Structure and Mechanical Properties of a Talc-Filled Polypropylene/Ethylene-Propylene-Diene Composite After Reprocessing in the Melt State

M. Sarrionandia,[1] A. Lopez-Arraiza,[1] J. Aurrekoetxea,[1] A. Arostegui[2]

[1]Mechanical and Industrial Production Department, Mondragon Unibertsitatea, Loramendi 4, 20500 Arrasate-Mondragón, Spain
[2]Department of Basic Sciences, Mondragon Unibertsitatea, Loramendi 4, 20500 Arrasate-Mondragón, Spain

ABSTRACT: A commercial talc-filled polypropylene/ ethylene-propylene-diene terpolymer composite was repeatedly injection molded up to five cycles to study the effects of reprocessing on the structure, morphology, and mechanical properties. Reprocessing did not change either the chemical structure or the thermal behavior of the composite, but led to a slight molecular weight reduction, due to shear–stress-induced chain scission. Talc and ethylene-propylene-diene terpolymer content remained unchanged with reprocessing, whereas size distribution slightly decreased. Young's modulus, yield stress, and Charpy impact strength of the composite did not significantly change even after five cycles, but deformation at break continuously decreased, and it was attributed to the slight molecular weight, talc, and rubber particle size reduction. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 114: 1195–1201, 2009

Key words: composites; recycling; degradation; structure; mechanical properties

INTRODUCTION

Polymeric and rubber waste recycling is a common practice in the plastics industry for both environmental and economic reasons, and therefore, recycling of polymers has received increased public attention in the past decades. The reuse of single-component scrap polymeric materials into new products with standard processing methods (primary recycling) and postconsumer waste (secondary recycling) have been, together with feedstock regeneration (tertiary recycling) and energy recovery by incineration (quaternary recycling), a method available for solution of the plastics waste problem.[1]

The application of successive high temperature or shear cycles during reprocessing can give rise to degradation of polymers. Degradation may appear in the form of either physical changes in the structure and/or chemical changes by chain scission, grafting, or crosslinking reactions, leading to a loss in appearance, chemical resistance, processability, and mechanical properties.[2]

One way to study the degradation process of polymers is by means of melt processing (extrusion or injection molding), where the quantitative effects of reprocessing on the properties can be determined. Furthermore, the property which is mainly affected by degradation, giving its decrease the maximum percent of reprocessed product to be added, and what is the nature of the degradation, determinating the selection of an appropriate stabilizer that can be added, can also be determined. Studies on the nature of degradation caused by reprocessing and its effects on the properties of commodity,[3,4] engineering,[5–8] and high-performance polymers,[9,10] and also in multicomponent materials or blends,[11–13] have been conducted.

The modification by rubber blending or filler addition is a well-known route to modify the properties of polymers. The incorporation of fillers is a cheap method to modify the properties of the base polymers, such as strength, stiffness, hardness, dimensional stability, and electrical and thermal conductivity;[14–16] however, the rubber addition enhances its notch sensitivity and impact toughness.[17,18] Thus, polypropylene (PP) composites with both fillers and rubber have been developed with the aim of generating a new material with a good balance of stiffness and toughness properties. In this way, thermal,[19–22] morphological,[21–23] and mechanical[21,22] properties of different PP/rubber blends filled with talc have been studied, using rubbers like ethylene-propylene,[19,20,23] poly(styrene-b-ethylene-*co*-

butylene-co-styrene) triblock copolymer,[21] and ethylene-propylene-diene (EPDM).[22] However, to our knowledge, the effect of thermomechanically induced degradation by means of successive reprocessing cycles has not been performed in these type of composites.

In this work, the effects of reprocessing by repeated injection molding on the structure and mechanical properties of talc-filled PP/EPDM composites were studied. The possible changes in the chemical structure, molecular weight, physical structure, and morphology were tested by Fourier Transform infrared spectroscopy (FTIR), melt flow index (MFI), differential scanning calorimetry (DSC), and scanning electron microscopy (SEM), respectively. The effects of structural changes of talc-filled PP/EPDM composites on the mechanical properties were determined by means of tensile and Charpy impact tests.

EXPERIMENTAL

Materials

The talc-filled PP/EPDM composite used in this work (M 1685L 5001, containing 13 wt % talc and 10 wt % EPDM) was kindly supplied by Galloplast S.L. Talc content was measured after ashing samples in a convection oven at 700°C for 2 h. An etching process with xylene at 50°C for 7 days was used to measure the EPDM content of the composites. The samples were weighed before and after the etching, and the weight obtained was related with EPDM extraction, and in consequence, with its content.

Sample preparation

The successive injection molding cycles were performed in a Battenfeld BA600CDC reciprocating screw injection molding machine, at a barrel and mold temperature of 270°C and 25°C, respectively. A screw speed of 200 rpm during plasticization and an injection pressure of 530 bar were used. These high-injection temperatures and barrel speed have been selected as processing parameters to increase the severity of the reprocessing effect. The mold provided tensile specimens (thickness 3.2 mm) and impact (thickness 6.3 mm). Then, the specimens were granulated in a knife mill (EQUIPER granulator, type ML 14/30), providing pellets with a mean size of 2–3 mm, for the next reprocessing step. This procedure was repeated up to five times.

The talc-filled PP/EPDM samples are designated PP/EPDM/talc-1 to PP/EPDM/talc-5, where the number indicates the reprocessing cycle number. For example, PP/EPDM/talc-4 indicates that the composite is reprocessed four consecutive times.

Testing methods

FTIR analysis was performed in a NICOLET MAGNA 850 with a resolution of 8 cm^{-1}. For each measurement, 50 scans were performed. MFIs, and in consequence, the melt viscosity, were measured with a CEAST 6540/011 apparatus at a melting temperature of 230°C and with a 2.16-kg load, according to ISO 1133. A minimum of three measurements were carried out for each reported value. For both analyses, the samples were obtained from the molded tensile specimens.

The DSC analysis was performed with a Perkin-Elmer DSC-7 differential scanning calorimeter. Firstly, the samples were heated from 30°C to 230°C at 10°C/min to avoid the thermo-mechanical history. Then, the samples were cooled to 30°C at 10°C/min, and finally, a second heating scan was performed. The melting temperature (T_m) and the melting heat were determined in both heating scans. The degree of crystallinity (x_c) of the samples was determined by means of the ratio between the melting enthalpy (the area between the measured curve and the baseline in the region of the peak) and the melting enthalpy of a completely crystalline isotactic PP (taken to be 207.1 J/g).[24]

The analysis of the composite morphology was performed by scanning electron microscopy (SEM) after fracturing tensile specimens in liquid nitrogen. The cryofractured surfaces were etched with xylene at 50°C for 7 days to dissolve out the elastomeric domains from the fractured surfaces. Then, the samples were dried and observed by SEM. A JEOL-JSM 5600LV electron microscope was used at an accelerating voltage of 20 kV. Direct observation of the fracture surfaces was performed under low-vacuum conditions (20–25 Pa).

Tensile tests (ASTM D-638-97) were performed using an Instron 4206 tensile tester at 23 ± 2°C and with a load cell of 5 kN. The mechanical properties (Young's modulus, yield stress, break stress, and deformation at break) were determined from the load–displacement curves. For Young's modulus, calculation strains have been measured by an extensometer at a crosshead speed of 1 mm/min; whereas, the yield stress and break properties were determined without an extensometer at a crosshead speed of 10 mm/min. At least five specimens were tested for each reported value.

The Charpy impact tests (ASTM D-6110) were performed on notched specimens using a CEAST 6545/000 pendulum at 23 ± 2°C. The notch (depth 3.1 mm and radius 0.25 mm) was machined after
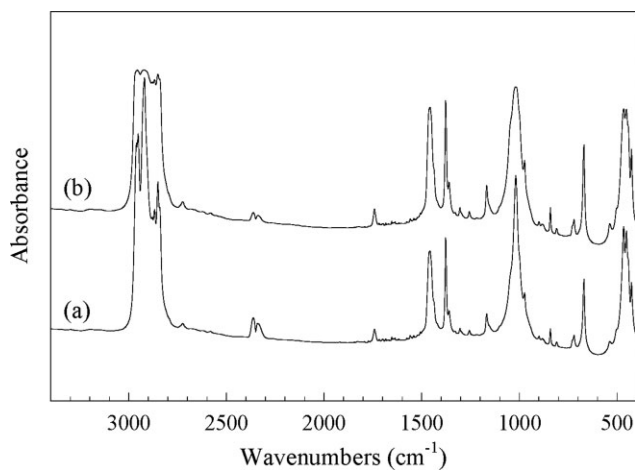
Figure 1 FTIR spectra of PP/EPDM/talc-1 (a) and PP/EPDM/talc-5 (b) composites. To aid clarity, the curves are shifted on the vertical axis.

injection molding. A minimum of eight specimens were tested for each reported value.

RESULTS AND DISCUSSION

Chemical and physical structure

The possible changes due to recycling in the chemical structure of PP/EPDM/talc composites have been studied by FTIR, and the corresponding results for PP/EPDM/talc-1 (a) and PP/EPDM/talc-5 (b) are shown in Figure 1. The rest spectra for other cycles was not shown because of their similarity to that shown in Figure 1. The absorption bands at 2950 (C—H stretching), 1460 (CH$_2$ bending), 1380 (side CH$_3$), and 1200–900 cm^{-1} are attributed to isotactic PP,[25] whereas the absorption bands at 2925 and 2580 (C—H stretching), 1630 (C=C stretching), 1460 and 720 (CH$_2$ bending), and 1375 cm^{-1} (CH$_3$ bending) are attributed to EPDM.[26] As it can be observed in Figure 1, after reprocessing up to five consecutive cycles, the most characteristic bands of PP and EPDM remained at the same wave numbers and with similar peak height ratios. These results indicate that possible changes of the chemical structure of PP/EPDM/talc composites, as they are not detectable by FTIR, if existing, would be very small.

When stabilized PP is subjected to high temperatures and shear–stress, chemical structure did not change,[3] whereas, when unstabilized PP was used oxidative reaction of tertiary carbons occurred in the presence of oxygen, leading to double carbon–carbon bonds and ester groups.[27,28] Therefore, it can be argued that the PP matrix of PP/EPDM/talc composites has some stabilizers against degradation, and, in consequence, the chemical structure of the composites did not change based on the experimental conditions used. These results agreed with those

observed during the recycling of different commercial polymers.[5,7,8,29]

Thermal, mechanical and/or oxidative degradation could have a great effect on the molecular weight of polymers. In this work, the effects of degradation on molecular weight of reprocessed PP/EPDM/talc composites were tested by MFI measurements. As it is known, the MFI allows an indirect analysis of the effects of degradation on viscosity, and indirectly, on the molecular weight. For this reason, the MFI of PP/EPDM/talc composites was measured after each reprocessing cycle, and the corresponding results are shown in Table I. As it can be observed, the MFI slightly increased with the number of cycles, from approximately 10.6 g/10 min for PP/EPDM/talc-1 up to 12.5 g/10 min for PP/EPDM/talc-5. Therefore, this slight MFI increase indicates that the molecular weight of the composite decreases slightly.

When neat PP[3,28] and talc-filled PP[13] were subjected to several reprocessing cycles, it was observed that the MFI values increased at least up to three times its original value, which is attributed to a high molecular weight decrease. The fact that the MFI of PP/EPDM/talc composites did not increase so much can be because of the presence of EPDM, probably hindering the molecular weight decrease of the matrix.

Degradation by chain scission could affect the molecular mobility of the whole polymeric chain or a part of it, and consequently the melting/crystallization could also be modified. For this reason, the effects of reprocessing on the physical structure of PP/EPDM/talc composites were studied by DSC. Figure 2 shows the heating thermograms of PP/EPDM/talc-1 (a) and PP/EPDM/talc-5 (b) composites. The results of the remaining reprocessing cycles are not plotted because of similarity to those of Figure 2. The melting temperature (T_m), the melting temperature range (ΔT), and the crystallinity (x_c) are reported in Table II. Heating DSC curves show always a single peak around 167°C, indicating that all the samples have α-form structure.[30] Similar behavior, i.e., a constancy of T_m, has been seen during the reprocessing of different semicrystalline polymers, such as PP,[3] poly(butylene terephthalate),[5]

TABLE I
MFI Values of reprocessed PP/EPDM/talc Composites

Reprocessed composite	MFI (g/10 min)
PP/EPDM/talc-1	10.6 ± 0.5
PP/EPDM/talc-2	11.6 ± 0.5
PP/EPDM/talc-3	11.3 ± 0.7
PP/EPDM/talc-4	11.7 ± 0.4
PP/EPDM/talc-5	12.5 ± 0.7
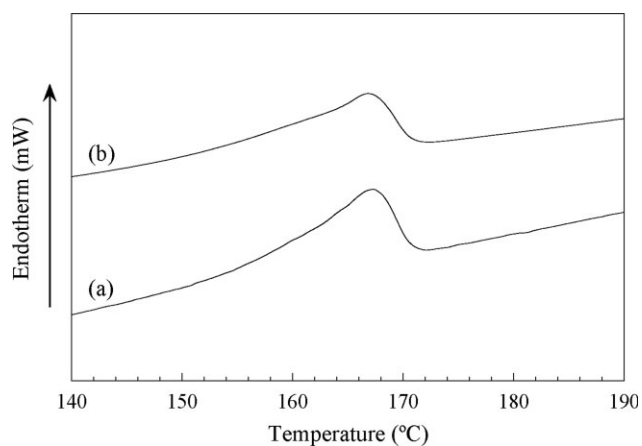
Figure 2 Second DSC heating scans of PP/EPDM/talc-1 (a) and PP/EPDM/talc-5 (b) composites. To aid clarity, the curves are shifted on the vertical axis.

poly(trimethylene terephthalate),[7] nylon-6,[8] and talc-filled PP.[13]

Even if the melting temperature is defined as the temperature at the peak, melting of semicrystalline polymers takes place on a broad temperature range, which is because of the lamellar thickness distribution.[30] The broader melting temperature of the recycled materials is probably because of their thicker lamellae, which is probably because of a consequence of the slightly higher molecular mobility.

The study of the crystalline content of semicrystalline polymers is of great importance in understanding the relationship between the structure and the mechanical properties. As it can also be observed in Table II, the x_c values of the composites remained practically constant with reprocessing, because the maximum change and the estimated deviation of the measurement (approximately 2–3%) were not very different. This result is in agreement with the lack of molecular weight variation observed by means of MFI results.

Morphological structure

The study of the morphology of composites reinforced with planar fillers and rubber-dispersed particles is of great interest because a decrease in the

TABLE II
Melting Temperature (T_m), Melting Temperature Range (ΔT) and Crystalline (x_c) Values of Reprocessed PP/EPDM/talc Composites

Reprocessed composite	T_m (°C)	ΔT (°C)	x_c (%)
PP/EPDM/talc-1	166.4 ± 1.3	9.0 ± 0.9	28 ± 2
PP/EPDM/talc-2	167.2 ± 1.8	12.8 ± 1.1	27 ± 2
PP/EPDM/talc-3	168.1 ± 1.7	14.4 ± 1.2	27 ± 2
PP/EPDM/talc-4	167.6 ± 1.4	13.6 ± 0.9	29 ± 2
PP/EPDM/talc-5	166.5 ± 1.9	14.2 ± 1.2	30 ± 1
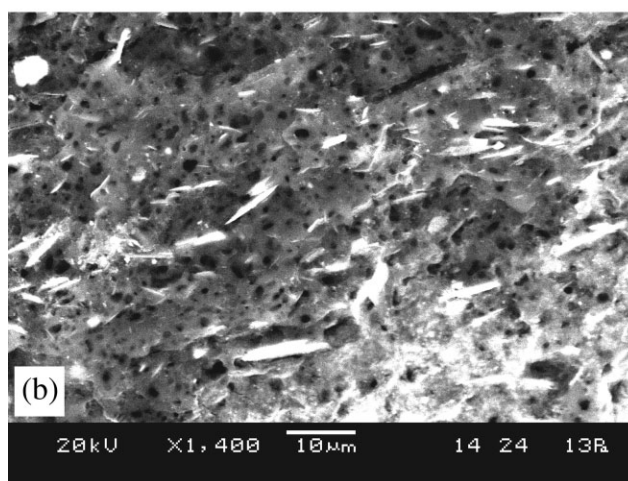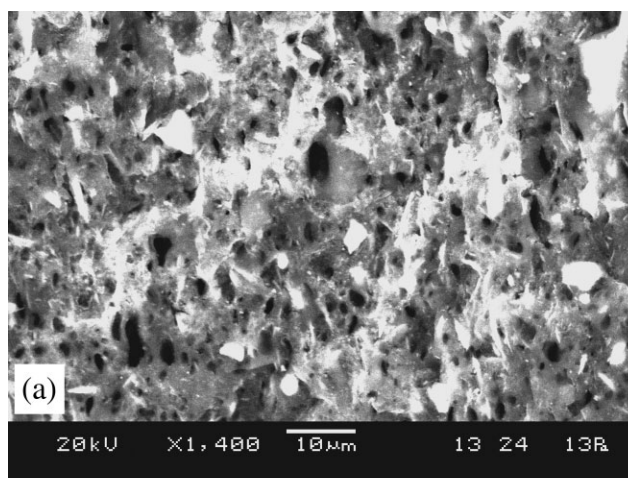
Figure 3 Cryofractured surfaces of the inner zone of injection-molded tensile specimens for PP/EPDM/talc-1 (a) and PP/EPDM/talc-5 (b) composites.

flake aspect ratio and/or particle size might give to a modification in the mechanical properties. For this reason, the possible effects of consecutive reprocessing cycles on the structure of talc-filled PP/EPDM composites were studied by SEM. Figure 3 shows the cryofractured surfaces of the inner zone of injection-molded tensile specimens of PP/EPDM/talc-1 (a) and PP/EPDM/talc-5 (b) composites. The morphology of PP/EPDM/talc-2, PP/EPDM/talc-3, and PP/EPDM/talc-4 composites is not shown because it was intermediate to that shown in Figure 3(a,b).

As it can be observed in Figure 3, the talc dispersion in PP/EPDM matrix was good whatsoever the number of reprocessing cycle. Furthermore, no large void presence was seen between the talc and the matrix, but the surfaces of the talc particles were quite clear without residual matrix on their surface. Thus, the adhesion appeared to be poor. When Figure 3(a) and Figure 3(b) are compared, it can be observed that there was a slight decrease in the size of the talc particles from the first up to the fifth reprocessing cycle. This is because the morphology

Reprocessed composite	E (GPa)	σ_y (MPa)
PP/EPDM/talc-1	1.2 ± 0.1	16.8 ± 0.4
PP/EPDM/talc-2	1.3 ± 0.1	15.6 ± 0.8
PP/EPDM/talc-3	1.3 ± 0.1	15.5 ± 0.4
PP/EPDM/talc-4	1.3 ± 0.1	15.5 ± 0.2
PP/EPDM/talc-5	1.2 ± 0.1	15.5 ± 0.1

of PP/EPDM/talc-5 showed the appearance of small particles in a greater proportion than that in PP/EPDM/talc-1.

Concerning the rubber particles distribution, as it can also be observed in Figure 3, the EPDM particles were finely dispersed in the matrix, and their sections had a more or less spherical shape; this behavior being independent of reprocessing cycle. However, it seems that the EPDM had more tendency towards the formation of agglomerates in the first cycle than in the fifth cycle. This can be explained by the fact that the composite was subjected to high shear–stress cycles for an increased period of time, decreasing the possibility to form agglomerates. As a consequence, the EPDM size slightly decreased with reprocessing.

Mechanical properties

Whitening of the specimens was observed during the tensile testing, irrespective of the processing number, which is because of the presence of a large amount of micro-voids and a highly fibrillar morphology[3] and the strain-induced crystallization.[7] This fact was corroborated by means of DSC analysis after the tensile tests. The obtained x_c values (approximately 37%) were independent of the reprocessing number cycle and were clearly higher than that before tensile testing (Table II). This indicates that the crystalline content increased as a result of drawing and that strain-induced crystallization took place.

The low-strain tensile test properties of PP/EPDM/talc composites as a function of reprocessing cycles are shown in Table III. As it can be seen, the Young's modulus remained constant with reprocessing cycles at a mean value of 1.2–1.3 GPa. This modulus constancy is in agreement with the lack of variation in crystalline and talc content observed before. The slight talc distribution variation seen in Figure 3 had no effect in the modulus values, as it took place during the reprocessing of PP/talc composites.[13] The lack of variation of Young's modulus is the usual behavior when neat thermoplastic polymers,[5–8] polymeric blends,[9] or filled polymers[13] are subjected to reprocessing in the melt state. However,

in some cases such as PP[3] and high density polyethylene,[4] the Young's modulus increased as a consequence of the crystalline content increase induced by molecular weight decrease.

As it can also be observed in Table III, the yield stress of PP/EPDM/talc composites slightly decreased from approximately 16.8 MPa for PP/EPDM/talc-1 composite to 15.6 MPa for PP/EPDM/talc-2 composite, remaining constant for further reprocessing cycles. The small decrease after the first reprocessing cycle can be related with the slight molecular weight and talc particle size reduction that took place after the first reprocessing cycle. Although, similar results have been observed in the reprocessing of other thermoplastic polymers,[4,8,31] it usually remained practically constant with reprocessing.[5–7,11,13]

Deformation at break of PP/EPDM/talc composites is shown in Figure 4 as a function of reprocessing cycles. As it can be observed, the deformation at break slightly decreased with reprocessing up to the third cycle, and then it maintained almost constant at a mean value of 80%. However, as all the specimens yielded, PP/EPDM/talc remained ductile even after harsh reprocessing.

The behavior showed by deformation at break of PP/EPDM/talc composites can be related with molecular weight and/or secondary phase size reduction. Firstly, as it was seen by MFI measurements, the molecular weight of the composites slightly decreased with reprocessing, and this behavior has been previously observed in many thermoplastics.[4–9] Secondly, the slight size reduction of talc particles seen by SEM, as reprocessing became harsher, could be responsible for the decrease in the deformation at break, which took place during the reprocessing of talc-filled PP composites.[13] Finally, the decrease of EPDM particle size responsible for the deformation at break behavior can be discarded because a greater
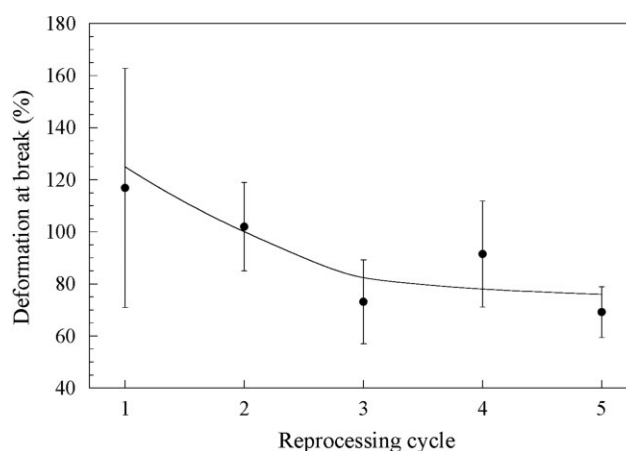
Figure 4 Deformation at break of PP/EPDM/talc composites as a function of reprocessing cycles.
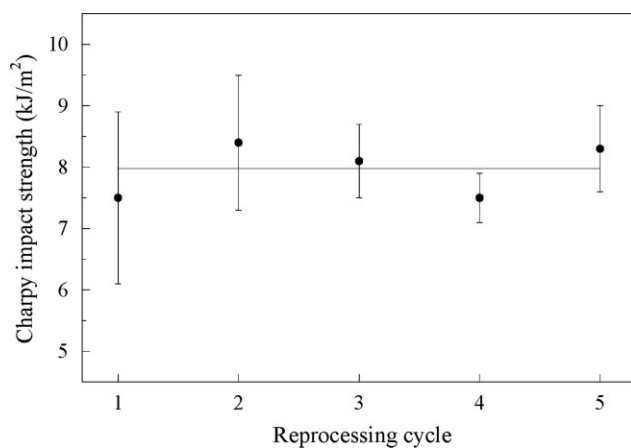
Figure 5 Charpy impact test data of recycled PP/EPDM/talc composites as a function of reprocessing cycles.

decrease is needed to observe reductions in break properties.

The break stress was almost constant (approximately 13 MPa) with reprocessing. This was because of the fact that specimens broke in the flat cold-drawing region of the tensile curves, and consequently the break stress was practically unaffected by changes in ductility.

The energy at break of PP/EPDM/talc composites was measured by means of notched impact tests. Figure 5 shows the Charpy impact strength evolution with the number of reprocessing cycles. As it can be observed, the impact strength of PP/EPDM/talc composites remained almost constant (approximately at 8 kJ/m^2) with the number of reprocessing cycles. Notch-sensitive polymers such as PP,[3] poly(butylene terephthalate),[5] poly(amino ether) resin,[6] and nylon-6[8] are subjected to successive reprocessing cycles and the impact strength decreased dramatically. However, in this work, as the composite has rubbery-dispersed particles, its notch sensitivity was less than that in neat PP, and in consequence, the impact strength remained unchanged.

Furthermore, as it is known, the rubbery-dispersed phase has a great effect on the impact strength behavior of materials, and parameters such as rubber particle diameter[32,33] and interparticle distance[34] are usually defined. When different impact strength values at a given volume fraction are compared, the rubber particle size can be used, obtaining good correlations between morphology and high-strain rates tests, i.e., a dramatic decrease in impact strength when the rubber particle size is less than a critical value. In the composites studied in this work, as the impact strength variation was negligible, it can be concluded that on the harsh conditions used in the melt state, and despite the slight EPDM size decrease observed by SEM, its size remained within the limits.

CONCLUSIONS

The effects of thermo-mechanical-induced degradation, by means of successive injection-molding cycles, on the structure and mechanical properties of talc-filled PP/EDPM composite have been investigated. Even if the reprocessing conditions used are extremely harsh (high temperature and high shear-stress), the absence of changes in the chemical structure and the slight increase of the MFI suggest that chain scission is the only degradation mechanism under the experimental conditions for the composites. This small molecular weight reduction is not enough to modify the crystallinity of PP in the composites, but slightly thicker lamellar structures are present in reprocessed composites. SEM micrograph analysis has shown that reprocessing induces a small reduction of the talc and EPDM particle size. Stiffness and impact strength are not affected by these microstructural modifications, but yield stress and deformation at break are slightly reduced.

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