

Partial replacement of EPR by GTR in highly flowable PP/EPR blends: Effects on morphology and mechanical properties

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ABSTRACT: This research analyzes the effect of ground tire rubber (GTR) and a novel metallocene-based ethylene-propylene copolymer (EPR), with high propylene content, on the morphology and mechanical behavior of ternary polymer blends based on a highly flowable polypropylene homopolymer (PP). The PP/EPR blends morphology, with very small domains of EPR dispersed in the PP matrix, indicates a good compatibility among these materials, which leads to a significant improvement on elongation at break and impact strength. The incorporation of EPR on the rubber phase of thermoplastic elastomeric blends (TPE) based on GTR and PP (TPE^{GTR}) has a positive effect on their mechanical performance, attributed to the toughness enhancement of the PP matrix and to the establishment of shell-core morphology between the rubber phases. The mechanical properties of the ternary blends reveal that TPE^{GTR} blends allow the upcycling of this GTR material by injection molding technologies. © 2015 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 2015, 132, 42011.

KEYWORDS: blends; compatibilization; mechanical properties; morphology; recycling

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INTRODUCTION

End of life tires (ELT) have been disposed for many years in landfills causing several environmental problems, such as water and air pollution or direct adverse effects for human health due to infestation risks. Specific regulations have been implemented to organize the waste collection and treatment as well to prioritize the reuse and/or valorization of these materials.¹

The ELT valorization has been done looking at two main areas of interest: energy recovery or material recovery. Due to the high-energy content of ELT, they can be used as an energy source in major fuel consumer industries. They are currently used as a complementary source of energy for cement kilns and thermal power stations, or as substitute for coal in steel plants. On the material recovery approach, the ELT, intact, shredded, or grounded, is a viable alternative to be used in several application fields, contributing to the reduction of economical, energetic, and environmental costs of exploration and processing of new raw materials. The total or partial substitution of new raw materials by ELT has been done mainly, but not limited to, in civil engineering projects like embankments and crash barriers or in other projects such as sports fields and molded parts and

products.² The use of GTR in injection-molded products has the advantage of reusing greater quantities of this material. However, its vulcanized nature and lack of compatibility with thermoplastics is still a limiting factor that has to be taken in consideration. Several strategies have been employed, many of them trying to reverse the nature of GTR particles to an unvulcanized state or acting on the interface between the thermoplastic matrix, usually a polyolefin, and GTR.^{3–8} Regeneration of the GTR material has been done by thermomechanical, thermochemical, or ultrasonic processes among others. Other strategies seek to obtain a chemical affinity between the materials either by surface functionalization of GTR or through the use of compatibilizing agents. This can be done usually by chemical grafting of reactive monomers such as maleic anhydride (MA) or acrylamide on the GTR surface or, in the later case, by using agents with chemical affinity to GTR and to the thermoplastic phase.^{9–11} The partial substitution of the fresh rubber phase by GTR in TPE blends is another research focus that can lead to ecological and economic benefits. Several works combine regeneration techniques of GTR with chemical functionalization of their surface, to achieve a better mechanical behavior of the TPE^{GTR} blends.^{12–14} Different rubber materials have been

Table I. Blends Nomenclature and Composition

Blends composition: PP (P); EPR (V); GTR (G) $P_{x_1}(E_{y_1}G_{y_2})_{x_2}$ (1) Constraints: $x_1 + x_2 = 100$ $y_1 + y_2 = 1$		Designation	PP (wt %)	EPR (wt %)	GTR (wt %)
Reference material		P ₁₀₀	100	0	0
Binary blends $P_{x_1}(V_{y_1}G_{y_2})_{x_2}$, $x_1 = (70;50;30)$, $y_1 = (0;1)$	$P_{x_1}V_{x_2y_1}$ with: $y_2=0$	P ₇₀ V ₃₀	70	30	0
		P ₅₀ V ₅₀	50	50	0
		P ₃₀ V ₇₀	30	70	0
Ternary blends $P_{x_1}(V_{y_1}G_{y_2})_{x_2}$, $x_1 = (70;50;30)$, $y_1 = 0.7$	$P_{x_1}G_{x_2y_2}$ with: $x_1=70$ and $y_1=0$	P ₇₀ G ₃₀	70	0	30
	$P_{70}(V_{y_1}G_{y_2})_{30}$	P ₇₀ (V _{0.7} G _{0.3}) ₃₀	70	21	9
	$P_{50}(V_{y_1}G_{y_2})_{50}$	P ₅₀ (V _{0.7} G _{0.3}) ₅₀	50	35	15
	$P_{30}(V_{y_1}G_{y_2})_{70}$	P ₃₀ (V _{0.7} G _{0.3}) ₇₀	30	49	21

analyzed, chosen due to their affinity with GTR, like styrene butadiene rubber (SBR) and natural rubber (NR) or with the thermoplastic phase, like ethylene propylene diene monomer (EPDM). PP or EPDM grafted with MA have also been analyzed as compatibilizing agents.

A sustainable ELT recycling approach requires the minimization or even exclusion of hazard chemical agents along the process. However, a general loss of the blends mechanical properties has been an important drawback of this approach that must be overcome. For a chemical agent-free PP/GTR blend, with 70 wt % PP content, the maximum tensile strength typically ranges between 13 to 15 MPa and for a 50 wt % PP content around 9 MPa.^{5,15} On the predictions made by Costa *et al.*¹⁶ for a TPE^{GTR} blend with PP, EPDM, and GTR, the maximum tensile strength of a 70 wt % PP blend ranged between 20 to 25 MPa and between 10 to 15 MPa for a 50 wt % PP content.

This work intends to contribute toward a sustainable approach on the development of TPE^{GTR} with high processability for the injection molding industry. A previous work on ternary blends based on EPDM, GTR, and a high melt flow PP, suitable for injection molding of thin complex parts, has shown that encapsulation of GTR by an EPDM fresh rubber can be a viable strategy to produce TPE^{GTR}.¹⁷ However, to achieve required properties the ratio of fresh rubber versus GTR and the size of the GTR particles must be taken in consideration. The present work continues studying sustainable experimental design strategies for the recycling of GTR without the use of thermochemical techniques and reports the effect of a different fresh rubber material, an EPR with a high melt flow index (MFI), on the morphology and mechanical behavior of these blends. EPR copolymers may significantly enhance the mechanical toughness of the TPE^{GTR} blends,^{18–21} especially those with higher propylene contents and also improve their processability. Scanning electronic microscopy (SEM) was used to analyze the morphology and also to study the encapsulation effect of GTR by EPR. Tensile tests, hardness, and impact tests were made to assess the mechanical performance of the resulting materials. Differential scanning calorimetry (DSC) was performed to verify the effect of crystallinity on the mechanical properties of the TPE^{GTR} blends.

EXPERIMENTAL

Materials

The elastomeric materials used on this work are as follows: GTR from mechanical ground scrap tires, FB 00-08, from Bio-safe S.A., Portugal, obtained by an ambient grinding process, sieving class 635 to 20 Mesh and density from 0.6 to 0.7 g cm⁻³ and a commercial available EPR, Vistamaxx 6202, from Exxon Mobile, with 15 wt % ethylene content, with a MFI of 26 g 10 min⁻¹ (230°C, 2.16 kg), density of 0.86 g cm⁻³ and elongation at break higher than 2000% (ASTM D412). For the thermoplastic phase, it was used as an isotactic polypropylene homopolymer, PPH10060 supplied by Total Petrochemicals, especially suited for injection molding of very thin and complex parts, with an MFI of 35 g 10 min⁻¹ (230°C, 2.16 kg) and density of 0.9 g cm⁻³.

Set of Experiments

An experimental procedure was established to evaluate the effect of EPR on TPE^{GTR} blends (Table I). A first set of binary blends was developed to study the effect of each of the rubber components, EPR and GTR, on the morphology and mechanical properties of PP-based blends. A second set of ternary blends was then formulated to study the effect of 30% EPR replacement by GTR.

Melt Mixing and Samples Preparation

Blending was carried out in two stages: a first mixing stage of EPR and GTR, as a procedure to promote a better encapsulation of the GTR particles by EPR.¹⁰ This operation was performed on a Brabender-type equipment at 180°C during 8 minutes, at a rotation speed of 60 rpm, to achieve a homogeneous mixture of the injection grade. The resultant formulation was then milled to obtain the granules for the next blending phase. On a second stage, PP was placed in the mixing equipment at 180°C and 60 rpm. After 2 minutes, the GTR/EPR mixture, prepared in the first stage, was added and mixed at 60 rpm for an additional 8 minutes period. Similar experimental mixing conditions were also established in other works without reporting any thermal degradation of the materials.^{10,22}

In the final stage, the materials were milled into granules and injected on a Inauton D65, a 65 tons injection-molding

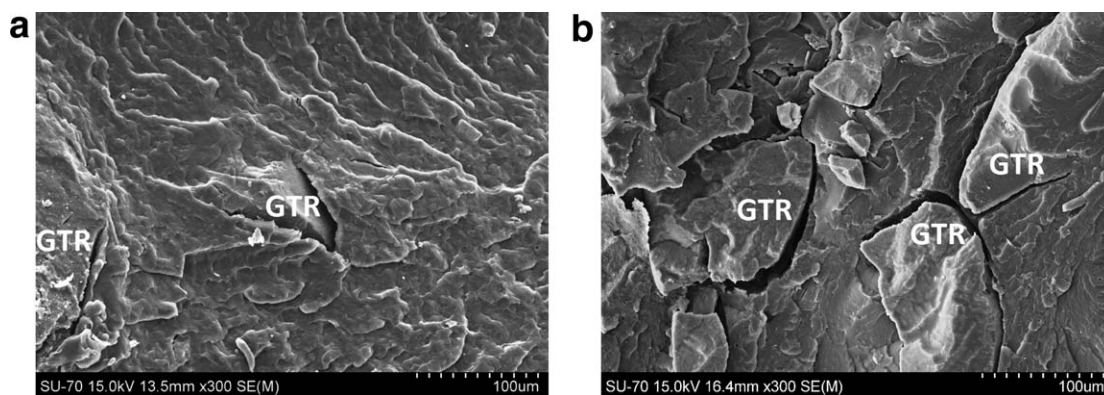


Figure 1. SEM micrographs of P₇₀G₃₀ blend, etched with xylene (15 min, 50°C). (a) P₇₀G₃₀ blend and (b) P₇₀G₃₀ blend.

machine, with the following injection-molding parameters: 220°C injection temperature, 35 bar holding pressure, and mold temperature of 40°C.

Morphological Characterization

A scanning electron microscope Hitachi S-4100 was used to obtain the morphological data of the analyzed blends. The samples were obtained by breaking the molded specimens in liquid nitrogen to avoid phase deformation. To accurately identify the blends constituent phases, the samples were etched with xylene (99%) at 50°C during 15 minutes for selective extraction of the uncured EPR phase. The samples were then mounted on aluminum stubs and sputter-coated with a thin layer of gold to avoid electrostatic charging during observation.

Mechanical Testing

The tensile strength and elongation at break were measured on an Autograph AG-IS universal testing machine from Shimadzu, using a 10 kN load cell. Tensile tests (Type 1 specimens) were made at a constant crosshead speed of 500 mm min⁻¹ in accordance with ASTM D412. Elongation was measured by a noncontact video extensometer, Shimadzu DVE-201, coupled to the universal testing machine. The hardness (shore *D*) of the samples was measured with a Teclock Hardness Tester accordingly to ASTM D 2240. Izod impact tests were done on a Ray Ran universal pendulum impact system. Impact specimens were prepared with 57 mm length, 13 mm width, and 3.2 mm thickness. A notch of 2.4 mm depth with an angle of 45° was made on the specimens for impact testing. All measurements were done at ambient temperature. The average values of these mechanical properties were evaluated using at least five samples.

Differential Scanning Calorimetry

The material's crystallinity was obtained using a Shimadzu DSC-60 equipment at a scan rate of 20°C min⁻¹. Samples, about 10 mg, of all the blends identified in Table I were used in this study. Two heating cycles were used on a temperature range between -120°C and 200°C. The samples were heated in the first cycle to 200°C and held at that temperature for one minute to eliminate the influence of thermal and mechanical history. Then, they were cooled with liquid nitrogen until -120°C and heated again to 200°C. The melting behavior was recorded during the second heating scan. The corrected crystallinity of the

materials was obtained taking in consideration the blend PP weight content, which can be done using the following relationship:

Corrected crystallinity:

$$X_c^c(\%) = \frac{\Delta H_m}{(1-\phi)\Delta H_0} \times 100 \quad (1)$$

where ϕ is the weight fraction of rubber in the blend.

ΔH_m —melting enthalpy calculated under the area of the endothermic peak obtained by DSC analysis (J g⁻¹).

ΔH_0 —melting enthalpy of 100% crystalline PP, taken as 209 Jg⁻¹.²³

Thermogravimetric Analysis

To evaluate the existence of thermal degradation at the processing temperatures, a TGA was performed on a Shimadzu TGA-50 equipment, within the temperature range 20–430°C, at a rate of 5°Cmin⁻¹, on 7 mg samples, under a controlled dry nitrogen flow of 20 mL min⁻¹.

RESULTS AND DISCUSSION

Phase Morphology

Binary Blends. The P₇₀G₃₀ blend morphology (Figure 1) shows the lack of adhesion between GTR and the PP matrix, proving that the recycling of GTR on PP-based blends without any compatibilization strategy will lead to a significant decrease in their mechanical properties.

The morphological observations of the P₇₀V₃₀ and P₅₀V₅₀ binary blends [Figure 2(a,d)] revealed a uniform dispersion of EPR with dimensions below 0.1 µm. These results seem to agree with those reported by Orazio *et al.*²⁴ about the effect of the EPR propylene content on the dispersion mode of the rubbery component. High propylene contents may contribute to a significant reduction of the interfacial tension between the components and to the existence of very small EPR domains highly dispersed in the thermoplastic matrix. The effect of the viscosity ratio between the materials, which is the ratio between the viscosities of the minor and major phases, may also be contributing to this type of morphology. Generally, the domains sizes reach a minimum when the viscosity of the minor phase is

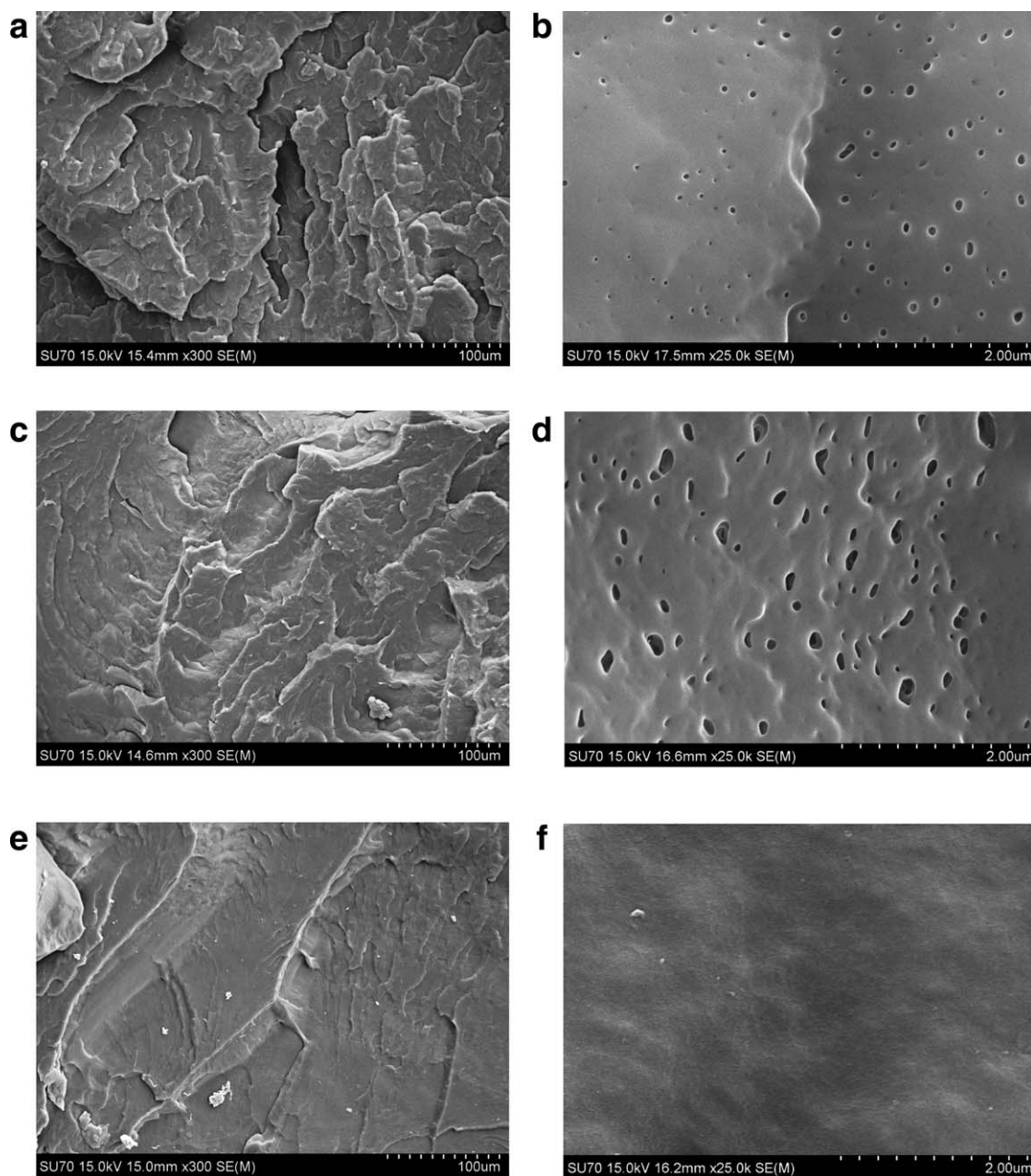


Figure 2. SEM micrographs of binary blends, etched with xylene (15 min, 50°C). (a) P₇₀V₃₀ blend, (b) P₇₀V₃₀ blend, (c) P₅₀V₅₀ blend, (d) P₅₀V₅₀ blend, (e) P₃₀V₇₀ blend, and (f) P₃₀V₇₀ blend.

similar or lower than the viscosity of the major phase.^{18,19,25–27} In this particular case, the similarity between the EPR and PP viscosities can be contributing to the resulting morphology. The morphology of the P₃₀V₇₀ binary blends [Figure 2(e,f)] indicates the existence of a single phase, due to the EPR high content and compatibility between the materials. These types of morphologies were already reported in other studies, suggesting a good compatibility between PP and EPR copolymers with propylene contents above 83%.^{20,21} This compatibility can be also explained by the capability of the EPR isotactic propylene sequences to be included in the PP crystal lattice, especially with high propylene content EPR materials.

Ternary Blends. The ternary blends morphology revealed some heterogeneity at the GTR particles interface (Figure 3). It is possible to observe the GTR particles completely or partially bounded to the PP matrix and the existence of fibrillar-type structures between the GTR particles and the matrix [Figure 3(b,d)]. This is an indication of compatibility between phases and may result from the EPR encapsulation of GTR and from the molecular entanglement between EPR and PP occurring at the interface layer.

The interface layer thickness may also contribute to the observed heterogeneity. A lower adhesion in regions with reduced thickness would lead to the observed voids and fibrils

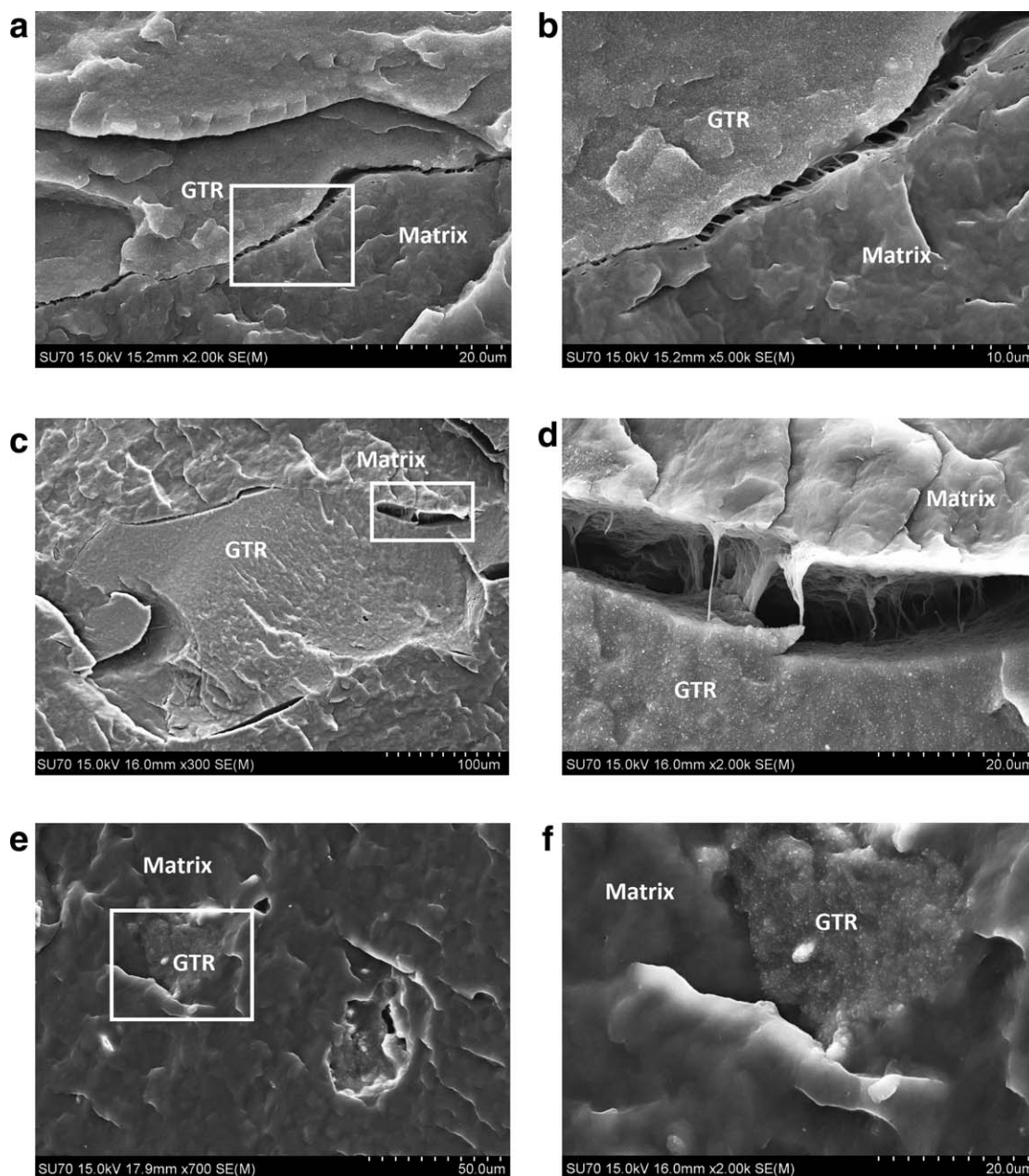


Figure 3. SEM micrographs of ternary blends, etched with xylene (15 min, 50°C). (a) $P_{70}(V_{0.7}G_{0.3})_{30}$, (b) $P_{70}(V_{0.7}G_{0.3})_{30}$ (left image magnification), (c) $P_{50}(V_{0.7}G_{0.3})_{50}$, (d) $P_{50}(V_{0.7}G_{0.3})_{50}$ (left image magnification), (e) $P_{30}(V_{0.7}G_{0.3})_{70}$, and (f) $P_{30}(V_{0.7}G_{0.3})_{70}$ (left image magnification).

along the interface. The existence of the fibrils may be explained by a slight dissolution effect of GTR by xylene, which we used to reveal the EPR domains.²⁸ This chemical attack leads to a morphological modification of the GTR particles size, pulling the surrounding material along the process and leading to the fibrils formation.

This effect seems also to be dependent of the GTR particle size and of the EPR blend content. This can be seen on the $P_{30}(V_{0.7}G_{0.3})_{70}$ blend [Figure 3(f)] where the smaller GTR particles are well embedded in the matrix, suggesting a complete encapsulation by EPR. This effect was also identified on a previous work by the same authors.¹⁷

Mechanical and Thermal Properties

The mechanical properties, the melting enthalpy, and the crystallinity of the formulated blends are listed in Table II.

The melting enthalpies were determined under the area of the endothermic peaks and are represented in Figure 4. The endothermic peak at around 165°C corresponds to the melting temperature of the PP material.

The TGA results revealed no thermal degradation around the injection molding temperatures. For the $P_{70}V_{30}$ blend the thermal degradation began around 300°C (Figure 5). Other authors have also obtained similar results for polyolefin blends with GTR.²⁹

Table II. Mechanical and Thermal Properties of the Blends

Blend	Tensile strength (MPa)	Elongation at break (%)	Hardness (shore D)	Young modulus (MPa)	Impact strength IZOD (kJ m ⁻²)	Melting enthalpy ΔH_m (J g ⁻¹)	Crystallinity (corrected) X_c^c (%)
P ₁₀₀	36.5 ± 0.4	79.7 ± 7.9	66.5 ± 0.6	428.9 ± 20.4	4.1 ± 0.3	98.1 ± 2.9	47.0 ± 1.8
P ₇₀ G ₃₀	18.8 ± 0.1	12.54 ± 0.2	62.1 ± 0.7	224.1 ± 2.2	2.9 ± 0.1	87.6 ± 2.5	59.9 ± 1.6
P ₇₀ V ₃₀	21.0 ± 0.4	257.6 ± 14.6	57.1 ± 0.2	170.1 ± 4.0	17.0 ± 1.1	66.3 ± 2.1	45.3 ± 1.4
P ₅₀ V ₅₀	13.2 ± 0.5	NB	44.7 ± 0.4	100.1 ± 0.2	64.7 ± 11.1	48.9 ± 1.8	46.8 ± 1.0
P ₃₀ V ₇₀	8.6 ± 0.6	NB	30.6 ± 4.3	43.7 ± 2.8	NB	32.3 ± 1.9	48.0 ± 0.9
P ₇₀ (V _{0.7} G _{0.3}) ₃₀	21.5 ± 0.1	55.3 ± 7.6	57.5 ± 0.7	182.3 ± 4.5	7.6 ± 0.8	69.5 ± 2.3	47.6 ± 1.1
P ₅₀ (V _{0.7} G _{0.3}) ₅₀	13.9 ± 0.2	129.3 ± 22.7	47.3 ± 0.9	91.8 ± 2.4	30.1 ± 1.7	56.0 ± 2.1	53.6 ± 1.3
P ₃₀ (V _{0.7} G _{0.3}) ₇₀	8.7 ± 0.2	827.4 ± 50.0	33.2 ± 1.4	43.9 ± 1.8	50.5 ± 7.1	35.1 ± 1.3	55.9 ± 1.4

NB = Nonbreak.

EPR and GTR Effects on the Binary Blends. The results show a significant increase on elongation at break and impact strength with the incorporation of EPR in PP-based blends. This behavior can be explained by the resulting morphology and elastomeric nature of EPR. On the P₇₀V₃₀ and P₅₀V₅₀ blends, the existence of very small EPR domains, below 0.1 μm , highly dispersed in a continuous thermoplastic phase and with smaller interparticle distances, can induce a predominantly shear yielding mechanism of failure, rather than crazing, enhancing the matrix toughness.^{30–32} This mechanism can be verified by the existence of a homogeneous fracture surface on a P₇₀V₃₀ blend specimen after the tensile test (Figure 6).

With increasing rubber content, the transition from a heterogeneous morphology to a homogeneous one, observed for the P₃₀V₇₀ blend, leads to a predominantly elastomeric behavior and consequently to an impact strength improvement.

The crystallinity of the blend may also contribute to the obtained results. In general, EPR dispersed in a PP matrix can act as nucleating agent, altering the crystalline size and morphology, and consequently the toughening mechanisms.³³ However, in the present work, this was not observed. The crystallinity results for EPR shows no significant effect but the GTR reveals a nucleating effect (Table II).

The effect of each rubbery material, EPR and GTR, on PP-based blends can be better understood analyzing the binary blends with 70% weight content of PP, P₇₀V₃₀, and P₇₀G₃₀. Taking 100% PP as the reference material, the results reveals a 223% increase on the elongation at break for the P₇₀V₃₀ blend and an 84% decrease for P₇₀G₃₀. Analyzing the blends impact strength behavior, P₇₀V₃₀ has a 312% increase and P₇₀G₃₀ a 29% decrease. These results reveal the GTR lack of compatibility with PP and its semirigid behavior.

The tensile strength and Young modulus decrease can be mostly explained by the reduction of the blends overall crystallinity with decreasing PP weight content. The softer nature of EPR leads to blends with the lowest hardness results.

Effects of the EPR Replacement by GTR. The ternary blends show slight changes on the tensile strength and Young modulus which agrees with the results obtained for the binary blends. The effect of EPR replacement by GTR is more pronounced on the material plastic deformation region. A 30% EPR replacement by GTR leads to a 79% decrease on elongation at break for P₇₀(V_{0.7}G_{0.3})₃₀ blends. No reference results were obtained for the other ternary blends due to the non-breaking of the P₅₀V₅₀ and P₃₀V₇₀ under the applied experimental conditions.

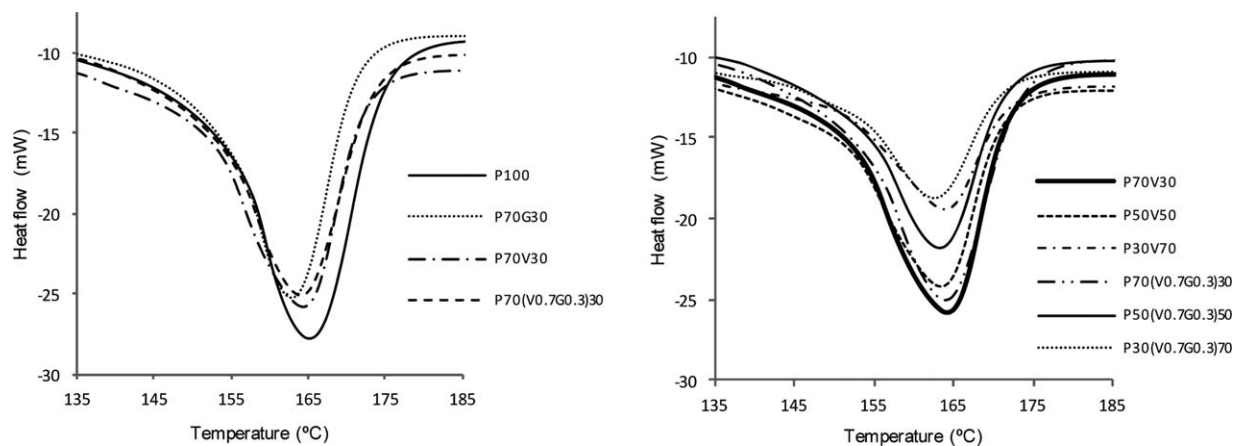


Figure 4. DSC curves of binary and ternary blends.

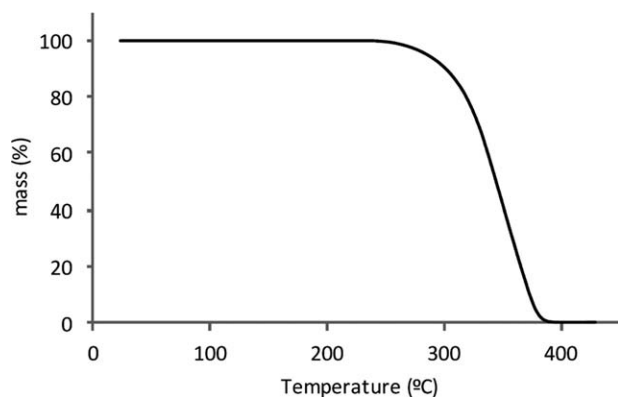


Figure 5. TGA curve of the $P_{70}V_{30}$ blend.

Hardness increases with the incorporation of GTR which is an expected result due to the vulcanized nature of this material. Results show that GTR increases the crystallinity of ternary blends, but with no significant influence on the tensile properties of the ternary blends.

The impact strength results are shown in Figure 7. A 30% EPR replacement by GTR, in the rubber phase, leads to a decrease in 56% on the impact strength for the $P_{70}(V_{0.7}G_{0.3})_{30}$ blend and a 54% decrease for the $P_{50}(V_{0.7}G_{0.3})_{50}$ blend. No reference data were obtained for the $P_{30}(V_{0.7}G_{0.3})_{70}$ blend due to the non-breaking of the $P_{30}V_{70}$ blend.

Effects of GTR Replacement by EPR. The replacement of 70% GTR by EPR on the $P_{70}G_{30}$ blend leads to a 162% increase in the impact strength, making EPR a viable material to be used on the formulation of ternary blends based on PP and GTR. These results can be explained mainly by toughness enhancement of PP but also by an encapsulating effect of GTR. The EPR-dispersed domains may be promoting a shear band effect on the PP matrix that is responsible for constraining the crazes that grow from the GTR surface. Encapsulation of GTR helps to reduce the stress concentration at the GTR–PP interfaces, leading to an improved impact behavior.³⁴

Effects of EPR and EPDM on TPE^{GTR} Blends. As stated before, the evaluation of different fresh rubber materials on the proper-

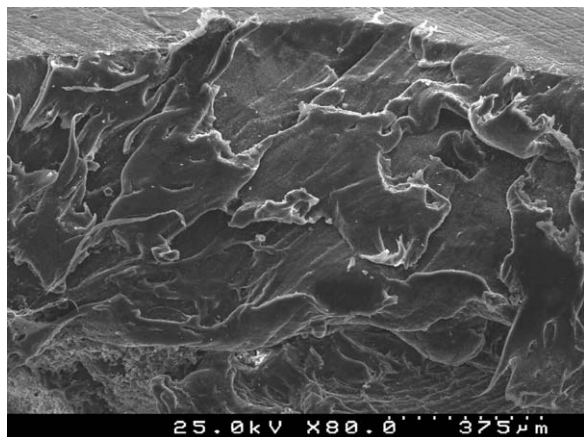


Figure 6. SEM micrograph of the $P_{70}V_{30}$ blend, after the tensile test.

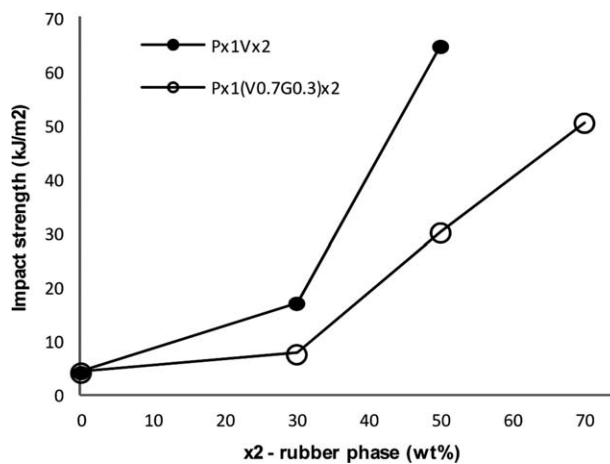


Figure 7. Impact strength of binary and ternary blends.

ties of TPE^{GTR} blends is part of the working strategy defined to obtain TPE^{GTR} materials with similar properties to a commercially available TPE material. On a previous work,¹⁷ an EPDM rubber material was analyzed, Buna EP G2470 from Lanxess, with 69% ethylene weight content (wt %), 4.2 wt % content of ethylidene norbornene (ENB), 0.86 g cm⁻³ density, and a MFI of 0.5 g 10 min⁻¹ (230°C, 2.16 kg). The different rubber materials effects on the mechanical properties of the TPE^{GTR} blends are mainly seen on the elongation at break and impact strength results. EPR reveals to have a stronger effect than EPDM on the binary and ternary blends (Figures 8 and 9).

The nature of rubber, the type of morphology, and the size and rubber domains dispersion in the matrix are the main reasons for the different behaviors of these blends.³⁵ On a continuous-disperse morphology, the existence of more finely dispersed EPR domains in the PP matrix than EPDM significantly contribute to the improved toughness behavior, either by crazing or by shear-yielding deformation mechanisms.^{27,36} This difference between EPR and EPDM can be also explained at a molecular level through the chemical affinity between EPR with high propylene content and the PP matrix. The partial incorporation of EPR molecules in the intercrystalline regions of PP spherulites

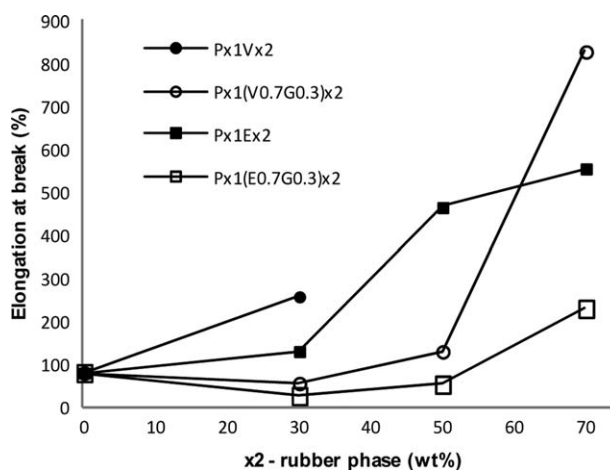


Figure 8. Effect of rubber weight content on the elongation at break of binary and ternary blends.

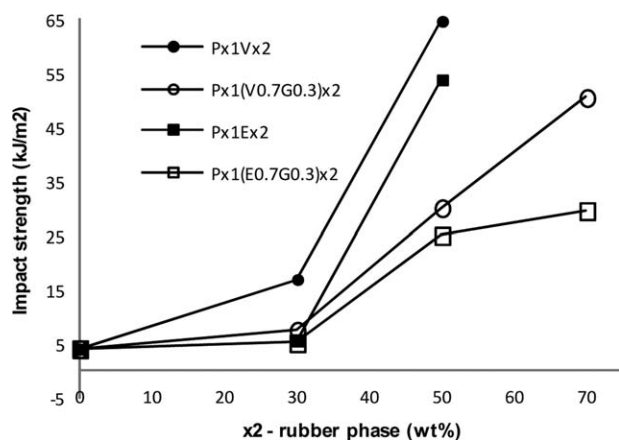


Figure 9. Effect of rubber weight content on the impact strength of binary and ternary blends.

and also in the amorphous regions³⁴ enable a more effective stress transfer between the materials, allowing EPR a higher deformation and an enhanced toughness behavior.

CONCLUSIONS

The ELT-recycling process can significantly benefit from a sustainable development of TPE^{GTR} materials.

The use of highly flowable materials in TPE^{GTR} blends enlarges the application field of GTR and can be an adequate strategy to upcycle this type of potentially waste material by injection molding technologies, even for thin and/or complex parts.

The mechanical properties of these blends are strongly dependent on the properties of the fresh rubber components, on the compatibility amongst the materials, on the resultant morphology (continuous disperse or cocontinuous) and on the dispersion and size of the rubber domains.

Highly flowable EPR, with high propylene content, induces morphological changes in a PP blend that leads to a significant improvement of the TPE^{GTR} toughness behavior.

The GTR particle size and the EPR content affect the level of the GTR encapsulation and must be taken in consideration for successful formulations of TPE^{GTR} blends.

Crystallinity results revealed that GTR influence the crystallization process of the TPE^{GTR} blends. This effect must be thoroughly analyzed for a better understanding of the blends mechanical behavior.

A rheological study is being considered for future work to assess the processability of these TPE^{GTR} blends.

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