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## A quantitative assessment of variability in impact polypropylene mechanical recycling

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**Abstract.** Plastic recycling has become an environmental and economic necessity. Mechanical recycling by extrusion and injection molding is the preferred method for thermoplastics recycling, owing to its energy efficiency and capacity to process large quantities of plastic solid waste at an industrial scale. For a sustainable plastics economy, it is crucial that mechanical recycling produces high-performance products, even from waste of variable quality. In this context we evaluated the performance of a recycled Impact copolymer polypropylene (PPc), considering a wide range of properties such as dimensions, chemical composition, morphology, melt viscosity, and appearance like color, and mass, as well as mechanical properties such as flexural strength and Charpy Impact resistance. This study highlights that reprocessing leads to changes in color, including a gradual shift from white to light yellow with successive injection molding cycles. A reduction in bulk density is also observed during the transition from pellets to reground flakes. Furthermore, significant chain scission in PP becomes evident from the first reprocessing cycle, reflected in increasing melt flow index (MFI) values. However, the recycling process does not alter the weight of the specimens molded.

### Introduction

Global demand for plastics is steadily increasing, with the volume in circulation in Europe expected to rise from 236 million tons to 417 million tons per year by 2030 [1]. Plastics have become indispensable across various sectors especially in automotive, construction, packaging, consumer goods, healthcare, and renewable energy. However, this widespread use comes with significant environmental challenges, particularly concerning plastic waste management, especially for polyolefins like polypropylene (PP) and polyethylene (PE). Indeed, the polyolefins represent nearly half of the European polymer demand. With growing regulatory pressure, polymer recycling is a way to reduce environmental problems caused by polymeric waste accumulation generated from every day applications of polymer materials such as packaging. Among all the recycling techniques available, mechanical recycling is the most known and developed technique in industries [2]. According to studies conducted by Plastics Europe, plastics production via mechanical recycling has reached 10% in 2022. Mechanical recycling of polyolefins plays a vital role in minimizing environmental pollution and preserving fossil fuel resources [3].

In the case of recycled polyolefins, several previous studies [4], [5] have found that PP suffer degradation during processing by chain scission, reducing molecular weight by cutting down the longest chain. This results in a large increase in fluidity as measured by MFI [6].

For instance, Bahlouli et al. [7] and Incarnato et al. [8] demonstrated through their analysis that the thermomechanical recycling process significantly influenced the rheological and mechanical properties. Notably, this process caused a reduction in molecular weight which involves a decrease

in failure stress, and a decrease of the impact strength. Additionally, Scanning Electronic Microscopy (SEM) pictures showed a modification of the fracture process (cavitation) due to the embrittlement of the amorphous matrix by the chain scission mechanism.

Furthermore, Jubinville et al. [9] also found that successive thermo-mechanical processing led to significant chain scission in PP, as evidenced again by the reduction in melt viscosity and average molecular weight. Nevertheless, despite these molecular changes, other properties, such as tensile strength and modulus, remained largely unaffected.

Moreover, Impact polypropylene copolymer (PPc) has a heterophasic morphology, consisting of a PP homopolymer matrix, an ethylene-propylene rubber (EPR) nodules dispersion and a relatively small portion of PE homopolymer phase, this is intended to improve impact strength especially at lower temperature. J. Tocháček et al. [10] found that the degradation processes for these types of material induced by extrusion are thus more complicated and influenced by the presence of the ethylene units, the quantity and distribution of which substantially influences the overall behavior of the material.

A previous study [11] showed that PPc can be recycled up to five times before significant degradation of its mechanical and physical properties occurs. However, its mechanical properties, including tensile strength, flexural modulus, and Izod impact strength, decrease with increasing injection cycles. The Izod impact strength was highly affected by multiple injections, as the morphology and dispersion of the EPR phase in PPc are key to withstanding external stress. A smaller average distance between EPR nodules improves toughness, while larger nodules and increased spacing reduce impact strength. An increased viscosity ratio caused EPR agglomeration and poor dispersion, significantly lowering impact strength in all PPc, showed the greatest variation in impact strength due to notable changes in EPR nodules size after five injections [12].

This paper focuses on analyzing the properties of recycled polypropylene Impact copolymer to evaluate the effects of degradation due to multiple reprocessing cycles by injection molding, through comprehensive rheological, mechanical, and morphological analyses. Additionally, SEM analyses were conducted to link and validate the experimental results observed at the macroscale with detailed microstructural observations. This study aims to deepen the understanding of the recycling process and optimize it to produce recycled materials with properties tailored for structural applications, while also investigating the effects of processing parameters and recycling on the properties of recycled polypropylene.

### Materials and Methods

In this study, a virgin copolymer polypropylene Impact (PPc X0) is used. This raw Impact copolymer polypropylene (PPc) was manufactured by Braskem and has properties as shown in Table 1. Braskem's C715 is a PPc specifically designed for injection molding applications [13].

*Table 1 – PPc properties [13]*

Material	Density [g/cm <sup>3</sup> ]	MFI [g/10 min]	Impact Strength at 23°C [kJ/m <sup>2</sup> ]	Flexural Modulus [MPa]
Braskem's C715	0.900	12	10	1450

The objectives within this framework (Fig.1) were to measure the variability in product performances after multiple processing cycles by injection molding of an PPc (X0). Standard specimens ISO 527 type 1A were produced by injection molding with the following dimensions (80 mm × 10 mm × 4 mm) specimens. PPc was subjected to three recycling processes as follows: (X1) was virgin PPc (X0) that had been injected once. A fraction of the molded specimens was weighed and set aside for mechanical testing (flexural and Charpy Impact tests), MFI and bulk density measurements, while the remaining fraction was ground. This material was then re-injected to produce (X2). As before, a fraction of the (X2) specimens was reserved for characterization, while the remainder was ground and re-injected to produce (X3) specimens.

Processing variables, such as injection pressure, cushion volume, plasticizing time, and screw retraction velocity, were monitored to detect any signature of material degradation after multiple reprocessing cycles. Material properties measurements including MFI, flexural modulus, and impact strength, were conducted at each step of the recycling process. Fourier Transform Infrared Spectroscopy (FTIR), Raman spectroscopy and SEM analyses were also performed to identify structural changes in the polymer chain.

Table 2 – Factor Levels

Factor Levels	N°1: Injection molded n times, Xn	N°2: Back pressure, CP [bar]	N°3: Screw rotation frequency, Trmin [rpm]
1	X1	0	50
2	X2	52	80
3	X3	100	-
-	X0	Virgin pellets	

Analyses of variance (ANOVA) were systematically performed, and the results were correlated with the plastication parameters and the number of reprocessing loops, which were chosen as independent factor levels listed in Table 2. We have a total of 18 combinations of process parameters Xn-CP-Trmin. In ANOVA practice, when a significance threshold ( $\alpha$ ) of 0.05 is chosen, the computed p-value must be lower than  $\alpha$  in order for the factor to be considered significant. Here,  $\alpha$  represents the significance level, which is the probability of rejecting the null hypothesis when it is actually true, and the p-value represents the probability of obtaining the observed data, or something more extreme, assuming the null hypothesis is true.

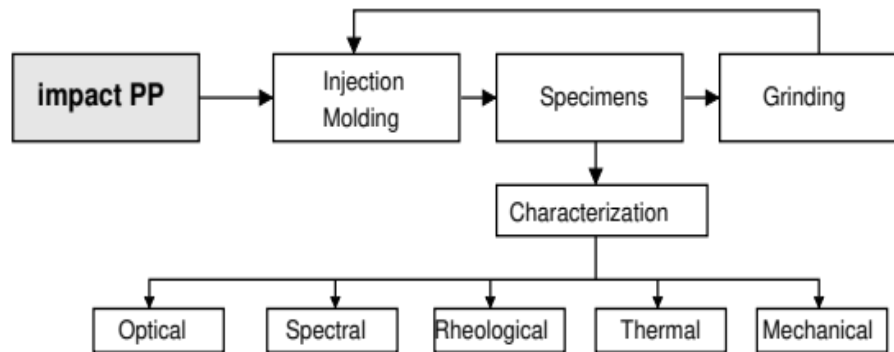


Figure 1 – Recycling methodology

Injection molding was carried out on a DK injection molding machine 110 M-4.02 with a single screw (diameter of 36 mm, length over diameter ratio of 18) with the following processing parameters listed in Table 3.

Table 3 – Injection molding parameters

Holding pressure [bar]	400	Screw rotation frequency [rpm]	50-80
		Injection time [s]	1.2
Holding time [s]	20	Set mold temperature [°C]	45
Cycle time [s]	51.4	Cooling time inside the mold [s]	18
Backpressure [bar]	0-52-100	Injection temperature [°C]	220

For this study, MFI testing was done according to the ISO 1133 at 230°C with a 2.16 kg weight and with a device MI-Tech M3350. To ensure reliability, the MFI of each sample was measured three times, and the average values were reported.

The measurements were carried out using a (PCE-XXM 30) colorimeter designed for quick and precise color measurements on surfaces. This device features a LED light source with a wavelength

range of 400-700 nm, a 8 mm aperture, and high repeatability ( $\Delta E^*_{ab} \leq 0.1$ ). It can be used to measure color differences by comparing specimens to a reference specimen from the first injection cycle (X1-cp17-trmin50). Relative to this reference specimen, the color evolution of all specimens from X1, X2, and X3 is compared to evaluate the effect of the number of recycling cycles on color. The color difference of the material was determined according to ASTM D2244-15, by measuring the differences in  $L^*$ ,  $a^*$ , and  $b^*$  values of the samples and a reference (X1-cp17-trmin50). The total color difference  $\Delta E^*$  was calculated using (Eq. 1), with the differences  $\Delta L^*$ ,  $\Delta a^*$ , and  $\Delta b^*$  determined by (Eqs. 2-3-4), respectively.  $L^*$  represents lightness from dark to light,  $a^*$  represents the color axis from green to red, and  $b^*$  represents the color axis from blue to yellow [14].

Where: (s: specimen and r: reference).

$$\Delta E^* = \sqrt{((\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2)} \quad (1)$$

$$\Delta b^* = b^*_s - b^*_r \quad (2)$$

$$\Delta a^* = a^*_s - a^*_r \quad (3)$$

$$\Delta L^* = L^*_s - L^*_r \quad (4)$$

The bulk density and weight of the specimens were measured using a high-precision Mettler Toledo PB1502-S balance with 0.1 g precision. The bulk density was then calculated using the mass-to-volume ratio.

Three-point bending tests were carried out on five specimens for each processing conditions during each evaluation to determine the flexural modulus. These tests were conducted according to ISO 178:2019 at a speed of 2 mm/min, using a Zwick /Roell BT-FB010TN-D30 testing machine. With 18 different parameter combinations, a total of 90 specimens were evaluated through bending tests with a span distance of 64 mm. We calculated the bending stress (Eq. 5) and bending strain (Eq. 6) using the following formula.

$$\sigma_f = \frac{3FL}{2bh^2} \quad (5)$$

- $\sigma_f$  is the flexural stress parameter considered, in MPa
- F is the applied force, in N
- L is the span, in mm
- b is the width of the specimen, in mm
- h is the thickness of the specimen, in mm

$$\varepsilon_f = \frac{6sh}{L^2} \varepsilon_f \quad (6)$$

- $\varepsilon_f$  is the flexural strain, expressed as a dimensionless ratio
- s is the deflection, in mm

A minimum of 10 specimens from the previously molded specimens for each parameter were tested. Since there are 18 different parameter combinations, a total of 180 specimens were prepared, notched using a Manual notch cutting plane Zwick /Roell, and tested with Charpy Impact testing according to ISO 179-1:2023. Distance between supports: 62 mm, thickness: 4 mm, width: 10 mm, length: 80 mm, with a type B notch and a tip radius of 2 mm. The impact energy of the Charpy pendulum was 2 J. The impact resistance is calculated using the following formula:

$$a_{cN} = \frac{w_c}{h \cdot b_N} * 10^3 \quad (7)$$

- $w_c$  is the corrected energy, in joules, absorbed during the specimen's fracture

- $h$  is the thickness of the specimen, in mm
- $b_N$  is the remaining width of the specimen, in mm

Fracture surfaces of the Impact test specimens were examined using a scanning electron microscope (Jeol NeoScope™ JCM-7000). Before observation, the samples were metallized with gold using a smart coater (DII-29030SCTR) for 1 minute. We used the following parameters for the **backscattered electron signal** analysis: Landing voltage 10 kV, work distance 11.9 mm, magnification \*1,000 and high vacuum mode. Image processing was performed using a specific feature in the JEOL software. Raman spectra of the impact fracture surface were obtained using a Renishaw InVia Reflex spectrometer coupled with a Leica microscope (50x magnification). The excitation wavelength was 514.53 nm, and spectra were recorded in the 300–1100  $\text{cm}^{-1}$  range. The spectral resolution was 3  $\text{cm}^{-1}$  with a wavenumber accuracy better than 0.8  $\text{cm}^{-1}$ . Measurements were performed in backscattering mode on a  $\sim 4 \mu\text{m}^2$  area.

### Results and Discussion

The effect of several processing cycles on the MFI of PPc is shown in Fig 2. It can be seen that the MFI increases moderately with every cycle. As an indicator of melt flowability, the MFI has an inverse relationship with melt viscosity, rising as molecular weight decreases. During multiple injections, the polymer chain of PPc undergoes chain scission, resulting in reduced molecular weight and higher MFI. These results suggest a reduction in melt viscosity and molecular weight during each cycle, with chain scission beginning from the first injection [5], [6].

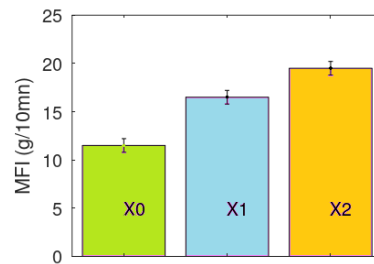


Figure 2 – MFI evolution over the number of processings of PPc

A color analysis was performed in order to evaluate the color change. During injection, a significant change in the color of the specimens could be observed. The specimens became more yellow and darker with every processing cycle. In order to verify this visual impression, a quantitative analysis was performed using a colorimeter. The results indicate that  $\Delta a^*$  and  $\Delta L^*$  show no significant changes, but  $\Delta b^*$  presents variations. For all specimens of X1, with the 18 parameters, no notable difference was observed compared to the reference X1-CP17-Trmin50. However, as shown in Fig. 3, changes are observed with X2 and X3, where the color evolves more significantly with the increase in the number of recycling cycles, rather than with changes in the processing parameters. This is as confirmed by the ANOVA p value equal to zero. Moreover this observation is consistent with the findings of C. Spicker et al. [14], who reported similar trends in their study.

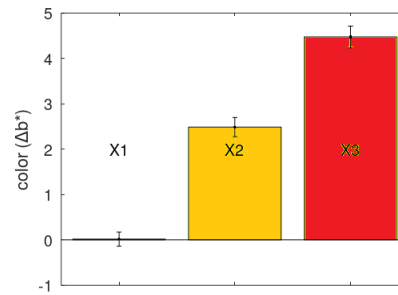


Figure 3 – Evolution of  $\Delta b^*$  over the number of processings of PPc

Bulk density measurements of virgin and regrind PPc are presented in Fig. 4. A notable decrease in bulk density is observed as PPc transitions from specimens to regrind flakes. This is expected from a shredding process. It follows that the screw channel cannot be filled with as much material with flakes than with pellets [14].

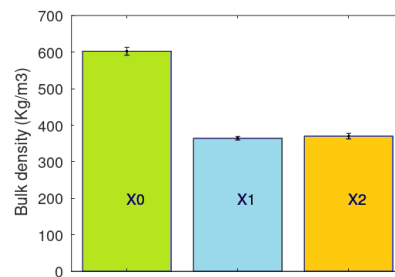


Figure 4 – Evolution of bulk density values over the number of processings of PPc

Recycling cycles do not alter the mass of the injection molded specimens (Fig. 5). Indeed, the p value associated with these factor levels is very large, equal to 0.5.

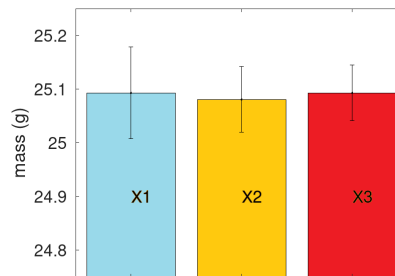


Figure 5 – Evolution of specimen weight values over the number of processings of PPc

Ethylene content in polypropylene copolymers decreases flexural modulus but increases impact strength. The flexural modulus monotonically decreases with repeated injections. Indeed Fig. 6 shows the flexural modulus as function of the number of recycling cycles. There is a small but certain decrease in flexural modulus with the number of recycling cycles, this is confirmed by the very low p-value equal to  $5 * 10^{-6}$ .

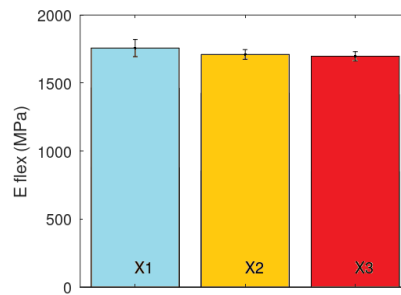


Figure 6 – Flexural modulus as a function of the number of recycling cycles

We studied the Charpy Impact as a function of the number of recycling cycles and noticed that there is no significant effect on impact strength, as shown in Fig. 7. This is confirmed by the ANOVA p value equal to 0.14. We can say that the number of recycling cycles has not influenced it.

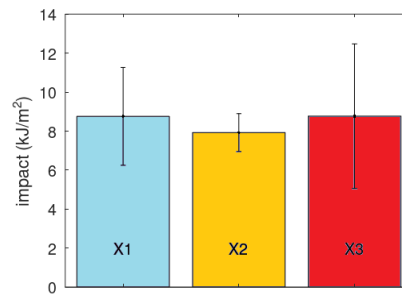


Figure 7 – Impact strength as a function of the number of recycling cycles

The morphology of PPc was studied using a SEM (Fig. 8). Nodules were observed on the fracture surfaces of PPc. Hongjun et al. [15] indicate that SEM results show that ethylene-propylene rubber (EPR) and part of the ethylene-propylene segmented copolymer disperse as toughening nodules in the PPc. In their work they noticed that the size of the EPR phase gradually increased with the number of multiple injections.

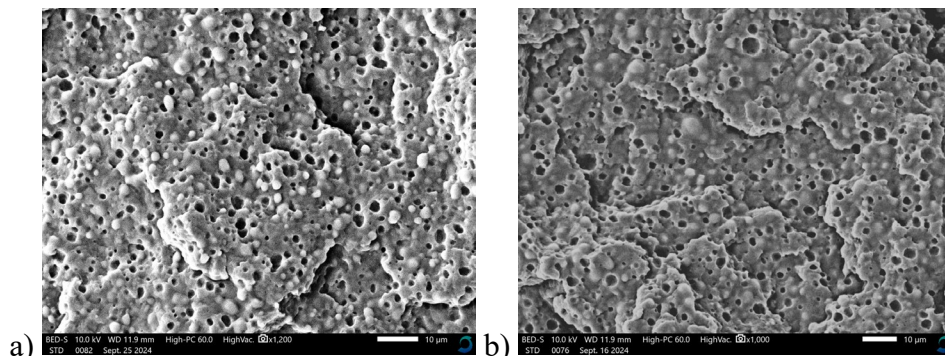
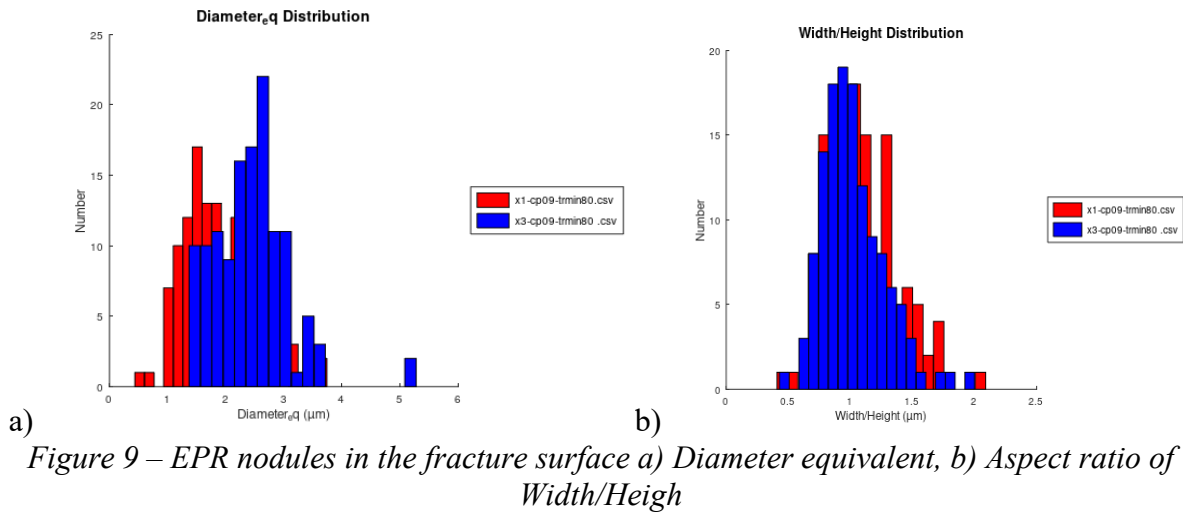


Figure 8 – FTIR of fracture surface of: a) PPc-X1-CP09-Trmin80 and b) PPc-X3-CP09-Trmin80

The X1-CP09-Trmin80 consists of a PPc matrix containing ethylene EPR nodules with an average diameter of 1.9 µm (Fig. 9a). In contrast, the equivalent diameter of X3-CP09-Trmin80 increased to 2.4 µm. This suggests that the EPR nodules size is indeed affected by the recycling process.

A plausible explanation of this result is that when the matrix viscosity decreases under reprocessing it can no longer hinder the coalescence of the ERP nodules. The main consequence

should have been a change in impact properties of the recycled material but there is no evidence yet in our results as shown in Fig. 7.



Moreover, the distribution of the EPR nodules, show that all the nodules have a maximum diameter of less than five microns. The nodule exhibits an aspect ratio close to that of a circle, as the width/height ratio for the majority of them is approximately 1, as shown in Fig. 9b.

Following the analysis conducted with SEM, Raman analysis was performed to further support our findings and confirm that the nodules are composed of EPR.

Regarding the Raman analysis, spectra were recorded from two different areas in Fig. 10: the matrix region on the fracture surface and the nodule region on the same surface. When comparing the matrix and nodule regions, characteristic PE bands are clearly visible in the nodule spectrum, particularly around 1062, 1078, and 1128  $\text{cm}^{-1}$ , as noted by Furukawa et al. [16]. Additionally, the band at 1294  $\text{cm}^{-1}$ , reported by Anderssen et al. [17], is also distinctly visible. This analysis effectively highlighted the presence of PE nodules within the PP matrix.

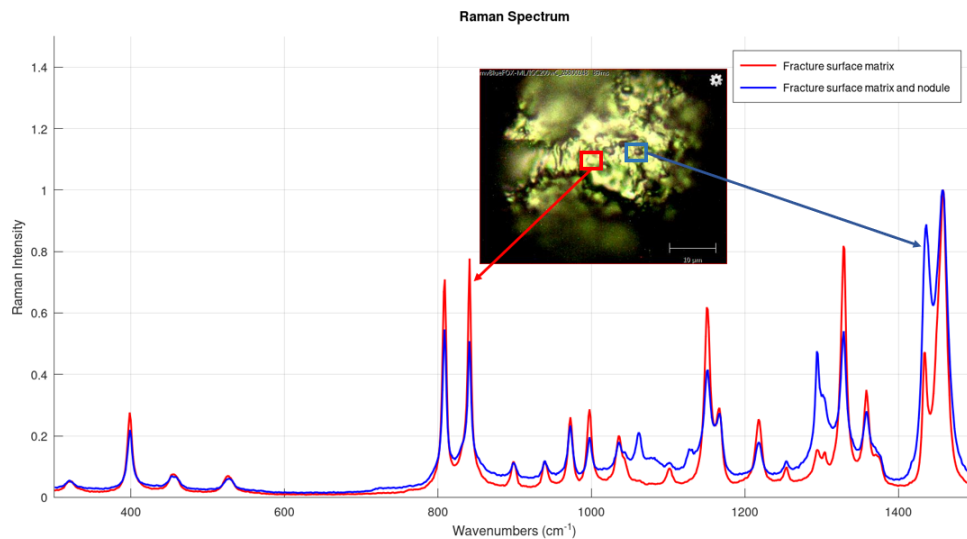


Figure 10 – Raman spectroscopy of the fracture surface of PPc-X1-CP09-Trmin80 (optical microscopy image in insert of the fracture surface)

### Conclusion

The effects of recycling on the structure and mechanical properties of PPc have been investigated. A significant chain scission phenomenon was observed as early as the first reprocessing cycle, as indicated by an increase in the MFI, which is associated with a decrease in viscosity. SEM analysis

revealed that the diameter of the nodules of PE was affected by the number of recycling cycles, showing a marked increase over cycles of recycling. Furthermore, a slight reduction in the flexural modulus was observed, suggesting a degradation in the material's stiffness following multiple recycling cycles. Impact strength, however, did not exhibit significant variation with recycling. While the material's weight remained relatively constant across cycles, noticeable color degradation was observed, with a shift from white to yellow. These results are crucial for optimizing the recycling process and improving the use of recycled materials in component design. Based on the findings, it can be concluded that mechanical recycling holds substantial potential within the framework of a circular economy. For the next steps, we plan to introduce colorant additives to mitigate the color degradation. The visual aspect is often a key factor for perceived quality and consumer acceptance. However, this could potentially affect the mechanical properties of the material. A thermal analysis will be performed to verify certain properties of the polymer (T<sub>g</sub>, T<sub>f</sub>, crystallization enthalpy).

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