

Article

Comparison between the Mechanical Recycling Behaviour of Amorphous and Semicrystalline Polymers: A Case Study

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Abstract: The increase in waste has motivated the adoption of the circular economy concept, which assumes particular relevance in the case of plastic materials. This has led to research of new possibilities for recycling plastics after their end-of-life. To achieve this goal, it is fundamental to understand how the materials' properties change after recycling. This study aims to evaluate the thermal and mechanical properties of recycled plastics, namely polycarbonate (PC), polystyrene (PS), glass fibre-reinforced polyamide 6 (PA6-GF30), and polyethylene terephthalate (PET). With this purpose, injected samples were mechanically recycled twice and compared through thermal and mechanical tests, such as differential scanning calorimetry, hardness, tensile strength, and the melt flow rate. The results show that the amorphous materials used do not suffer significant changes in their properties but exhibit changes in their optical characteristics. The semicrystalline ones present some modifications. PET is the material that suffers the biggest changes, both in its flowability and mechanical properties. This work demonstrates that the mechanical recycling process may be an interesting possibility for recycling depending on the desired quality of final products, allowing for some materials to maintain comparable thermal and mechanical properties after going through the recycling process.

Keywords: injection moulding; thermal and mechanical properties; mechanical recycling; polycarbonate (PC); polystyrene (PS); glass fibre-reinforced polyamide 6 (PA6-GF30); polyethylene terephthalate (PET)



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1. Introduction

With ever-increasing plastic waste, it is essential to work towards understanding both possibilities as well as limitations on the re-introduction of materials into new products [1]. The adoption of the new circular economy action plan (CEAP) by the European Commission in March 2020 made it one of the building blocks of the European green deal [2]. Thus, it is of utmost importance to understand the behaviour of recycled materials in order to allow for increased economic interest on the company's side due to the reduction of virgin material used in addition to being an option for waste reduction, while answering some of the prerequisites of the European Union's (EU) 2050 climate neutrality plan.

According to the already-withdrawn ASTM D7209-06 [3], recycling can be divided into four categories, while ISO 15270:2008 [4], shown in parenthesis and which is still active, divides it into three categories: primary (mechanical recycling), secondary (mechanical recycling), tertiary (chemical recycling), and quaternary recycling (energetic valorisation). According to Goodship et al. [5], mechanical recycling is one of the most common ways of recycling in the plastic industry. Although the ASTM standard divided mechanical recycling into two categories, there are similarities between them; the main difference lies in the fact that primary recycling focuses on grinding down and reprocessing the material, while secondary recycling centres its process in acquiring materials from other sources and might require pre-processing the material by going through some or all the following

steps: size reduction, cleaning, sorting, and re-granulating to a suitable pellet size. In this type of recycling, there may exist some difficulties due to the unknown history of the raw material. Goodship et al. [5] also emphasise that these difficulties may affect the properties of the material depending on: the “Amount of re-processing steps; extent of thermal degradation; contamination and/or a mixture of materials; mono or co-polymer; typology of previous use; and service-life conditions”. The answers to these questions allow companies to focus more on primary recycling since there is knowledge about the material’s history (closed-loop cycle). The fact that secondary recycling implies a more complex procedure does not make it less used, but it is more focused on specific conditions of the material such as non-PSW (Plastic Solid Waste) and plastic mixtures of variable composition, for example.

In an attempt to comply with the EU’s 2050 climate neutrality plan, companies have been working on reducing the PSW that they produce. To do this, plastic manufacturers have made some adjustments to their manufacturing strategies in order to fulfil the plan’s demands. Nevertheless, manufacturers still find it hard to implement waste management systems that can guarantee the demands; the first reason lies in the fact that there are still many companies that lie on a linear economy strategy, and secondly, this is due to the lack of infrastructure for the recycling process [6–8]. To greatly implement mechanical recycling into a company’s core, there is the necessity to answer some of the challenges that still exist. Hahladakis et al. [9] define as challenges to mechanical recycling: composition, which can lead to promoting rejection of recycled plastic due to deterioration of material properties; sorting ability, which can reduce the quality of the recycled material; contamination, which greatly affects any attempt at a closed-loop process and promotes rejection of the material; degradation both at reprocessing and at service life due to oxidation and illumination for the latter, implying material instability and consequently leading to rejection; compatibilization, this leads to allowing for only one cycle of recycling; substitute ability, this regards mixing virgin and recycled plastics and may lead to faster degradation; marketability, this is affected by the supply-and-demand dynamics and worsens the ability of a company to recycle; and feasibility, this is one of the most important aspects since it can affect the ability to recycle certain types of materials.

By progressively understanding the limitations of the materials after re-processing them through mechanical recycling, it is possible to ease the transition to a more circular economy with respect to plastics. This is the main objective of this work: to understand how some types of polymeric materials are affected through the successive use of mechanical recycling. This study aims to evaluate the variation in thermal and mechanical properties that occur after each cycle of processing while trying to understand to what extent the performance of each of the materials is affected or not. Thus, samples of virgin and recycled materials (polycarbonate, polystyrene, glass fibre reinforced polyamide 6, and polyethylene terephthalate) were obtained by injection moulding to evaluate their thermal behaviour (differential scanning calorimetry), flowability (melt flow rate), and mechanical properties (hardness and tensile tests) [10–16].

This study showed that some of the materials possess variability in their properties, namely, at the level of their optical characteristics, thermal and mechanical properties, and in their flowability. It reinforces that an understanding of the properties of recycled materials is of utmost importance to guarantee an increase in transitioning to a circular economy.

2. Results and Discussion

2.1. Injection Moulding

With the injected samples, the first analysis to be done was the optical characteristics of each material. Thermoplastic materials can be of two types: amorphous or semicrystalline. In this work, there are two of each type: PC and PS on the amorphous side, and PA6-GF30 and PET as the semicrystalline ones.

Due to their disorganized chains, amorphous materials are either transparent or translucent materials, which makes one of the desired properties when used in injection moulding. Figure 1a,b show the variability in optical properties in PC and PS, which show a loss of transparency after each cycle and a visible degradation of the material that can be identified through its increased opacity.

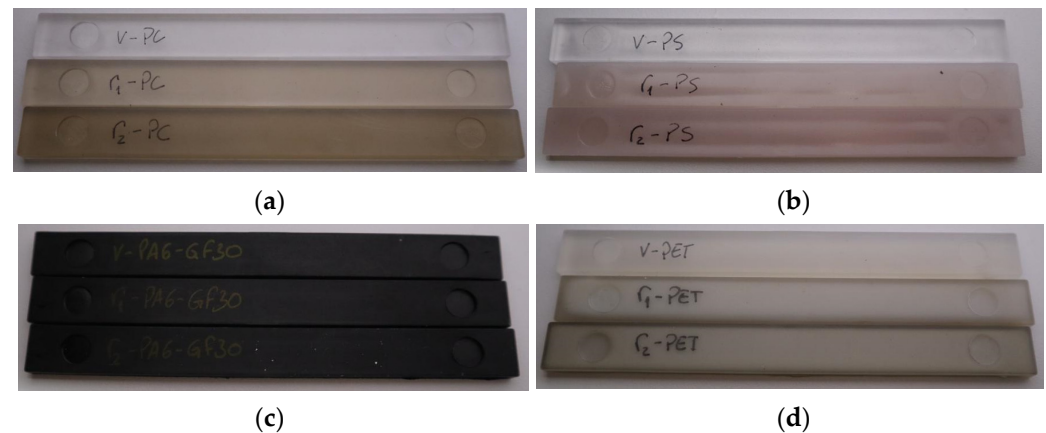


Figure 1. Examples of the injection-moulded specimens: (a) PC; (b) PS; (c) PA6-GF30; (d) PET.

When looking at semicrystalline materials, the optical characteristics to look for is the appearance of a yellowish tone, which may indicate degradation. On the one hand, in PA6-GF30 (Figure 1c), there is no clear variability by observing the specimens; on the other hand, PET (Figure 1d) has a particular optical analysis to be done. When looking at the recycled materials, it is possible to see that there is some degradation after each reprocessing cycle through the appearance of a yellow tone and that with the successive processing of the material, this ends up having high changes in its morphology. When looking at the virgin material, it is similar to the amorphous material, but when recycled, it starts to lose its transparency, mainly at its core. As described by Lin et al. [17], this may be justified by the fact that PET exhibits a moderate crystallization rate, and when cooled too quickly it will retain its amorphous state since the chains do not have enough time to reorganize. Meanwhile, the recycled materials appear to have time for this reorganization in their core, which might be related to the property found in the DSC analysis defined as “cold crystallization”.

2.2. Density Analysis

Table 1 shows the density results for each material. As it is possible to understand, there is little variation in density between recycling cycles in PC and PS. In the case of PA6-GF30, due to the shortening of the glass fibres triggered by the mechanical recycling process, an increase in density is expected, which happens because of the decrease in fibre length, which in turn increases the fibre weight ratio, i.e., a higher volume of fibres that are denser than polyamide. It is also possible to observe a slight increase in density between each cycle of PET, which may happen due to the decrease in the ability to absorb humidity, as stated by Nadali et al. [18].

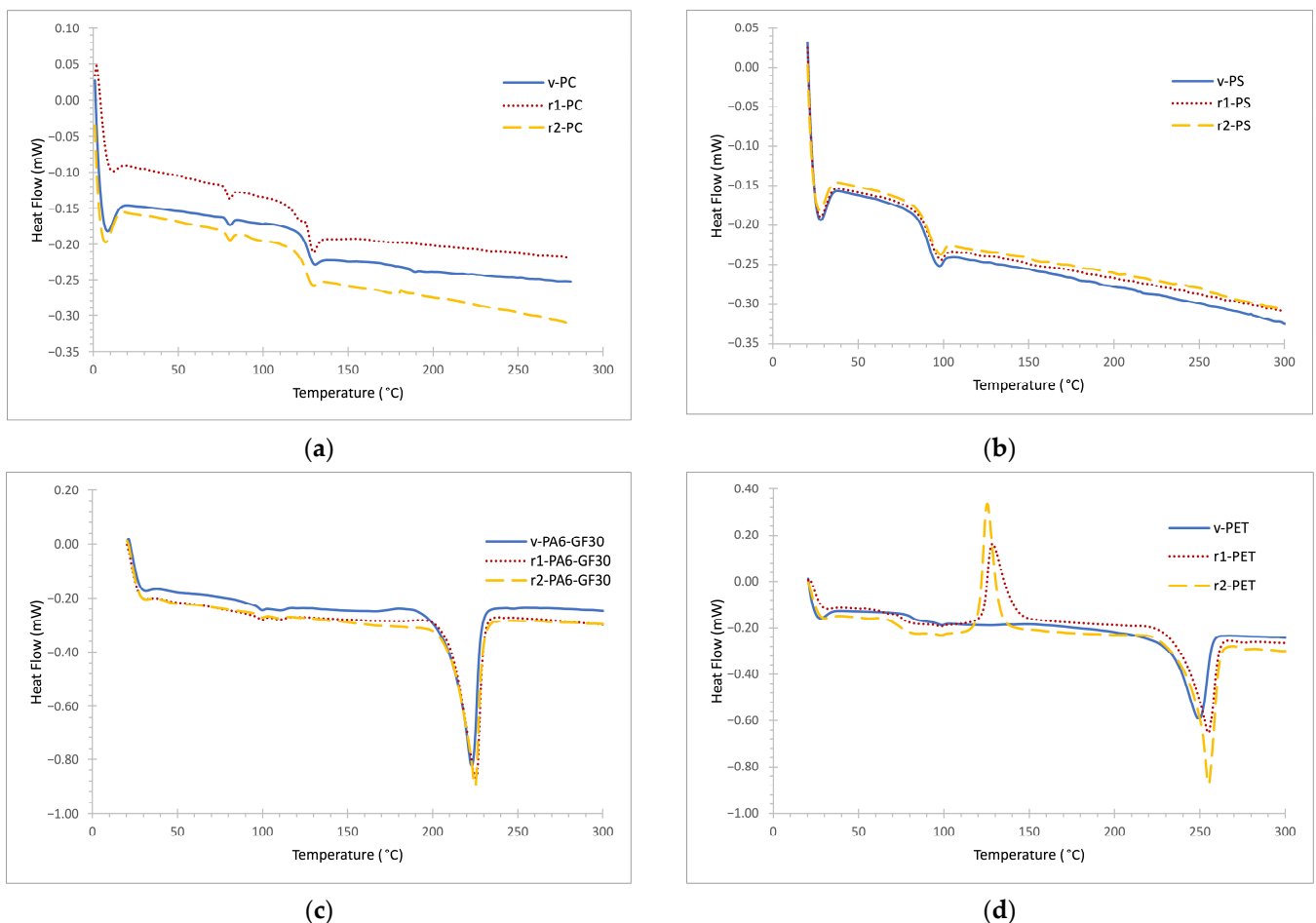
Table 1. Density of the studied materials was tested according to the guidelines in ISO 1183-1:2019 [19].

Material	Density (kg/m ³)
v-PC ¹	1199.1 ± 1.6
r1-PC ¹	1196.9 ± 0.5
r2-PC ¹	1197.7 ± 0.4
v-PS ²	1043.9 ± 0.2
r1-PS ²	1043.3 ± 0.3
r2-PS ²	1043.8 ± 0.6
v-PA6-GF30 ¹	1295.8 ± 14.8
r1-PA6-GF30 ¹	1337.3 ± 18.6
r2-PA6-GF30 ¹	1346.3 ± 2.8
v-PET ¹	1331.7 ± 2.6
r1-PET ¹	1346.2 ± 1.5
r2-PET ¹	1359.4 ± 2.0

¹ H₂O density @ 24 °C: 997.5 kg/m³; ² H₂O density @ 23 °C: 997.3 kg/m³.

2.3. DSC Analysis

The differential scanning calorimetry results shown in Figure 2 were performed to understand the eventual degradation in thermal properties. This study tries to understand if it is possible to reuse the material when it is mechanically recycled, only considering thermal properties. In other words, if the processability of the material remains intact or if it has meaningful changes.

**Figure 2.** DSC analysis of the virgin and recycled materials: (a) PC; (b) PS; (c) PA6-GF30; and (d) PET.

In the case of amorphous materials, it is only possible to observe the glass transition temperature (T_g), since in these types of polymers the macromolecules are randomly organized. For semicrystalline materials, due to the presence of crystallites, along with T_g , it is possible to observe an endothermic peak that corresponds to the melting temperature (T_m). Table 2 summarizes the DSC results for virgin and recycled materials.

Table 2. Glass transition and melting temperature for virgin and recycled materials.

Material	Glass Transition Temperature ($^{\circ}\text{C}$)	Melting Temperature ($^{\circ}\text{C}$)
v-PC	144.3	-
r1-PC	143.7	-
r2-PC	142.8	-
v-PS	89.3	-
r1-PS	89.7	-
r2-PS	89.2	-
v-PA6-GF30	44.0	221.6
r1-PA6-GF30	42.5	225.6
r2-PA6-GF30	43.5	222.7
v-PET	79.7	249.4
r1-PET	72.2	254.9
r2-PET	73.7	255.0

When comparing v-PC to r1-PC and r2-PC, as shown in Figure 2a, it is possible to detect that T_g does not suffer significant variation, meaning that reprocessing does not have an effect on this property. In the case of PS, another amorphous polymer (Figure 2b), there are no significant changes in T_g values.

Regarding PA6-GF30, presented in Figure 2c, there is almost no change in T_g , but there is a slight increase in melting temperature from the virgin to the recycled materials.

Finally, Figure 2d shows that for PET there is little variation in T_g and T_m between processing cycles. In this case, it is important to highlight the presence of an exothermic peak during heating. This peak, defined as “cold crystallization” temperature (T_c), appears at 128.4°C and 126.7°C for r1-PET and r2-PET, respectively, but not in v-PET. Although the temperatures are similar, the appearance of this cold crystallization peak might explain the differences in optical properties mentioned in Section 3.1.

Similar behaviour for PET is reported by Torres et al. [11] and Cañadas et al. [20], who state that the exothermic peak arises from the “crystallization of the amorphous phase” and is typical in polymers such as PET, PPS, and PEAK. According to Torres et al. [11], this phenomenon “arises from the weak mobility of planar benzenic nuclei and can easily be explained: chains being frozen, the heating involves a critical mobility leading to the reorganization of the structure”.

2.4. MFR Analysis

The MFR is a property of plastic materials that allows an understanding of the ease of flow of the material at a specific temperature. Throughout this work, measurements were made to compare the ease of flow between the virgin and recycled samples. In some cases, as shown in Table 3, the reprocessing completely changes the flowability of the material. Although a high MFR can benefit the injection moulding process, if this value is too high it can lead to the appearance of defects in the injected components.

Table 3. Melt mass flow rate results for virgin and recycled materials tested according to the guidelines in ASTM D1238-13 [21].

Material	Melt Mass Flow Rate (g/10 min)
v-PC ¹	17.1 ± 1.2
r1-PC ¹	18.9 ± 0.9
r2-PC ¹	25.1 ± 0.8
v-PS ²	15.7 ± 0.5
r1-PS ²	18.3 ± 0.8
r2-PS ²	18.3 ± 0.9
v-PA6-GF30 ³	9.6 ± 0.8
r1-PA6-GF30 ³	20.5 ± 0.7
r2-PA6-GF30 ³	28.8 ± 0.9
v-PET ⁴	23.1 ± 1.1
r1-PET ⁴	112.4 ± 4.7
r2-PET ⁴	126.0 ± 3.7

¹ 300 °C–1.20 kg; ² 235 °C–2.16 kg; ³ 200 °C–5.00 kg; ⁴ 260 °C–2.16 kg.

As expected, all materials present an increase in melt mass flow rate after successive mechanical recycling processes. Nonetheless, two materials are deserving of some more critical analysis, namely PA6-GF30 and PET, since they present a significant increase after recycling.

2.5. Hardness Tests

Hardness evaluation allows to understand the resistance towards the indentation of a material. The results of the hardness tests (Shore D) are shown in Table 4.

Table 4. Shore D hardness results of virgin and recycled materials were tested according to the guidelines in ISO 868:2003 [22].

Material	Hardness (Shore D)
v-PC	68.6 ± 1.9
r1-PC	72.4 ± 1.6
r2-PC	72.5 ± 1.2
v-PS	79.4 ± 0.7
r1-PS	76.0 ± 0.8
r2-PS	73.9 ± 1.1
v-PA6-GF30	71.1 ± 1.5
r1-PA6-GF30	71.3 ± 1.6
r2-PA6-GF30	70.4 ± 1.7
v-PET	62.9 ± 1.8
r1-PET	68.8 ± 1.4
r2-PET	69.7 ± 1.7

As it is possible to realize in Table 4, there are minimal changes in hardness between the virgin and recycled materials.

2.6. Tensile Tests

The most representative test to evaluate mechanical properties is the tensile test. In this work, the test was carried out according to the guidelines in ISO 527:2019 [23], with the objective of understanding if the ability to deform elastically would change with the recycling process and if tensile strength would suffer any significant modification. The results of the tensile tests for the PC, PS, PA6-GF30, and PET virgin and recycled specimens are shown in Figures 3–6. The corresponding values of Young's modulus (E) and tensile stress (σ_y) are shown in Table 5.

The results for polycarbonate (PC), as shown in Figure 3, indicate a minimal loss in strength between each recycling cycle while maintaining its value for Young's Modulus (E).

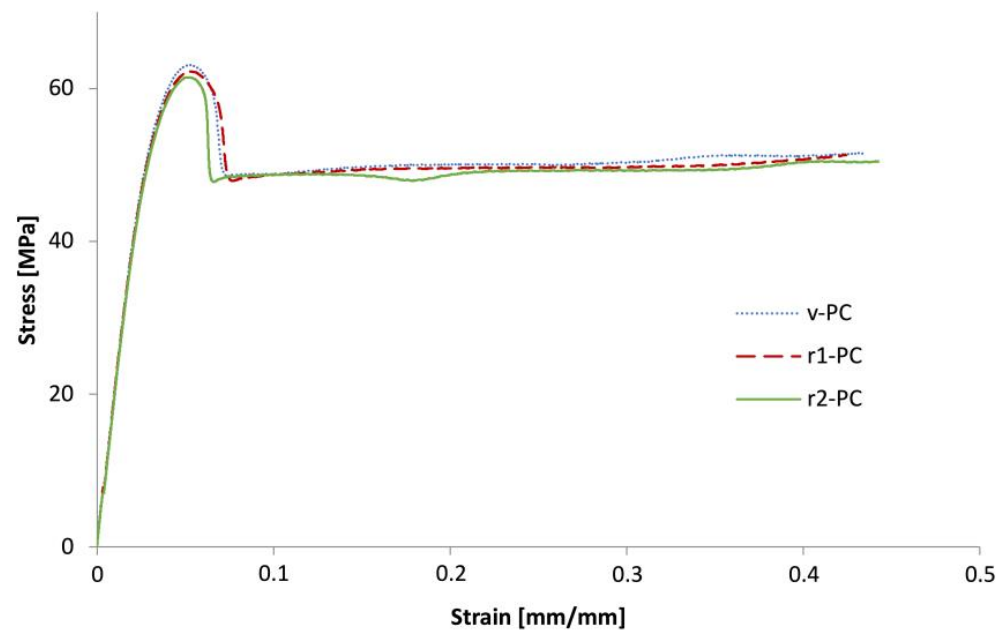


Figure 3. Stress–strain curve of virgin and recycled PC.

The tensile properties of polystyrene (Figure 4) show a reduction in its strength after recycling while observing a slight variation in the elasticity modulus.

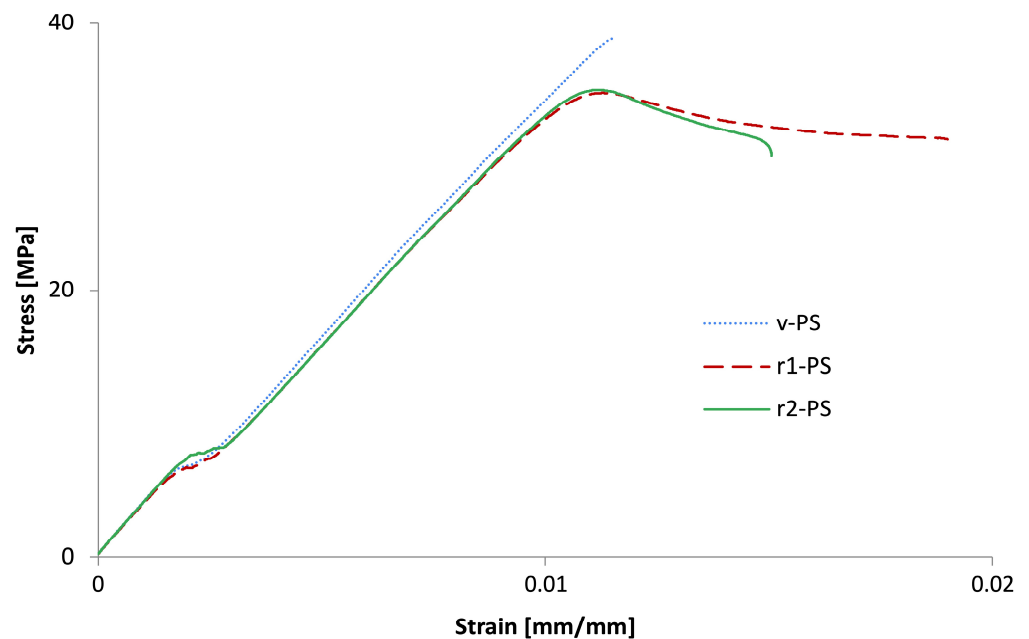


Figure 4. Stress–strain curve of virgin and recycled PS.

By analysing the mechanical properties of PA6-GF30 (Figure 5), it is possible to observe that there is not a significant change. This means that after recycling the material twice, it can still resist similar tensile loads.

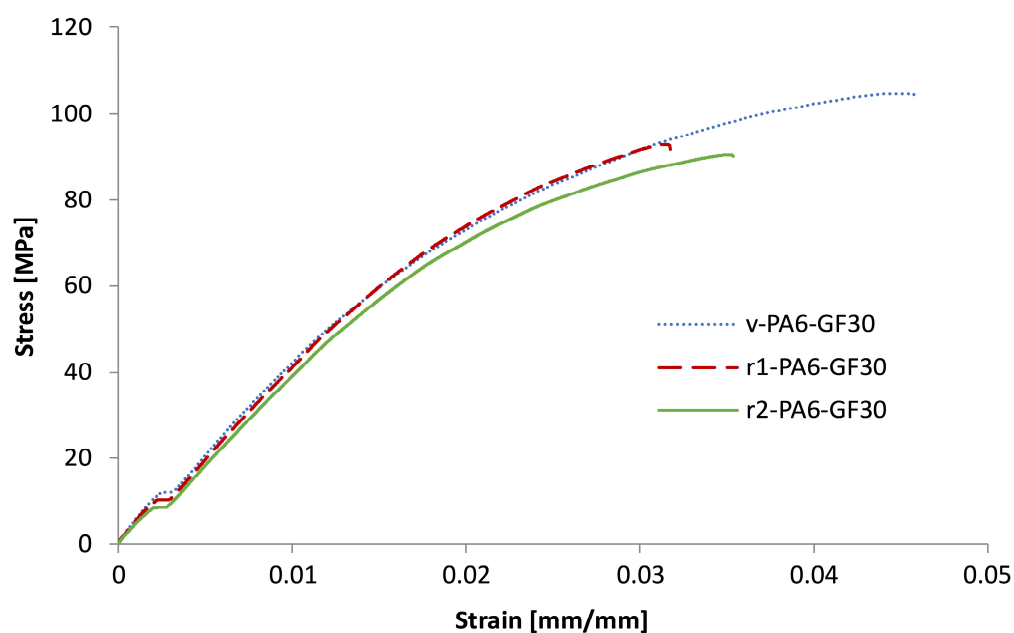


Figure 5. Stress–strain curve of each processing cycle of PA6-GF30.

The changes in mechanical properties (Figure 6) from v-PET to r1-PET can be related to the higher melt flow rate, as shown in Table 5, which might promote higher molecular orientation. After a second recycling cycle, the results show that there is a notorious loss of properties, displaying the characteristics of a fragile material.

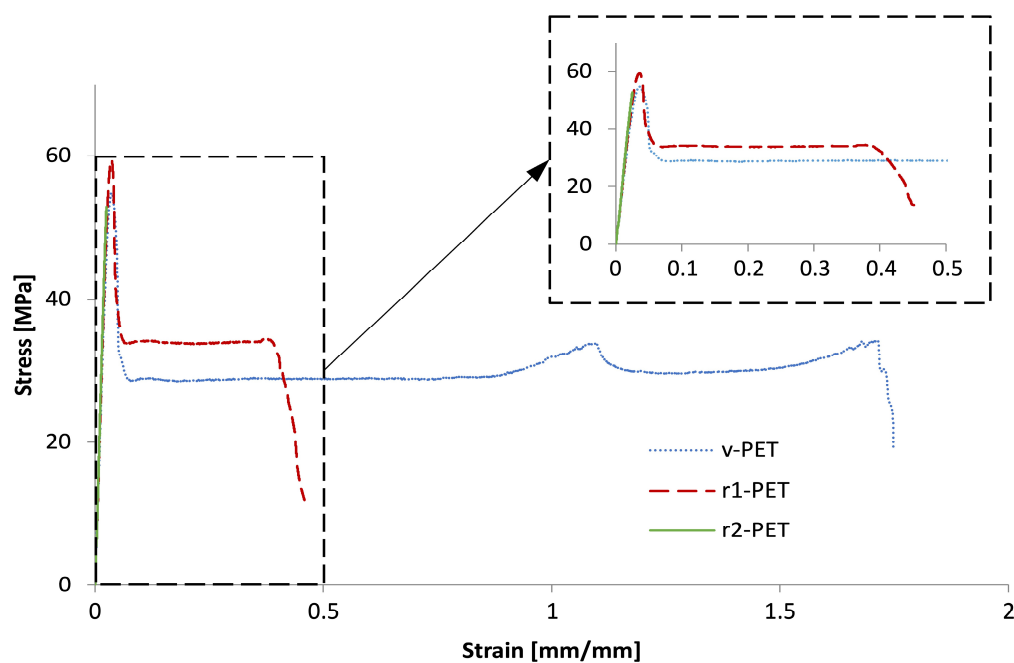


Figure 6. Stress–strain curve of each of processing cycle of PET.

Table 5. Mechanical properties for virgin and recycled materials tested according to the guidelines in ISO 527:2019 [23].

Material	Young's Modulus (MPa)	Tensile Stress (MPa)
v-PC	1992.7 ± 48.3	63.3 ± 1.9
r1-PC	1957.2 ± 25.9	61.9 ± 0.6
r2-PC	1953.4 ± 15.5	61.8 ± 0.4
v-PS	2707.6 ± 23.6	38.6 ± 0.6
r1-PS	2664.3 ± 25.6	35.1 ± 1.1
r2-PS	2621.5 ± 46.9	34.9 ± 0.7
v-PA6-GF30	4049.6 ± 61.1	103.1 ± 1.0
r1-PA6-GF30	4271.7 ± 54.9	101.5 ± 1.6
r2-PA6-GF30	4086.5 ± 21.1	91.5 ± 1.5
v-PET	1847.3 ± 19.6	53.8 ± 1.0
r1-PET	2103.8 ± 69.6	60.8 ± 1.7
r2-PET	2269.7 ± 11.5	49.7 ± 1.0

3. Materials and Experimental Work

3.1. Materials

The virgin materials used in the present work were two amorphous polymers—polycarbonate (v-PC) named Lexantm Healthcare Resin HP3REU and supplied by SABIC (Riyadh, Saudi Arabia), and polystyrene (v-PS) provided by INEOS Styrolution (Frankfurt am Main, Germany) named Styrolution PS 124N/L—and a semicrystalline one, namely, glass-fibre-reinforced polyamide 6 (v-PA6-GF30) named Ultramid B3EG6 Polyamide 6 from BASF SE (Ludwigshafen, Germany). Since there was interest in also analysing two semicrystalline materials, polyethylene terephthalate (v-PET) was selected due to its high use in the packaging industry, which is associated with a high volume of solid residues. However, although there was the availability of this material, there was not any information about its source. To confirm the presence of the functional groups of PET, Fourier-Transform Infrared Spectroscopy (FTIR) analysis was carried out.

FTIR analysis was performed with an Alpha-P ATR FTIR from Bruker (Billerica, MA, USA) using a KBr disk. The spectra were collected from 400 to 4000 cm^{-1} with a 2 cm^{-1} resolution on three samples of each virgin material.

Figure 7 shows the spectre of the virgin PET used throughout this study. The results were corroborated by comparing this material with the literature (Table 6) [24,25].

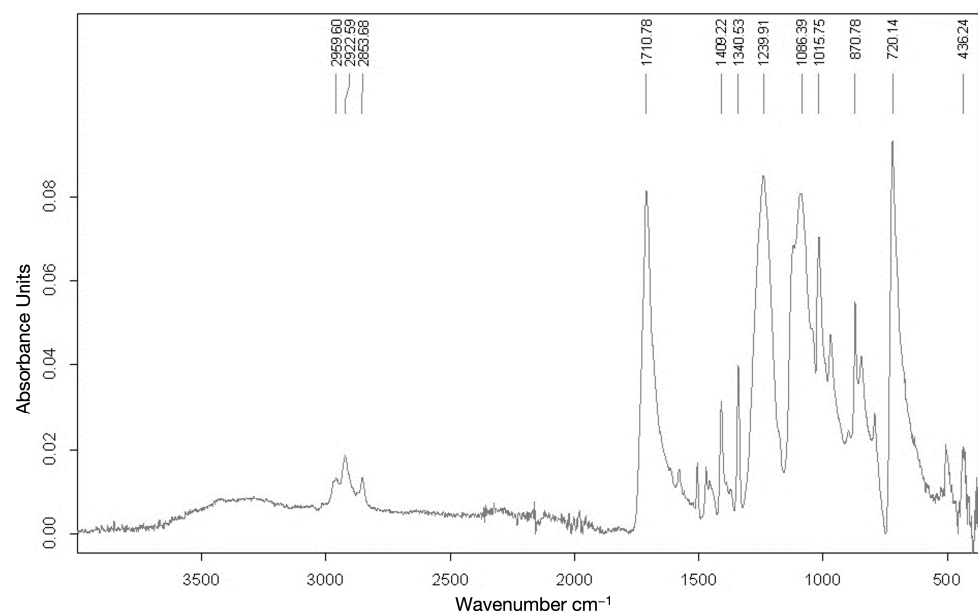
**Figure 7.** FTIR analysis of v-PET.

Table 6. Wavenumbers and respective identifiable functional groups for v-PET [24,25].

Wavenumber (cm^{-1})	Functional Groups
2959.59 and 2853.68	Symmetrical stretching of C-H
1710.78	Stretching of C=O of the carboxylic acid group
1409.22 and 1340.53	Stretching of the C-O group and deformation of the O-H group
1239.91	Terephthalate group ($\text{OOC}_6\text{H}_4\text{-COO}$)
1086.39	Methylene group
870.78	Aromatic rings
720.14	Benzene ring and polar ester group interactions

3.2. Experimental Work

3.2.1. Injection Moulding

The processing method to obtain the recycled materials was injection moulding through a EUROINJ D80 from LienYu Machinery Co., Ltd. (Tainan, Taiwan) injection machine with a clamping force of 80 tons. The materials were injected into a cavity with geometry to obtain tensile and flexural specimens (Figure 8).

**Figure 8.** Example of the geometry of the injected geometry.

To obtain the first cycle of the recycled specimen (referred to as r1-PC, r1-PA6-GF30, r1-PS, and r1-PET), the virgin material was initially injected. A part of the specimen was kept for density analysis and mechanical testing (hardness and tensile tests), and another was mechanically recycled by grinding. A portion of the recycled materials (samples/granules) was saved to evaluate thermal behaviour and the melt flow rate, and the other portion was used to repeat the grinding and injection process to obtain the second cycle of the recycled specimen (referred to as r2-PC, r2-PA6-GF30, r2-PS, and r2-PET).

The injection moulding injection conditions are presented in Table 7. Since the materials may degrade or change their properties, such as their melt flow rate, the premise of the setup parameters was to maintain the injection and mould temperatures the same for each group of materials (virgin, cycle 1 recycled, and cycle 2 recycled).

Table 7. Injection moulding processing conditions.

Injection Parameters	PC ¹	PS	PA6-GF30 ²	PET ³
Injection Temperature (°C)	270 ± 15	230 ± 15	225 ± 15	255 ± 15
Mould Temperature (°C)	85	40	65	70
Injection Pressure (MPa)	10	10	10	10
Packing Pressure (MPa)	8	8	8	8.5 ⁴ /8 ⁵
Packing Time (s)	5	4	3	20 ⁴ /4 ⁵
Cooling Time (s)	50	25	35	50

¹ Material previously dried for 4 h at 120 °C. ² Material previously dried for 4 h at 83 °C. ³ Material previously dried for 12 h at 140 °C. ⁴ Parameters for v-PET. ⁵ Parameters for r1-PET and r2-PET.

3.2.2. Density

Density measurements were obtained through a Mettler Toledo's AG204 (Greifensee, Switzerland) scale and performed according to the ISO 1183-1:2019 standard [19] by using the immersion method. The temperature of the distilled water was 23 °C for the PS samples and 24 °C for the rest of the materials. To understand if there was any variation in density after each recycling process, five tests were performed for each material.

3.2.3. Differential Scanning Calorimetry

Differential Scanning Calorimetry (DSC) analysis was carried out to evaluate the thermal behaviour of the virgin and recycled samples, namely glass transition and melting and/or crystallisation temperatures, using a DSC 124 Polyma from NETZSCH (Selb, Germany) equipment. The samples (11–15 mg) were placed on metal crucibles, and then the measurements were performed in two runs in an inert gas atmosphere (N₂) at a heating rate of 10 °C/min from 20 °C to 300 °C, the second run was intended to delete the material's thermal history, and this is where the value for T_g (glass transition temperature) was taken from.

3.2.4. Melt Flow Rate

The melt flow rate (MFR) was determined by a Ray-Ran 6 MBA by the DKSH Group (Zurich, Switzerland), where the same conditions were used for each type of virgin and recycled sample. PC samples were tested with a load of 1.2 kg at 300 °C, PA6-GF30 samples were subjected to a 2.16 kg load at 235 °C, PS samples were tested with 5 kg at 200 °C, and PET samples were tested with 2.16 kg at 260 °C. All tests followed ASTM D1238-13 [21].

3.2.5. Hardness (Shore D)

The Shore D hardness measurements of each material followed the ISO 868:2003 standard [22]. The measurements were taken at the 15 s mark. The equipment used for the analysis was the Shore D Härteprüfer HP series by Bareiss (Oberdischingen, Germany). To execute the test, a 5 kgf weight was applied at room temperature.

3.2.6. Mechanical Analysis

To perform the tensile tests, the machine used was a Zwick Z100 from Zwick Roell (Ulm, Alemanha). The tests were performed at room temperature without drying the samples.

The system recorded both the load applied and the displacement of grips automatically. The tests were performed through the application of displacement-controlled loading. The test speed used in all materials was 5 mm/min with a distance between grips of 100 mm, and the load cell used was a 10 kN maximum load. The mould cavity used for injecting the samples followed the basis of the ASTM D638-02A standard [23], and therefore the specimens used were of type I.

To obtain the stress–strain curve, the tensile stress was calculated as the force related to the initial cross-section area of the gauge length of the tested sample. The standard also defines tensile strength as the maximum recorder tensile stress and elongation at break as the gauge length deformation at break. For these tests, the effect of transverse deformations of the sample during the test was not considered.

4. Conclusions

The study shows that some properties change between virgin and recycled materials. Amorphous materials do not present significant variations in their thermal and mechanical properties, with around a 2–4% decrease in tensile stress. Nonetheless, their optical characteristics show a loss of transparency after recycling once. For the semicrystalline materials shown throughout this study, both increase their flowability after reprocessing, but PET is the material that shows the biggest variability in properties, where a high MFR and complete change in mechanical behaviour are the main changes detected.

The results of this work allow understanding that recycled materials may come with some compromises depending on the objective of their use.

It is important to emphasise that if plastics are to be continuously used, the focus should lie on improving the implementation of a circular plastics economy. This means that when designing plastic components, the recycling of these materials should be considered.

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Conflicts of Interest: The authors declare no conflict of interest.

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