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# Effect of Reprocessing Cycles on the Degradation of Polypropylene Copolymer Filled with Talc or Montmorillonite During Injection Molding Process.

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**Abstract.** Mechanical recycling of polymeric materials is a favorable technique resulting in economic and environmental benefits, especially in the case of polymers with a high production volume as the polypropylene copolymer (PP). However, recycling by reprocessing techniques can lead to thermal, mechanical or thermo-oxidative degradation that can affect the structure of the polymer and subsequently the material properties. PP filled with montmorillonite (MMT) or talc are widely produced and studied, however, its degradation reactions by reprocessing cycles are poorly studied so far. In this study, the effects of reprocessing cycles in the structure and in the properties of the PP/MMT and PP/Talc were evaluated. The samples were mixed with 5% talc or MMT Cloisite C15A in a twin-screw extrusion. After extrusion, this filled material was submitted to five reprocessing cycles through an injection molding process. In order to evaluate the changes induced by reprocessing techniques, the samples were characterized by DSC, FT-IR, Izod impact and tensile strength tests. The study showed that Young modulus, elongation at brake and Izod impact were not affected by reprocessing cycles, except when using talc. In this case, the elongation at brake reduced until the fourth cycle, showing rigidity increase. The DSC results showed that melting and crystallization temperature were not affected. A comparison of FT-IR spectra of the reprocessed indicated that in both samples, between the first and the fifth cycle, no noticeable change has occurred. Thus, there is no evidence of thermo oxidative degradation. In general, these results suggest that PP reprocessing cycles using MMT or talc does not change the material properties until the fifth cycle.

**Keywords:** Polypropylene copolymer, Reprocessing Cycles, Mechanical Recycling
**PACS: 81.05.Qk**

## INTRODUCTION

Polypropylene (PP) is one of the most versatile commodity polymers because it possesses exceptional properties, including excellent chemical and moisture resistance, good ductility and stiffness and low density [1-3]. Also, PP is very easy to process and versatile in use and application in the polymer industry [3]. The mechanical recycling of PP is routinely done by milling post-consumer goods followed by extrusion and pelletization. This solution does not require modification of the polymer but, after several cycles, the properties of the recycled polymer will deteriorate and applications with lower requirements need to be found [4]. The repeated reprocessing casings influence the physical and chemical properties of the polymer. In general, multiple processing of PP causes molecular chains to undergo chain scissions due to mechanical stresses and the formation of free radicals during the autooxidation step of melting [2, 5]. Scission of the larger molecular chains increases the number of shorter chains of the respective polymer, which would lead to fewer entanglements that can decrease mechanical properties [5]. While the degradation mechanism of PP homopolymer has been well described, information on the degradation of PP copolymer is almost missing. PP copolymer has the typical heterogeneous structure, consisting of PP homopolymer, ethylene-propylene rubber and a relative small portion of PE homopolymer phases [2, 5]. The degradation process induced by reprocessing of injection molding are thus more complicated and influenced by the presence of the ethylene units, the quantity and distribution of which substantially influences the overall behaviour of the material. Investigation of the degradation process of PP copolymer during five reprocessing cycles is the objective of this work.

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## EXPERIMENTAL

### *Materials*

Commercial Polypropylene copolymer with a melt flow index (MFI (230 °C/2.16 kg) of 6 g/10 min and density 0.905 g/cc (23 °C) was obtained from Braskem S.A. The talc powder, referenced Talmag GM-20, purchased from Magnesita S.A. Density of 0,75 g/cm³ and superficial area of 5 $m^2.g^{-1}$ and Cloisite C15A obtained from Southern Clay Products. The amount of talc or C15A was fixed at 5wt%. The mixture between PP and filler was done using twin screw extrusion process.

### *Degradation study*

A sufficient amount of virgin polypropylene copolymer was subjected to five cycles of reprocessing by injection molding using a Battenfeld injection molding machine. The pieces were reground and enough sample was taken for analysis and the molding of impact, tensile test pieces, DSC and FT-IR. The rest of the sample was then subjected to further recycling by injection molding and regrinding.

### *Characterization*

Samples for testing were prepared in a Battenfeld injection molding according to ASTM D 4101. Tensile testing according ASTM D638, specimen type I, was carried out on a universal tensile machine EMIC DL 10000 at speed 50 mm/min. Impact Izod test was carried out at 23°C using a pendulum-type impact tester (Ceast, Resilimpact), according to ASTM D 256. Thermal behavior was studied by differential scanning calorimetry (DSC) using a 2100 TA Instruments, where linear heating and cooling experiments were obtained at 10°C/min under a constant flow of nitrogen and calibrated by an indium standard. The measurements were made in the second heating and cooling cycle. The degree of crystallinity was determined using $\Delta H_m^o$ 190 J/g for PP [6, 7]. The FT-IR spectrum was obtained in a PerkinElmer Spectrum 100, with a resolution of 4$cm^{-1}$ and 16 scans in the region between 4000 and 600 cm-1. The samples were obtained from the injection molded specimens.

## RESULTS AND DISCUSSION

### *Mechanical properties*

The influence of reprocessing cycles on mechanical properties of the PP filled samples is shown in Fig. 1. The results showed that the Young modulus and impact Izod remained relatively unchanged over the five reprocessing steps. When use talc a slight increase of stiffness and the deformation at break values decreased until fourth cycle. A little increase of the value occur in the fifth cycle showing a brittle effect of the matrix. In this case, the increase of reprocessing cycles of PP results in breakage of its chains [2, 8]. Smaller chains can act as lubricants between them and thus increase the elongation at break, as was seen with greater emphasis in the reprocessing cycle.

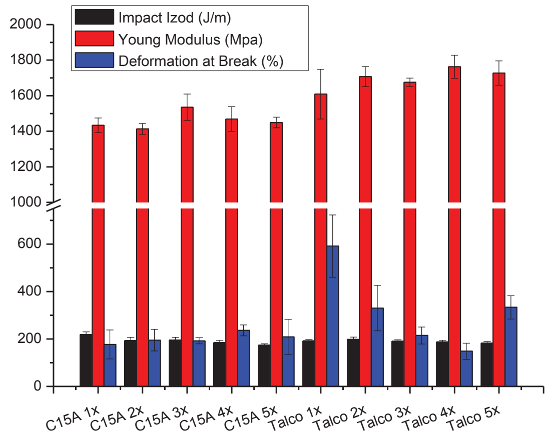


**FIGURE 1**. Mechanical properties of reprocessing PP.

*Thermal Properties*

The $T_m$, $T_c$ and $X_c$ results are show in Table 1. The values obtained for $T_m$ and $T_c$ and $X_c$ are constant.

**TABLE 1.** DSC results for PP/Filler reprocessing cycles

| Sample | $T_c$ (°C) | $T_m$ (°C) | $X_c$ (%) |
|---|---|---|---|
| PP C15A 1x | 119 | 164 | 45 |
| PP C15A 2x | 118 | 165 | 39 |
| PP C15A 3x | 118 | 166 | 38 |
| PP C15A 4x | 119 | 164 | 37 |
| PP C15A 5x | 118 | 165 | 38 |
| PP TALC 1x | 125 | 167 | 39 |
| PP TALC 2x | 126 | 166 | 39 |
| PP TALC 3x | 127 | 166 | 42 |
| PP TALC 4x | 126 | 167 | 39 |
| PP TALC 5x | 127 | 166 | 39 |

*FTIR analysis*

The results of FT-IR (Fig. 2) showed no degradation of the PP by multiple reprocessing cycles. There is no formation of carbonyl groups in the region at 1750 $cm^{-1}$ [9] attributed this effect to a blocking effect of the filler, which prevents macroradicals from reacting with oxygen [9]. In thermoxidation reaction the oxygen diffusivity plays an important role and is responsible for the degradative extension. The range between 1000 – 1100 $cm^{-1}$ where some differences as the appearance of the Si-O stretching vibration characteristic of the organoclay and talc [4]. These levels of degradation did not significantly affect the properties of the material.

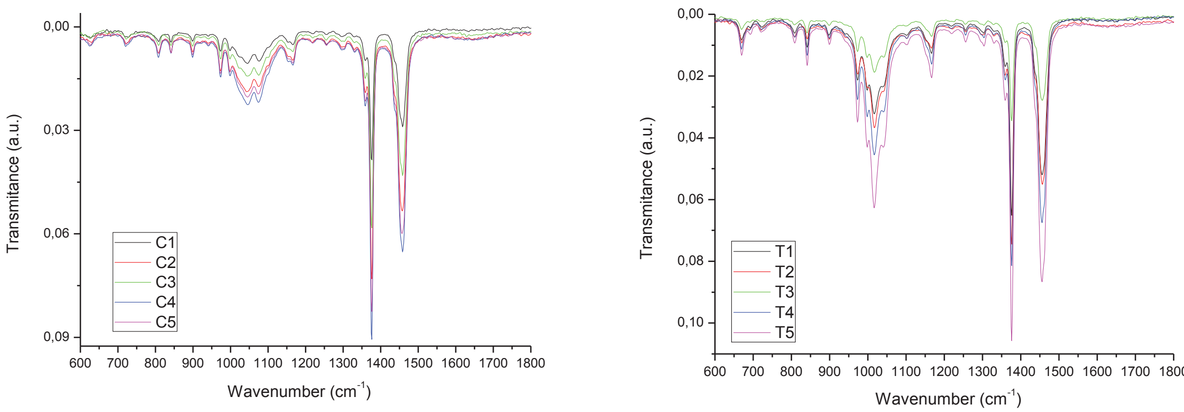


**FIGURE 2**. FTIR results for reprocessing PP.

## CONCLUSIONS

The reprocessing cycles of PP copolymer does not affect significantly the mechanical properties. The thermal stability shows a slight decrease over the reprocessing steps. A comparison of FT-IR spectra indicated changes only in the fifth reprocessing step. Thus, the results demonstrated the feasibility of reuse of PP in commodities applications.

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