, which have proved the contribution of elastomer on the material plasticity.

The aim of this study is to evaluate the mechanical characteristics of PA6+EPDM (60/40, mass concentration) and to comparev them to those of neat PA6.

2. Materials and methods

General short-term mechanical properties facilitate the comparison with other materials and for referring to standards and their measuring methods, including strain-stress curves. These curves and the mechanical properties on short-term have a limited applicability in design because on long term, these values could differ, but they offer o good reference in quality control and material selection [42].

The new standard ISO 527-1 [43] does not have definitions for proportionality limit and yield limit becaus the idea of extend the linear segment of the curve was abandoned [42]. Specimens were fixed in the grips of the universal test machine INSTRON 2736-004 and pulled until failure. For ISO 527, the test speed is typically 5 mm/min or 50 mm/min for measuring strength and elongation and 1 mm/min for measuring modulus. For evaluating the dynamic response of the material, researchers use other values of the test speed. In this study, the authors selected the following test speeds: 10 mm/min, 250 mm/min and 1000 mm/min, as the test machine allow for testing at higher speeds as those in the standard [44-46].

Figure 1 presents the shape and dimensions of the specimens.

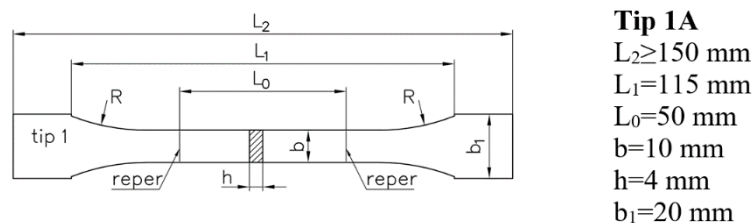


Figure 1. Specimen dimensions, as in SR EN ISO 527-1:2020 [43]

For tensile tests, the results could be given in:

true stress

$$\sigma = F/A \quad (1)$$

engineering stress

$$\sigma_{eng} = F/A_0 \quad (2)$$

engineering strain

$$\varepsilon_{eng} = \Delta L/L_0 \quad (3)$$

where F is the applied force on the specimen, at moment t, A – cross section of the specimen at moment t, A_0 – initial cross section of the specimen, L_0 - the initial length of the specimen between marks.

When testing polymeric materials, conditioning is very important. The specimens were maintained at $23 \pm 2^\circ\text{C}$ and humidity $50 \pm 10\%$, for at least 20 h.

In literature, strain rate could be approximate with:

$$\dot{\varepsilon} = \frac{v}{L_0} \text{ [s}^{-1}\text{]} \quad (4)$$

For the specimens used in this study the calculated values are given in Table 2:

Table 1. Calculated values for the strain rate

v [mm/min]	v [m/s]	L_0 [mm]	$\dot{\varepsilon} = \frac{v}{L_0} \text{ [s}^{-1}\text{]}$
10	0.000166	50	3.32×10^{-3}
250	0.00416		83.2×10^{-3}
1000	0.0166		332×10^{-3}

There are different technologies for obtaining PA + elastomer maleate + clay blends [47, 34].

The elaboration of the recipe of this blend was based on the results of some blends based on PA and EPDM or another elastomer [48-52].

Pre-mixtures of PA6, or PA6+EPDM granules are performed in a mixer (high speed mixer), with a capacity of 200 l, mixing speed 475/950 rpm, provided with heating system with electric resistances of 11 kW and pneumatic discharge system. Pre-mixing of components in a mixer before being introduced into the extruder is important because the used raw materials have different densities and, implicitly, a high tendency of stratifying on density if they are not pre-mixed. Thus, a higher degree of dispersion is realized.

A pre-mixing drying stage is done by mixtures of polymer and adding material from the high-speed mixer will be placed in a drying hopper, at a temperature of $80\text{-}100^\circ\text{C}$. The dryer is equipped with an automatic mixing loading and unloading system, with a capacity of 1500 l and a working flow of 200 kg/h.

The premixtures in the drying hopper are automatically loaded into the primary dosing system. The primary dispenser has the following technical data: dosing flow = 150 kg/h, dosing system = with double screw, dosing speed = max 100 rpm; feed hopper volume = 150 l.



Figure 2. EC 52 double screw granulator extruder (SC Monofil Săvinești SA, Romania)

The compounding of the mixtures of polymer and EPDM was performed on an extruder type EC 52 with double screw (Figure 2), with simultaneous rotation. The extruder is equipped with a cooling system of the zones using cooled softened water, with a vacuum pump (with a power of 2.2 kW), with water jacket, respectively, a hydraulic system for continuous filtration of melts provided with an engine of 1.5 kW and a maximum working pressure of 20 MPa. The extruder cylinder, has a modular structure, each module having a length to diameter ratio of 4 (L/D). Module 1 is provided with a supply port for additived polymeric matrices. Module 5 is provided with a hole located at the top for ventilation and/or feeding with long fibers and a side hole for dosing chopped organic or inorganic fibers and/or mineral fillings. Module 9 has a hole for the injection system.

Because the quality of the composite or thermoplastic blends depends on many factors, such as temperature, screw rotation speed, extruder length, the optimal technological parameters that will be used to make this polymer blend are indicated in Table 2.

Table 2. Technological parameters

Parameter		Musteață [49]	Ahn [34]
Machine	Extruder machine type EC52 with double screw with simultaneous rotation, diameter 51.4 mm, ratio L/D=40, modular screw structure, with 5 types of sections of different lengths, axial pressure of 4.5-5.5, main engine power of 55 kW		Haake extruder with double screw with 30 mm diameter, wheel 26 mm and a length of 305 mm.
Processing temperature, °C*			
Zone I		130-150	240
Zone V		240-260	280 (injection nozzle)
Zone IX		220-230	
Rotational speed, rpm		160	280
Power supply flow			980 g/h
Mold temperature, °C		80...90	80
Injection pressure maximum pressure		50 bar	70 bar
Maintenance pressure		20 bar	35 bar
Maintenance pressure time in mold for cooling, s			9.0

* Working temperature on the 3 zones (from 9 available on the equipment)

3. Results and discussions

Figure 3 presents the samples made of PA6m after being tested in traction, under different test rates.

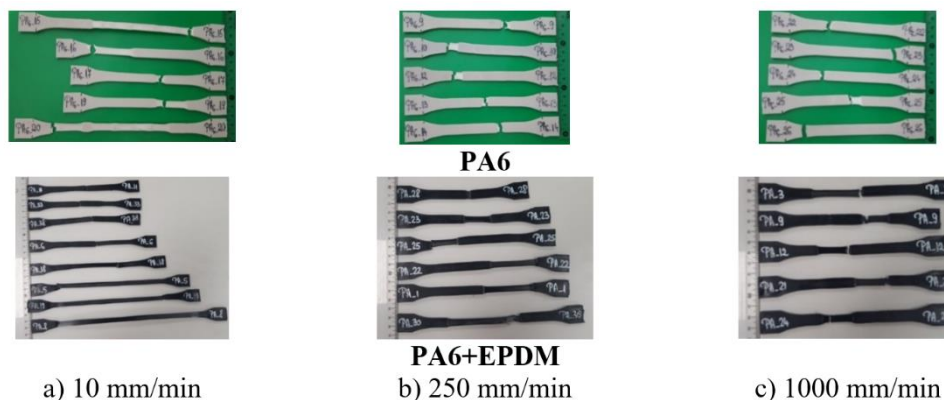
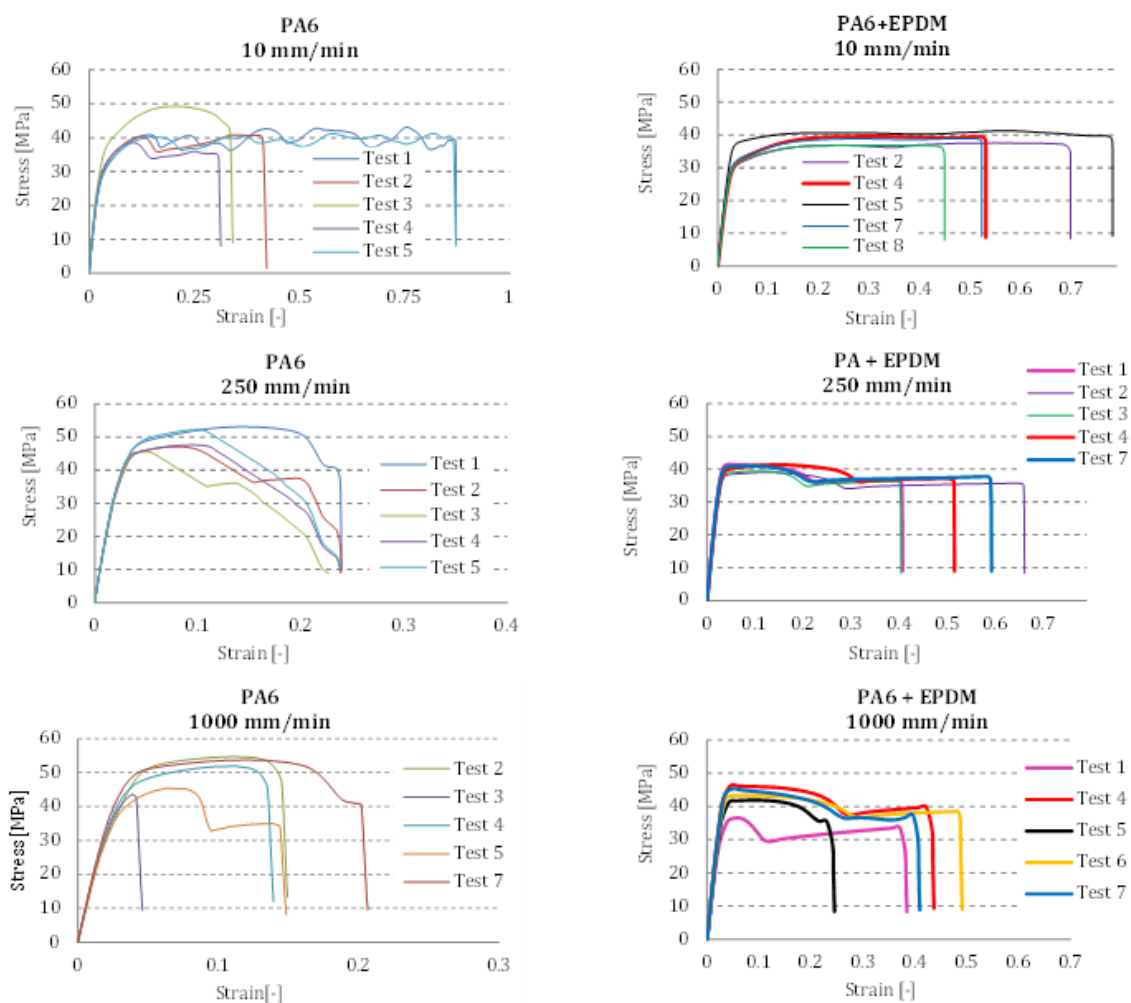


Figure 3. Broken tensile specimens at different test speeds

From Figure 4, one may notice that the polymer and the blend have the proportionality zone very well contoured, the line being overlapping, but when the proportionality limit is overpassed, the curves differentiate and the material behavior is less predictable. This means that in the visco-plastic domain (for impact resistant application) the design should be done with larger safety coefficient.



a) Stress- strain curves for PA6

b) Stress- strain curves for 60%PA6 + 40%

Figure 4. Curbe tensiune-deformație pentru materialul PA6

At 10 mm/min, sample PA6_20 was excluded, but it was kept in the photo for underlining the very differentiated behavior of the samples, from break with small necking and elongation at break to

specimens with large elongation and multiple necking to specimens that broke outside the marked zone. Specimens made of PA6+EPDM could be grouped in specimens with low strain at break (40%) and in specimens with very large elongation (more than 80%). Comparing stress-strain curves of the tested materials for test speed of 10 mm/min (Figure 4, first line), one may notice that adding EPDM in PA6 decreases and levels the plastic yield plateaux of the specimens.

When testing at 250 mm/min (Figure 4, second line), PA6 has a proportionality domain maintain or all specimens but the plastic behavior is very different, even if two zones could be point out, one of almost constant stress in yielding and one with different slopes till break. This is why component made of polyamide 6 are not recommended to be designed outside the proportionality domain. The blend has a similar proportionality line, but yielding process under load is similar for all the tested specimens, meaning its behavior in this domain is more predictable.

At the highest test speed (here, 1000 mm/min, Figure 4 third line), the materials behave as under test speed of 250 mm/min, but with curves are more spread in the plastic domain.

Both materials have large strain-at-break intervals, the largest being for the lowest test speed (10 mm/min).

Table 3 presents the evaluation, in percentage, of the same characteristics for each material. The last line points out that energy at break has a greater decrease for the neat polymer and only 17.4%.

Table 3. Relative results for mechanical characteristics, calculated as percentage difference for two values: for 10 mm/min, indexed (10) and for $v=1000$ mm/min, indexed (1000)

	PA6	PA6m
$\frac{E_{(1000)} - E_{(10)}}{E_{(10)}} \cdot 100 [\%]$	27.30	11.61
$\frac{\sigma_{break(1000)} - \sigma_{break(v=10)}}{\sigma_{break(10)}} \cdot 100 [\%]$	20.13	-5.01
$\frac{\varepsilon_{break(1000)} - \varepsilon_{break(10)}}{\varepsilon_{break(10)}} \cdot 100 [\%]$	-77.26	68
$\frac{\Delta E_{break(1000)} - \Delta E_{break(10)}}{\Delta E_{break(10)}} \cdot 100 [\%]$	-73.32	-17.4%

Figure 5 presents the average values of the mechanical properties and the maximum and minimum values. Adding EPDM in PA6 makes Young modulus to decrease, but it seems less affected by test speed. PA6 has a similar value as the blend at $v=10$ mm/min but young modulus increases more for the other two test speeds. Also, it seems less sensitive to the speed between 250 mm/min and 1000 mm/min.

As expected from literature related to polymer, at higher test speed the stress at break increases for PA6 but for the blend this characteristic could be insensitive to speed test as it increases with only 2 MPa from $v=10$ mm/min to $v=1000$ mm/min (meaning 5.7% of the value for $v=10$ mm/min). A good mark represents the fact that spread interval is narrow for the blend.

Strain at break is similar at low speed, but at higher test speeds, the blend has higher values. The designer will be interested in energy at break when using a material for its impact resistance. Thus, analyzing Figure 5, one may notice that, at low speed, both materials have close values (90 J for PA6 and 87.7 J for PA6+EPDM). At higher test speed, the blend has this characteristic more than double meaning it is more adequate for impact application

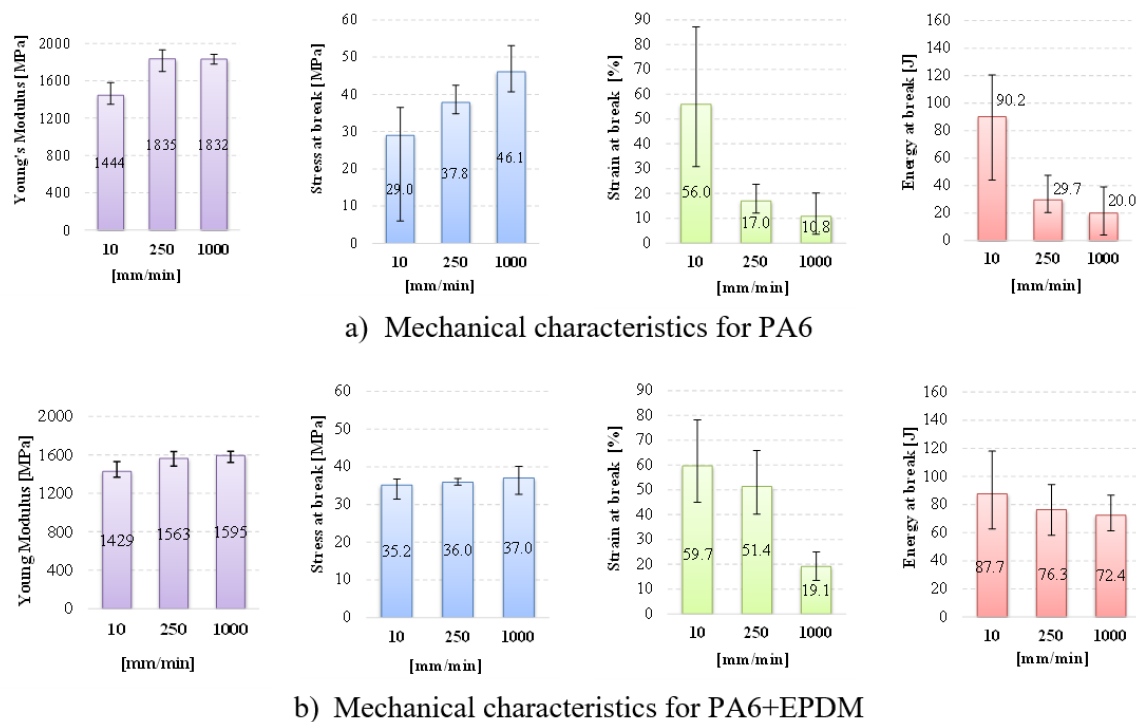


Figure 5. Average values and spread interval of the tensile tests for the two tested materials

Figures 6, 7 and 8 present SEM images. Broken surfaces were golden coated in order to obtain a better resolution of the investigated area. Each line gives images of the same broken surface of the specimens after being tested, with different magnifications in order to point out the failure mechanisms characterizing each material.

At 10 mm/min (Figure 6), the ductile character of PA6 and PA6+EPDM are evident, but specimens made of PA6 presents cavities due to the difficulty of injection this polymer under the processing parameters (Table 2). Craze process is localized. Similar aspects were obtained for PA6 tested at 20 mm/min, in [17]. The blend has also local crazing but it is obvious there are no pores and the mix is quite homogenous.

Figure 7 present SEM images obtained for broken surfaces of specimens tested at 250 mm/min. Both materials present more points where break was initiated but aspect of broken surface is more uniform for the blend. For the blend, except for the conical zone where the breaks are initiated, the surface aspect of the blend is with small volumes drawn from the bulk material, elongated till break, pointing out the local ductile character, resulting a texture with elongated fibrils of several microns. These mechanism of supporting the load by local micro-deformation is beneficial for increasing the energy at break for the blend. Local crazing, as it happens in PA6, does not offer too much resistance as it is happening with small but many and relatively uniform fibrils characterizing the failure in tension of the blend.

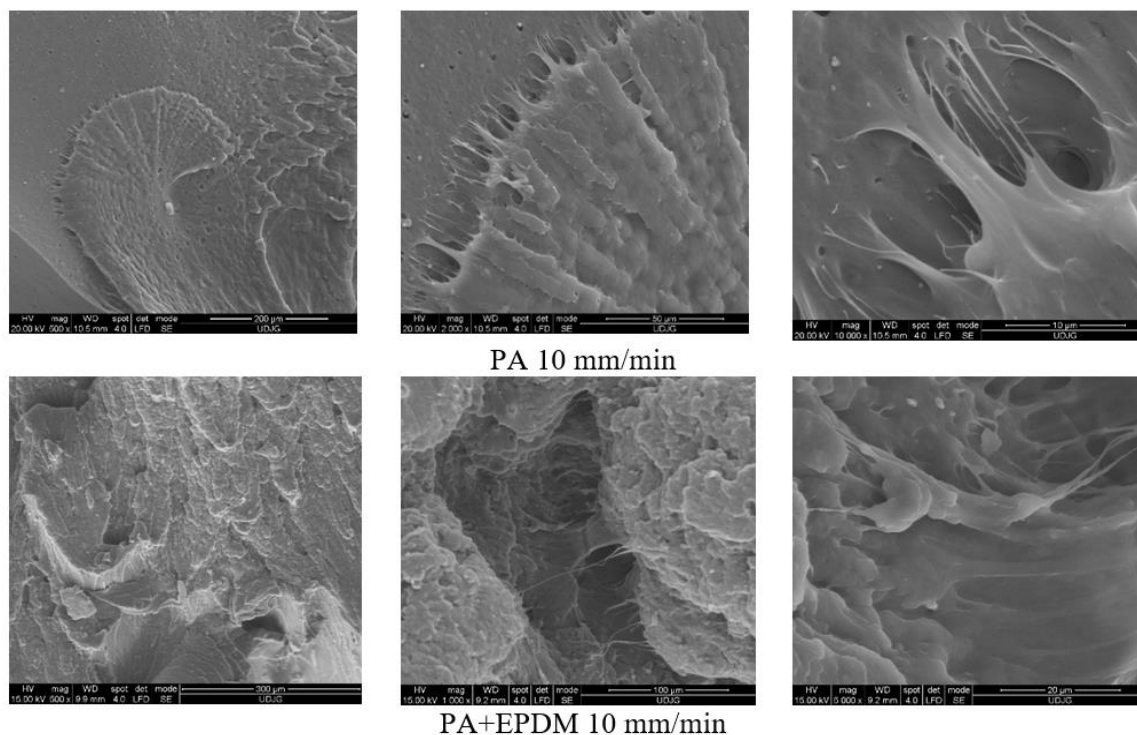


Figure 6. SEM images at different magnifications of broken surfaces at 10 mm/min

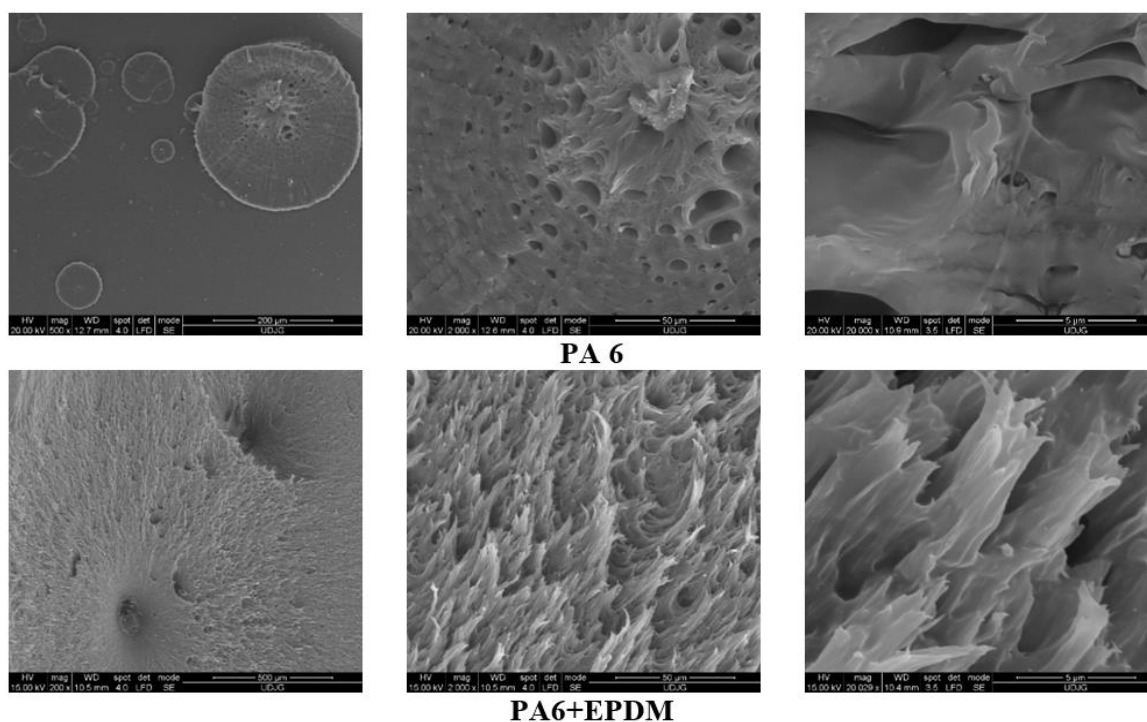


Figure 7. Broken surface of tested material, at 250 mm/min

At 1000 mm/min (Figure 8), the polymer presents several points where the break is initiated, but the increase in test speed generated small fibrils of polymers, or similar geometry and heights. Pores and cavities are visible on the broken surface of PA6 (the corner of the first SEM image of PA6, where the

wall cavity is visible and also small pores). The aspect of the broken surface of the blend is similar, but with a visible more ductile character. The presence of EPDM makes the tensile load to be uniformly supported on entire cross section of the specimen.

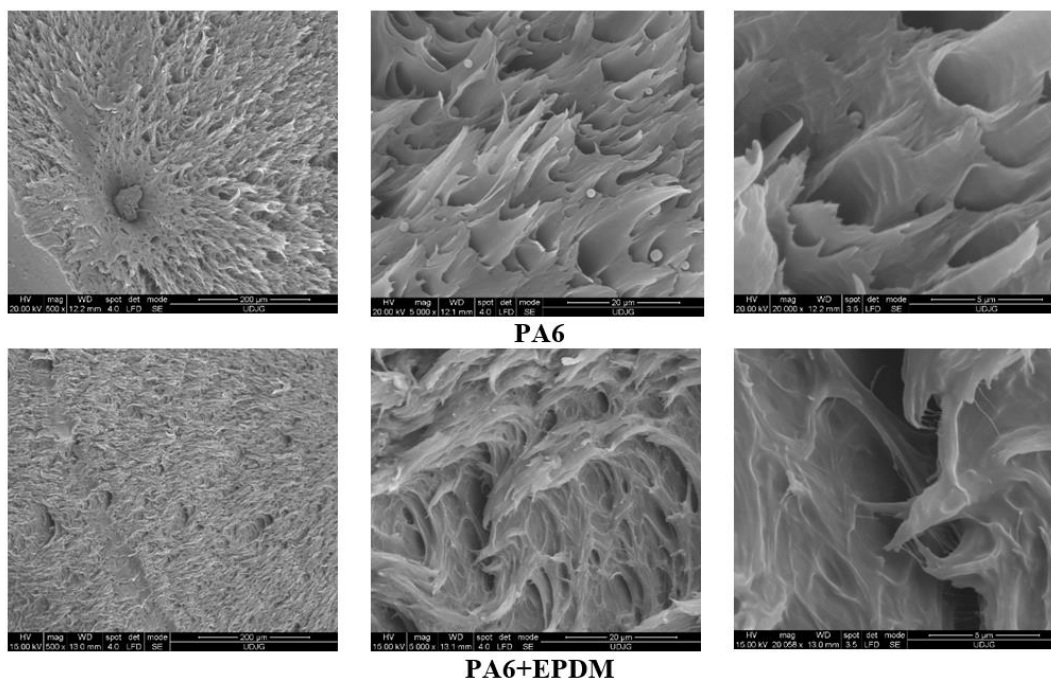


Figure 8. SEM images on break surface of a specimen at 1000 mm/min

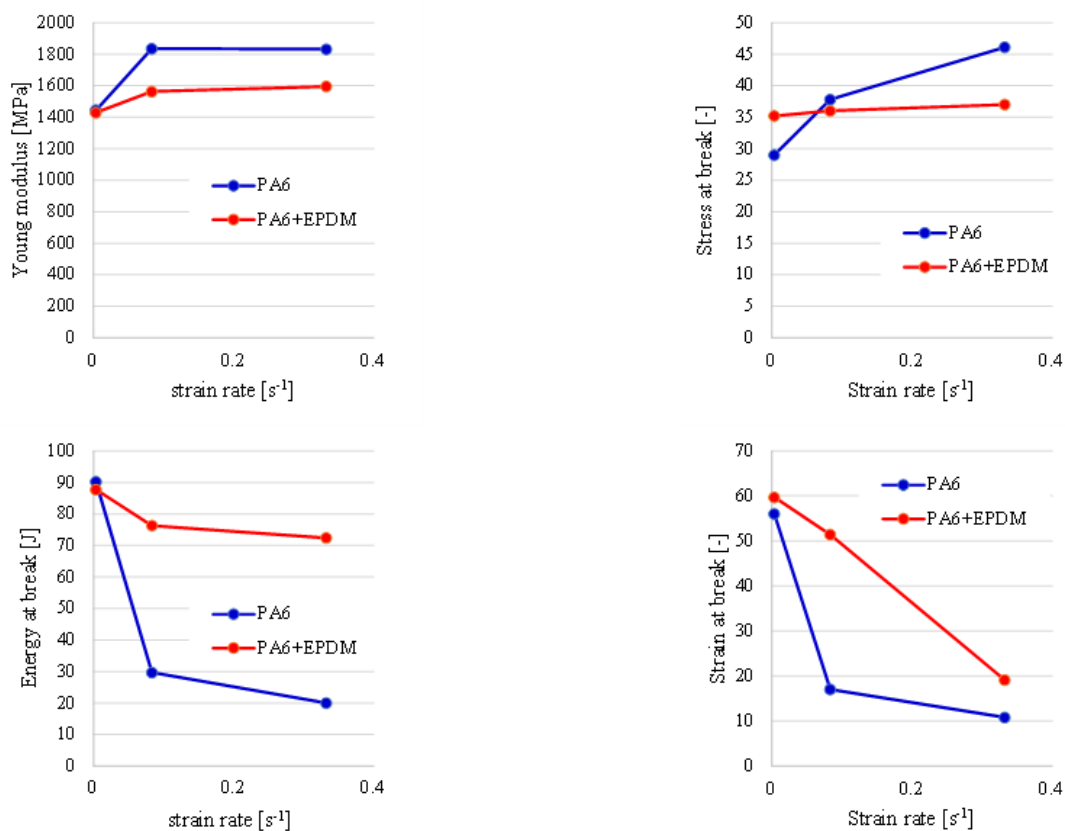


Figure 9. Comparison of mechanical characteristics for PA6 and 60% PA6+40% EPDM, as functions of strain rate

Analyzing Figure 9, the following conclusions may be formulated:

- Young modulus has close value at the low test speed (1444 MPa for PA6 and 1429 for the blend, but at higher test speeds, the influence of this parameter is insignificant: PA6 keeps the average of Young modulus around 1835 and the blend around 1570 Mpa;
- the stress at break increases with test speed for PA6 but for the blend this characteristics remains between 35...37 Mpa;
- for PA6, energy at break and strain at break have a similar allure of the curve as function of strain rate, but the blend has the strain at break more sensitive to strain rate;
- energy at break has higher values for the blend, meaning that adding EPDM helps this newly formulated material to store more energy before breaking.

4. Conclusions

Polymeric materials are very sensitive to test speed in traction.

The blend 60% PA6 +40% EPDM exhibit good characteristics for impact resistance application, including a higher energy at break for higher test speeds, as compared to PA6.

SEM investigation point out a homogenous structure. The blend has better value of energy at break, for the higher test speed: for $v=250$ mm/min this characteristic has the value of energy at break 29.7 J and the blend has 76.3 J. At 1000 mm/min, PA6 has this characteristic of 20 J, but for the blend, it is almost insensitive for the two higher test speeds (76.3 J at 250 mm/min and 72.4 J at 1000 mm/min, respectively) as compared to the neat polymer that decreases this feature when the test speed increases. At the lowest test speed, the values of energy at break for the materials in this study are close (90.2 J for PA6 and 87.7 J for the blend). The results from tensile tests pointed out that the formulated blend is recommended for impact resistance applications.

The injection molding technology was the same for PA6 and the blend, but specimen quality was better for the blend, several specimens made of neat polymer having cavities. EPDM in the proportion used by the authors helps improving the impact resistance (expressed by energy at break), with acceptable consequences on other mechanical characteristics.

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