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Development of Polyamide 6/Compound by Recycled Rubber Blends Using Graphitized Polyethylene or Polypropylene with Maleic Anhydride as Compatibilizer Agent

Divânia Ferreira da Silva[1], Edcleide Maria Araújo[1], Tomás Jeferson Alves de Melo[1]

ABSTRACT: The toughening of polyamide 6 is desirable for many applications and may be obtained by adding a phase to the elastomeric matrix. However, this leads to a loss in its rigidity characteristics. Therefore, this study aimed at developing blends of polyamide 6/compound by recycled rubber (SBR-R), with the addition of compatibilizer graphitized polyethylene and polypropylene with maleic anhydride method by mixing in the molten state in order to obtain a balance between stiffness and toughness. The systems were prepared in several compositions, and their rheological properties and spectroscopy in the Fourier transform infrared were studied by means of rheological curves obtained in an internal mixer of Haake Buchler. The results obtained with the rheological study and Fourier transform infrared showed that mixtures of polyamide 6/graphitized polyethylene with maleic anhydride presented the best results compared to those of polyamide 6/graphitized polypropylene with maleic anhydride, probably indicating reaction between the components. Thus, it was chosen graphitized polypropylene with maleic anhydride as a compatibilizing agent for carrying out other characterizations. Blends of polyamide 6/compound by recycled rubber/graphitized polypropylene with maleic anhydride and their properties were analyzed by means of mechanical tests (tensile and impact), dynamic mechanical thermal analysis, differential scanning calorimetry, and scanning electron microscopy. The impact strength and elastic modulus of the blends compatibilized reduced somewhat when compared to polyamide 6. Therefore, these results indicate a good prospect of application of industrial waste, minimizing the negative effect on the environment and adding value to a disposable material.

KEYWORDS: Polymers blends, Polyamide 6, Recycled rubber, Compatibilizers.

INTRODUCTION

Blending polymers with different molecular structures or mechanical properties have recently become a useful route in developing new, high-performance polymeric materials. Improved mechanical properties, processibility, barrier behavior, and electrical properties can be achieved through such technology. Choosing suitable polymers is the primary task in the preparation of polymer blends; however, more attention has been paid to control the morphology of blends, which has been found to have a great impact on the properties of polymer blends. Actually, most polymers used in blends are immiscible or partially miscible due to their high molecular weight and unfavorable interactions, resulting in the multiphase morphology. For binary polymer blends, when the content of one component is much lower than the other one, the minor usually forms droplet in the matrix of the major component, which is usually known as the sea island morphology (Yu *et al.*, 2010).

Since last century, the development of polymeric materials has expanded greatly, which can be seen in our daily lives, through the composition of numerous utilities. With the development of new technologies, polymerization produced a series of new polymers. However, in some situations, rather than synthesizing a new polymer material, the search was directed to the study of physical mixtures of polymers, in other words, polymer blends. These consist of a mixture of two or more polymers and/or copolymers (Utracki, 2000).

1.Universidade Federal de Campina Grande – Campina Grande/PB – Brazil

Author for correspondence: Divânia Ferreira da Silva | Departamento de Engenharia de Materiais | Universidade Federal de Campina Grande | Rua Aprígio Veloso, 882 – Bodocongo | CEP 58.109-970 Campina Grande/PB – Brazil | E-mail: divaniaf@yahoo.com.br

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The blends of polymers with different physical properties can help improving the properties of the material through the contribution of each system component. Among the advantages of the blends, we can mention the ease of adjusting the properties of plastics usage needs. They are primarily important for generating mechanical, chemical, or unwanted barriers and improving the processibility of high-performance polymers (Vossen, 2009). For example, the application of some polymers is limited by the low impact on performance under room temperature conditions. This situation gets worse, especially when using in temperatures below 0°C (Newman, 1978).

One way around this limitation is to prepare polymer blends, in which the dispersed phase is an elastomer, and it is then classified as an immiscible blend. The blends with this feature have a high interfacial tension and weak adhesion between the continuous and dispersed phases. Studies have been directed to assess the shape, size, and distribution of the dispersed phase domains, relating them to macroscopic properties of the resulting material. The small amounts addition of a third component to the miscible blend can change the dispersion and interfacial energies between the phases, acting as a compatibilizer agent. These are generally block copolymers or grafted and can cause a decrease in size of the dispersed phase, modifying the toughness of the blends (Maglio and Palumbo, 1984).

The interfaces at the nano scale are fundamental to the macroscopic physical properties of binary polymer blends, for example, morphology, mechanical strength, fracture toughness, thermal stability, and surface wetting. The interface property is intimately connected to an adhesion at the interface and interfacial tensions, which quantify miscibility and compatibilization capabilities. The characterization and understanding of the interfacial properties of phase-separated blends on a microscopic level are of great importance, both from practical as well theoretical points of view. On a larger length scale, one can envisage the blend material as an ensemble of interfaces. The structure and thermodynamics of these interfaces determine many practically important properties of the blend (Ramya *et al.*, 2012).

Among the several polymer matrices, polyamide 6 – PA6 (also known as nylon 6) was chosen as an engineering polymer, providing some advantages such as dimensional stability, good impact strength without notch, excellent chemical resistance, high melting temperature, among many others. Aiming at improving its performance, it has been used with toughening of polyamide-based elastomer materials (Fornes *et al.*, 2001).

The styrene-butadiene copolymer (SBR) is considered a general-purpose elastomer. It has some properties as reasonable aging resistance, good abrasion resistance, and good resistance to sea water (Anjos, 2007).

These materials have good aging stability when protected by additives. About 50% of car tires are made from various types of SBR. The styrene/butadiene ratio influences the properties of the polymer, with high styrene content, and the rubbers are harder and less rubbery (Obrecht *et al.*, 2012).

Bassani *et al.* studied blends of nylon-6 with acrylonitrile/EPDM/styrene (AES) using a series of methyl methacrylate maleic anhydride (MMA-MA) copolymers, at the same time that compatibilizing agents were being prepared. The MA units in the copolymers are capable to react with the nylon-6 end groups. The MMA-MA copolymer has a potential to form *in situ* copolymers at the blend interface during melt processing as indicated by torque rheometry tests. This study focused on the effects of functionality and concentration of the reactive MA units of the compatibilizer on the their blends' mechanical properties. The results show that incorporation of the MMA-MA copolymer significantly improves the impact strength of nylon-6/AES blends. The blend containing 1.3 wt% of MA in the copolymer is supertough at room temperature, and it remains tough at subzero temperatures.

The aerospace industry is characterized by always making use of the latest technology, and recently its evolution is connected to the advent of research and knowledge related to new materials, as for example, polymer blends, leading to the appearance of new applications for new products before reserved for traditional materials such as steel, bronze, or brass. The polymer blends have the ability to meet some requirements like reduced weight without loss of strength and stiffness, and for this reason it is gaining prominence, and wide acceptance in structural projects. Still, many companies are producing aircraft with polymer blends, and these can be used in internal and external components, landing gear, structural parts, leading edges etc. The blends stand out when compared to other materials for presenting some advantages, such as higher values of impact resistance and service temperature, low moisture absorption, low processing costs, transport, and storage (Oliveira and Botelho, 2007).

Besides the described advantages, the polymer blends do not permanently harden and can be repeatedly reheated and shaped, have high fatigue strength, ease in carrying out repairs, and especially greater possibility of recycling waste contributing to eliminate or minimize the environmental impact. This shows mechanical properties equal or superior to those shown by the conventionally used thermosetting composites in aerospace (Oliveira and Botelho, 2007).

The market for polymer blends has grown steadily over the past decades, being the largest ones: automotive, electrical and electronics, packaging, construction, and aviation industries (Fiegenbaum, 2007).

Although it is known that the polymer blends are economically viable alternatives for obtaining new materials, many polymers, when mixed, form immiscible blends and/or present incompatible properties unsuitable for use in the aircraft industry. The final ones of an immiscible polymer blend are influenced by their morphology, which in turn is very complex and depends on many factors, such as: composition, thermal and rheological properties of the components, processing conditions, among others (Fiegenbaum, 2007). This study aimed at developing blends of polyamide 6/compound by recycled rubber (SBR-R), with the addition of a compatibilizer.

MATERIALS AND METHODS

MATERIALS

A PA6 was used, Technyl C216, molecular weight (10.500 g/mol), viscosity index (VI=134 mL/g) in the form of pellets, supplied by Rhodia (São Paulo, Brazil).

Waste rubber (SBR-R) was used from the shoes industry São Paulo Alpargatas SA, located in Campina Grande, Paraíba. These compounds consist of a mixture of SBR as a main component, fillers, additives of processing, curing agents, stabilizers, and other types of rubber. The residues were used as powder with a particle size of 425 μm passed in 35-mesh sieve.

The used compatibilizers were: PE-g-MA, Polybond 3009, with melt flow index of 5 g/10 min. and grafted with 1% by weight of MA; and polypropylene (PP-g-MA), Polybond 3200, with melt flow index of 110 g/10 min. grafted with 0.2 wt% MA both supplied by Crompton (São Paulo, Brazil).

PREPARATION OF POLYMERS BLENDS

Before each processing step of the PA6 and compatibilizers, they were dried in vacuum oven at 80°C for 48 hours. The binary blends were prepared in the proportions of: PA6/SBR 90/10, 80/20, 70/30, and 60/40. Ternary mixtures were done in: PA6/SBR/compatilizers 87.5/7.5/5.0; 77.5/17.5/5.0; 67.5/27.5/5.0, and 57.5/37.5/5.0 using a internal mixer of Haake Buchler. Subsequently, the following compositions were chosen: 90/10; 80/20; 87.5/7.5/5; 77.5/17.5/5 to be processed by simultaneous extrusion of all the components in a twin screw extruder modular co-rotating ZSK-18K from Coperion with a 240°C temperature at all zones, 300 rpm speed, and feed rate of 4 kg/h. Also, the PA6 was extruded under the same conditions of the blends as reference. These compositions were chosen with lower levels of SBR-R, because they are in powder form and the polyamide and compatibilizer as granules were blended and introduced simultaneously into the extruder. Higher concentrations of SBR-R could compromise the homogeneity of the mixture.

CHARACTERIZATION OF MIXTURES

Torque rheometry tests were carried out in an internal mixer RHEOMIX 600 (Haake Büchler) coupled to a Torque Rheometer Haake System 90 of Büchler, operating with roller type rotors, rotating at 60 rpm at a temperature of 240°C for 20 minutes in air atmosphere. The total mass within the mixing chamber was kept constant at 55 g for all compositions.

Rheological curves were obtained from the following materials: PA6, SBR, PE-g-MA, PP-g-MA binary and ternary blends.

The Fourier transform infrared (FTIR) spectroscopy was used to characterize binary and ternary blends with 5 wt% PE-g-MA and PP-g-MA in a SPECTRUM 400 spectrometer from PerkinElmer with a scan of 4,000 to 650 cm^{-1}. FTIR analyzes were performed on films made of blends.

The tensile tests were conducted on specimens injected, according to the American Society for Testing and Materials (ASTM) D638 using a universal testing machine model AG-Is 100KN from Shimadzu, with loading speed of 50 mm/min. The tests were conducted at room temperature and the results were analyzed from an average of five specimens.

Impact tests were performed on specimens notched Izod type according to ASTM D256, on a CEAST instrument brand model Resil 5.5, operating at 2.75 J hammer and the conclusions were analyzed based on a weighted average of five specimens.

The dynamic mechanical thermal analysis (DMTA) was performed on a DMTA equipment from TA Instruments Explorer brand, model Q 800. The request mode dynamic-mechanical bending was used at one point, suitable for the type Izod specimens applied in the tests. The heating rate was 10°C/min. and it had a frequency of 1 Hz in a temperature range from -100 to 150°C.

Thermal analysis using differential scanning calorimetry (DSC) was performed on DSC Q20 machine from TA Instruments, under the following conditions: heating from room temperature to 300°C at a rate of 10°C/min. under nitrogen atmosphere. The amount of sample employed was 5 mg.

The scanning electron microscopy (SEM) analysis was performed on fracture surface of the specimens subjected to impact test in a SEM, Shimadzu SSX-550 Superscan, at a voltage of 15 kV, under high vacuum, and the surfaces fracture of notched specimens coated with gold.

RESULTS AND DISCUSSION

TORQUE RHEOMETRY AND SPECTROSCOPY OF THE FOURIER TRANSFORM INFRARED

Figures 1 to 4 present the variation curves of torque *versus* time of pure polymers, and the blends with and without compatibilizer. Figure 1 illustrates the torque curves for pure polymers used in this work. It can be observed that after three minutes of the beginning of the process, the torque tends to be practically constant with small oscillations around a mean value. This behavior indicates viscosity stability to the process used conditions, in other words speed of 60 rpm and temperature of 240°C. The high-density PE grafted with MA had the highest torque compared to other polymers and, therefore, higher viscosity under these conditions. Figure 2

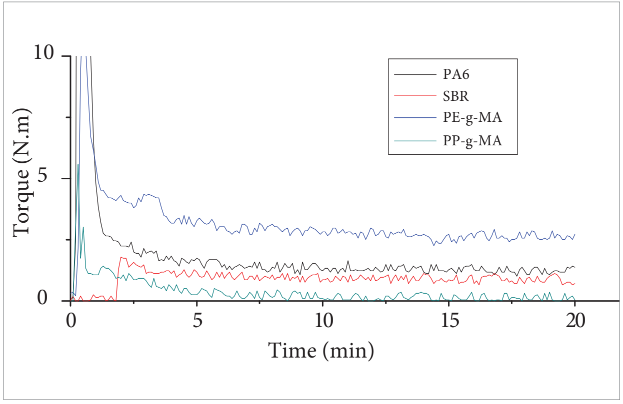

Figure 1. Torque curves of pure polymers.

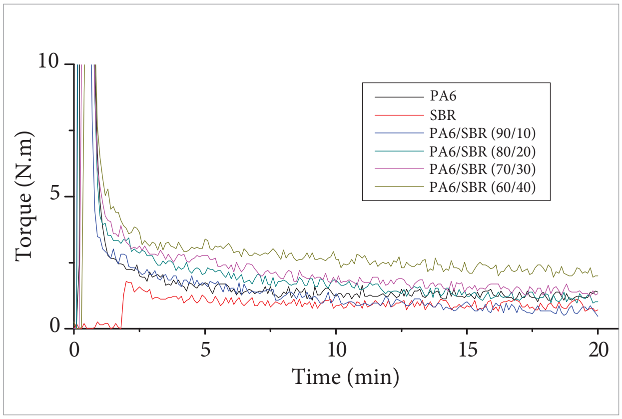

Figure 2. Torque curves of the binary blends.

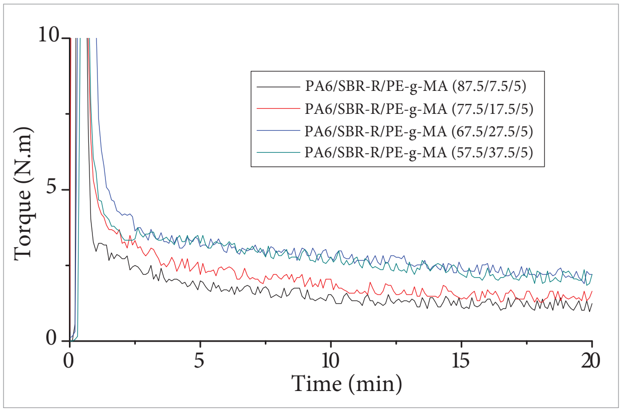

Figure 3. Torque curves of the polyamide 6/SBR-R/PE-g-MA blends.

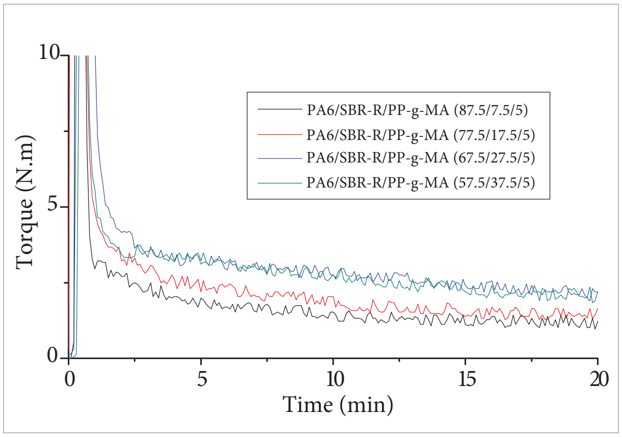

Figure 4. Torque curves of the polyamide 6/SBR-R/PP-g-MA blends.

shows the torque rheometry curves for binary blends of PA6/SBR (90/10, 80/20, 70/30, 60/40). It can be observed that such values of the blends increase with a superior percentage of the added SBR, implying an increase of viscosity, probably because the SBR is acting as a load. Figures 3 and 4 show the torque rheometry curves of ternary blends (PA6/SBR/PE-g-MA) varying the content of SBR and let remaining constant at 5% (by weight) the content of compatibilizer PE- and PP-g-MA. It was found that the torque values of the blends PA6/SBR/PE-g-MA (67.5/27.5/5) and (57.5/37.5/5) with higher percentages of SBR-R were superior when compared with others, which show that increasing percentage of SBR-R in the blend involves a higher increase in viscosity and torque with the addition of PE-g-MA than with PP-g-MA, indicating possible bigger reactions of this compatibilizer.

When the PE-g-MA is added to the blend of PA6/SBR-R, here is an increase in torque. According to Roeder *et al.* (2002), Jiang *et al.* (2003), and Bassani *et al.* (2005), anhydride groups of PE-g-MA react with the amine terminal ones of PA6 forming the imide group and resulting in a copolymer *in situ* at the interface. The reaction between the anhydride groups of PE-g-MA and amine terminal ones of PA6 involves the formation of water as a byproduct, which can lead to degradation of the PA6 chains by hydrolysis. However, Fig. 3 shows that the torque of the blends and PA6/SBR-R/PE and PP-g-MA were constant, which is an indication of degradation nonoccurrence. One explanation for this behavior is that the PE-g-PP and MA-g-MA has only 1% MA, which would not be sufficient to induce degradation. If we compare the torque curves in function of time (Figs. 3 and 4) of the compatibilized blends, one may see that the blend torque (PA6/SBR-R/PE-g-MA) was greater than that of PA6/SBR-R/PP-g-MA blends, which indicate that PE-g-MA is more reactive with PA6 compatibilizer than the PP-g-MA.

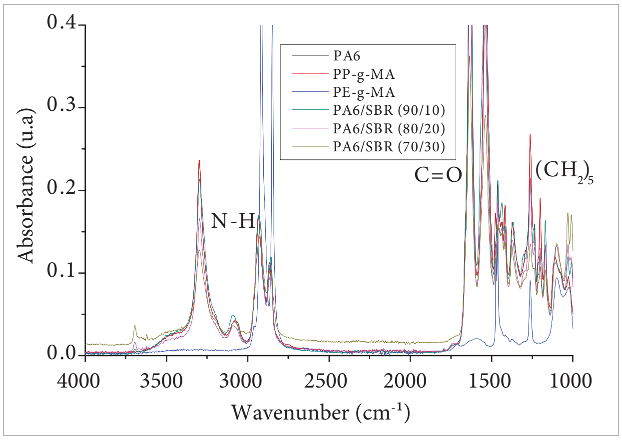

Figure 5. Fourier transform infrared spectra of binary blends.

Figures 5 to 7 present the FTIR spectra of PA6/SBR-R binary blends containing 10, 20, and 30% by weight of SBR and PA6/SBR-R/compatibilizer ternary blends containing 5 wt% of the PE- and PP-g-MA compatibilizers. It is possible to observe that the characteristic bands of PA6 were not modified by the presence of SBR and not by increasing its share in the binary blends and the presence of PE-g-MA and PP-g-MA in the ternary blends, showing that their chemical groups were not affected. The profiles of the spectra remain with predominant features of the polyamide, which has a band above 3,000 cm^{-1} as a characteristic NH stretch of bending and other bands near 1,700 cm^{-1} referring to carbonyl groups (C=O). The presence of SBR-R does not cause significant changes in the polyamide spectrum, therefore this compound

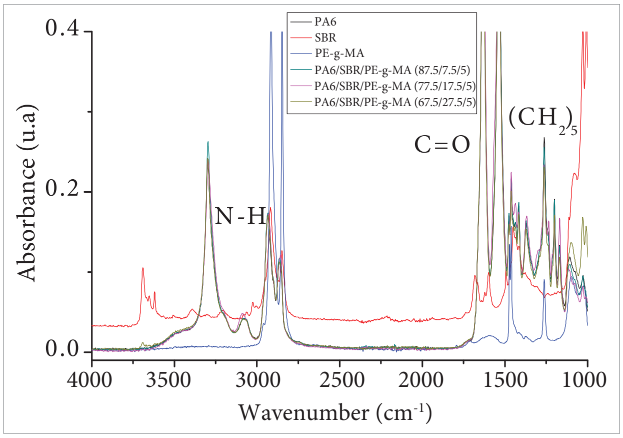

Figure 6. Fourier transform infrared spectra of ternary blends with PE-g-MA.

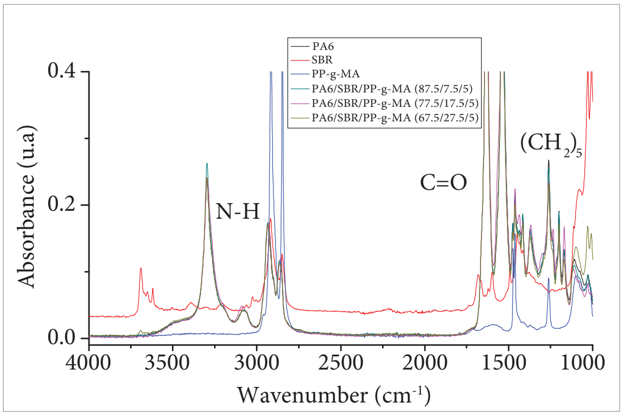

Figure 7. Fourier transform infrared spectra of ternary blends with PP-g-MA.

does not cause alterations in the chemical structure of the polymer after processing.

From these results, it seems that the molecular weight of the rubbery phase is not an important parameter in this SBR-R concentration. One possible explanation for this behavior is that at this SBR concentration the percolation condition is reached for both molecular weights. The band increases with the SBR-R concentration correspond to the stretching molecular vibrations. This is an important band for it allows determination of the rubbery phase content in the blend.

MECHANICAL TESTS UNDER TENSILE AND IMPACT

In Table 1 and Figs. 8 to 10, it is possible to check the results of the mechanical properties of tensile and impact PA6, binary (PA6/SBR-R) and ternary (PA6/SBR-R/PE-g-MA) blends with 5% of PE-g-MA weight. Selected compositions were only 90/10% and 80/20%, they were chosen with lower levels of SBR-R, because it is in powder form and polyamide and compatibilizer are as granules were blended and introduced simultaneously into the extruder. Higher concentrations of SBR-R could compromise the homogeneity of the mixture.

It is observed in Table 1 and Figs. 8 to 10 that the impact strength, elastic modulus, and maximum stress in the flow of compatibilized blends somewhat reduced when compared with the PA6 values. However, when one considers that the incorporation of recycled material cross-linked in amounts of up to 20% by weight in PA6, this reduction is acceptable because it does not compromise the properties and significantly contributes to cost reduction of the final compound.

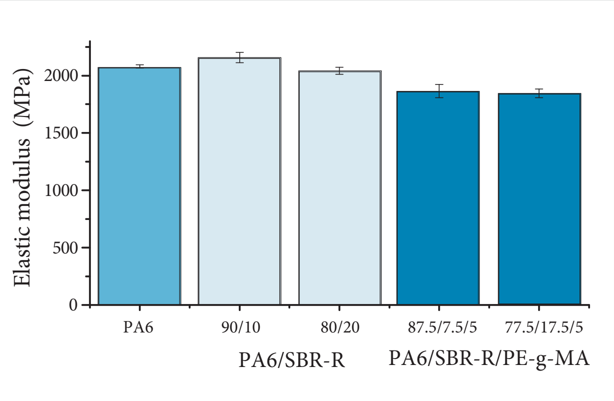

Figure 9. Elastic modulus of polyamide 6 and of binary and ternary blends.

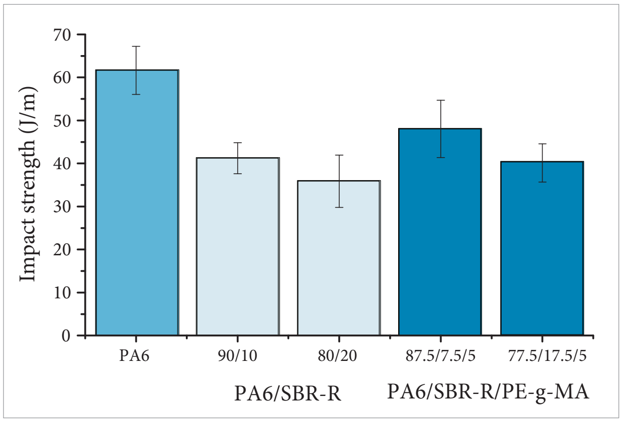

Figure 8. Impact strength of polyamide 6 and of binary and ternary blends.

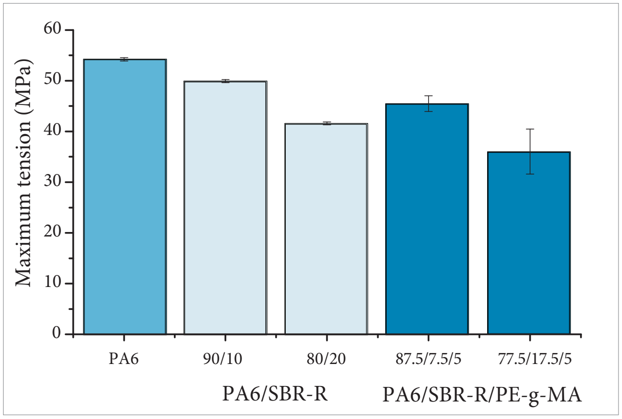

Figure 10. Maximum tension of polyamide 6 and of binary and ternary blends.

Table 1. Mechanical properties of polyamide 6 and of binary and ternary blends.

Compositions	E (MPa)	MT (MPa)	Tenacity* (J)	IS (J/m)
PA6	2,080±13.4	54.2±0.3	94.1±4.1	51.9±11.8
PA6/SBR-R 90/10	2,157±44.7	49.9±0.3	26.5±11.2	41.2± 3.6
PA6/SBR-R 80/20	2,042±30.1	41.6±0.3	14.9±5.2	35.9±6.1
PA6/SBR-R/PE-g-MA (87.5/7.5/5)	1,864±57.7	45.5±1.6	32.3±5.4	48.0±6.7
PA6/SBR-R/PE-g-MA (77.5/17.5/5)	1,845±37.8	39.5±0.3	18.4±8.1	40.1±4.4

E: elastic modulus; MT: maximum tension, IS: impact strength; *calculated by area under the curve of tension-deformation.

DYNAMIC MECHANICAL THERMAL ANALYSIS

Through DMTA, one can estimate changes in molecular relaxations that occur in polymeric materials in a wide temperature range. Knowledge of these changes allows relating molecular parameters and mechanical properties of polymers.

Figure 11(a) shows the values of tanδ and the storage modulus (E') of PA6 and binary and ternary blends with 5% by weight of PE-g-MA in function of temperature, respectively. It was observed that the PA6 presents two peaks at approximately -7 and 61°C. The first transition may be related to β relaxation and the other would be the very Tg, assigned to the α relaxation. For the binary and ternary blends, there was displacement of the peaks to about -10 °C and the relaxation of displacement α of PA6 to larger values.

In Fig. 11(b), it was observed that the peak height of the storage modulus seen after the Tg peak of PA6, which appeared to 25°C, may be related to the elastic modulus of the blend. Therefore, the higher, greater is the stored energy and higher elastic modulus of the material. Thus, as the amount of rubber increased, the greater the height and intensity of the peaks and elastic modulus, as also seen in Fig. 9, for the elastic modulus.

DIFFERENTIAL SCANNING CALORIMETRY

Figure 12(a)-heating and (b)- cooling presents the DSC curves of the blends of PA6 and PA6/SBR-R with 10 and 20 wt% of SBR-R ternary and with 5 wt% of the compatibilizer PE-g-MA. It is noted that the present PA6 melting temperature (Tm) at approximately 224°C and its crystallization starts at about 195°C. The melting enthalpy (DHm) for PA6 is approximately 191 J/g. In the ternary blends peak melting and crystallization compatibilizing agent for the PE-g-MA are observed, in which the crystalline melting temperature

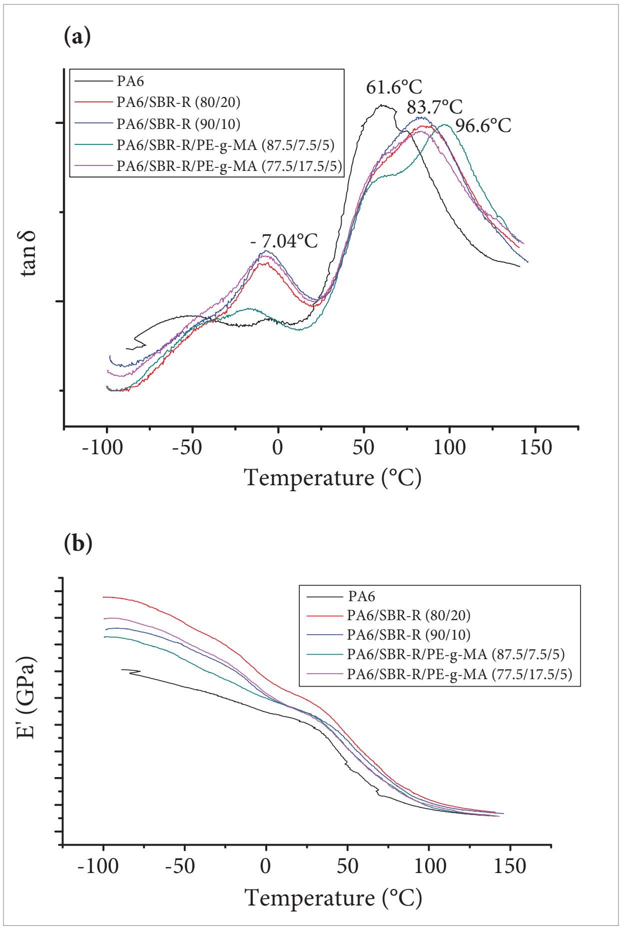

Figure 11. tanδ and E' curves a function of temperature for the polyamide 6 and binary (PA6/SBR-R) and ternary (PA6/SBR-R/PE-g-MA) blends.

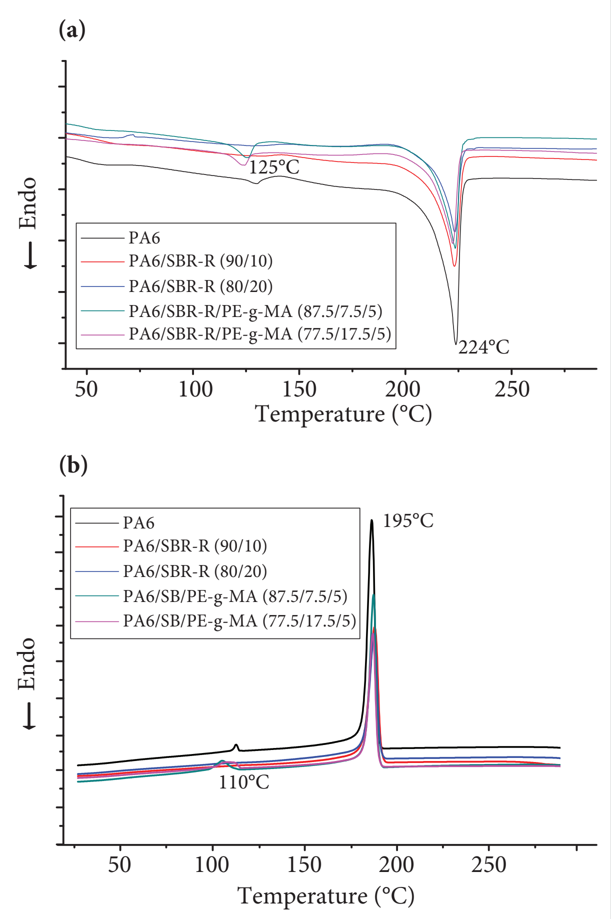

Figure 12. Differential scanning calorimetry curves of polyamide 6, binary (polyamide 6/SBR-R) and ternary (polyamide 6/SBR-R/PE-g-MA) blends: (a) Heating; (b) cooling.

(Tm) is approximately 125°C and the crystallization ones (Tc) is approximately 110°C.

In all curves, the peaks crystallization of PA6 in both binary and ternary blends indicated that the compatibilizer is not directly influencing the crystallinity of PA6.

SCANNING ELECTRON MICROSCOPY

In Figs. 13 to 17 the SEM photomicrographs of PA6 and binary and ternary blends with 5% by weight of PE-g-MA and increases of 100 and 2,000 X are shown. These photomicrographs were obtained from the fracture surface of the specimens of PA6 and of binary and ternary blends subjected to Izod impact testing.

In Fig. 13 there is a regular morphology with a ductile fracture aspect which is characteristic of PA6. In Figs. 14 to 17, it can be observed poor adhesion between the phases, probably due to poor interfacial interaction between these, indicating the immiscibility of the system and also due to this weak interaction interfacial voids exist between the domains and the matrix rubber, which decrease with the addition of compatibilizing agent percentage in the blend.

The addition of the compatibilizing agent to the blend stimulated a better interfacial adhesion between the domains and the matrix and size of the fields altered in relation to binary blends. The morphology appears with some degree

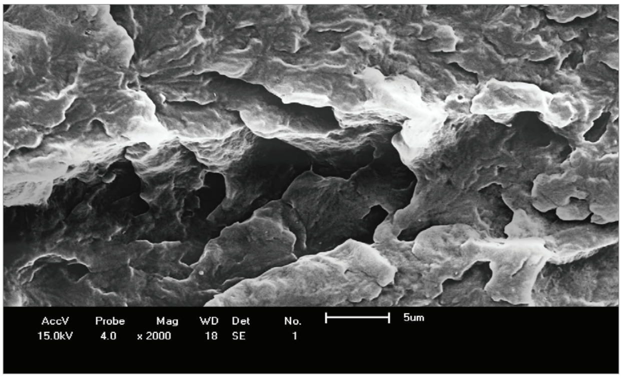

Figure 13. Scanning electron microscopy micrograph of polyamide 6.

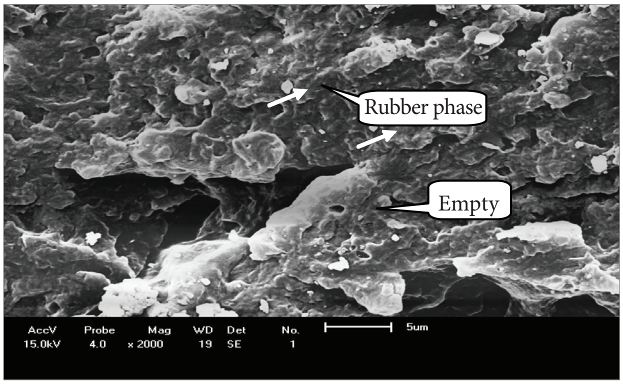

Figure 14. Scanning electron microscopy micrograph of polyamide 6/SBR-R of the blend (90/10).

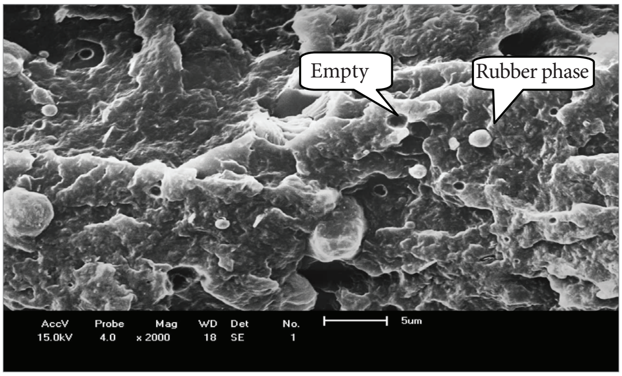

Figure 15. Scanning electron microscopy micrograph of polyamide 6/SBR-R of the blend (80/20).

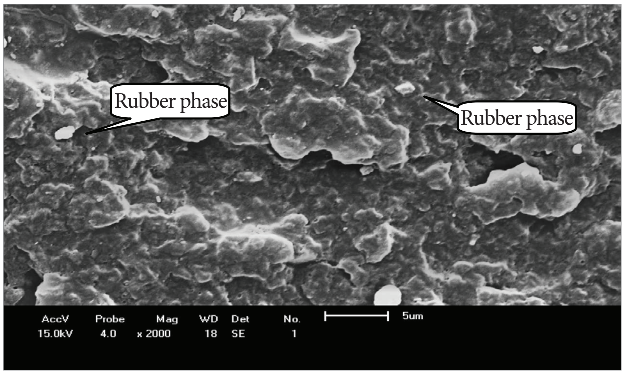

Figure 16. Scanning electron microscopy micrograph of the blend polyamide 6/SBR-R/PE-g-MA (87.5/7.5/5).

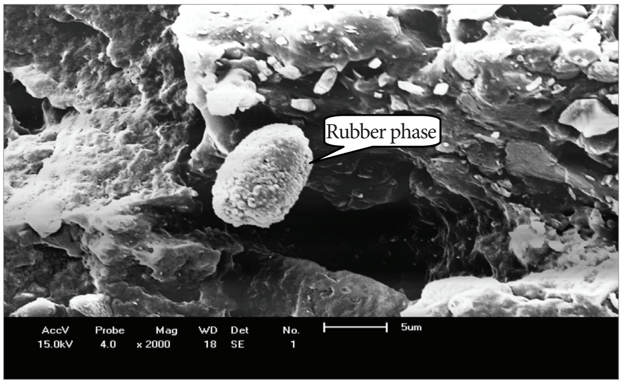

Figure 17. Scanning electron microscopy micrograph of the blend of polyamide 6/SBR-R/PE-g-MA (77.5/17.5/5).

of co-continuity for the addition of the compatibilizing agent causes a refinement of the co-continuous structure, which can be explained by the reduction in interfacial tension, stabilizing interface and preventing coalescence (Oliveira, 2009). However, the introduction of this was not enough to significantly improve the mechanical properties of the blend PA6/compound by recycled rubber. It can be attributed to the quantity or type of compatibilizing agent used. Therefore, it was observed that the morphology presented by binary and ternary blends rubber particles has not been homogeneously distributed, the average distance between the particles is not uniform and, in certain regions of the fracture surface of the particles, almost touch and in other regions, their distance is greater.

The main feature observed for this material, however, was the relative lack of compatibility particle/matrix. We observed particles torn and totally broken matrix interfaces. These two aspects are evidences of a low interfacial adhesion.

CONCLUSIONS

Blends of PA6/compound recycled rubber (SBR) were produced. In rheological, it was found that the addition of the compatibilizer PE-g-MA showed higher torque values both for the binary mixtures and for the ternary ones. This result may indicate a higher efficiency of PE-g-MA with respect to the PP-g-MA compatibilizer.

The impact resistance and elastic modulus of the compatibilized blends reduced somewhat when compared to PA6. Also, the impact resistance of the compatibilized blends increased as compared to noncompatibilized ones.

The addition of the compatibilizing agent was not sufficient to provide significant differences in the mechanical properties. Even so, the blends of PA6/compound recycled rubber are a good alternative for recycling the SBR-R, resulting in a new material with specific properties.

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