Research Article

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Effects of mechanical recycling on the properties of glass fiber–reinforced polyamide 66 composites in automotive components

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Abstract: In this study, we aimed to reveal the effective reusability of waste generated during the injection molding process of polyamide 66 (PA66) reinforced with 30 wt% of short glass fiber (PA66-GF30) widely used in the automotive industry. PA66-GF30 was subjected to the three mechanical recycling cycles, including regranulation and reinjection molding steps, and the recycled materials obtained in each of these cycles were included at the ratios of 15, 20, 25, and 30 wt% to the virgin composite. Thermogravimetric analysis and differential scanning calorimeter analyses showed that the number of recycling cycles and recycled material content in the composite had no significant change in the thermal stability and crystallinity degree of the PA66-GF30. The average fiber length determined by optical microscope analysis shifted to lower values from 300–350 to 150–250 μm by increasing the number of recycling cycles and the recycled material content. The fact that the recycled material content in the composite exceeds 25 wt% and the recycling cycle is applied three times played a key role in changing the mechanical and melt flow behaviors of the composite. Tensile strength, elastic modulus, and impact energy slightly decreased while the elongation at break and melt flow index increased.

Keywords: mechanical recycling, polyamide 66, glass fiber–reinforced composites, automotive components, injection molding

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1 Introduction

In the last few decades, plastics have become essential and indispensable materials that are widely used in packaging, construction, automotive, electrical and electronic, aerospace, energy, and medical industries due to their versatile, unique properties such as lightweight, ease of processing, low cost, excellent mechanical properties, good corrosion resistance, durability and recyclability (1–4). Therefore, the production of plastics is increasing year by year, and the growth trend is expected to continue for the next decade. It has been reported that in 2021, global fossil-based plastic production rose about 4% to reach 352.3 million metric tons, indicating continued strong demand for plastics (5). On the other hand, the increasing production and consumption of plastics bring large volumes of plastic waste, adversely affecting human health, the environment, and the economy, as well as their many advantages. Since synthetic plastics, most of which are produced from fossil fuels, are not easily degraded in nature due to their strong physical durability and chemical stability, they takes a long time to decompose and create a space problem in the collection of plastic wastes. In addition, the improper accumulation of this non-biodegradable waste leads to serious environmental pollution and resource depletion, which destabilizes the ecosystem during the long extinction period (6–8).

Recycling plastics is one of the most effective ways to reduce the negative effects of accumulated plastic waste on ecosystems and to encourage the most efficient use of resources (7,9). With this approach, waste plastics can be converted into new products, extending their useful life and reducing waste generation, creating a more sustainable system (10,11). Although only 8% of the world's plastic production has been recycled, the plastic waste recycling rate has been showing a remarkable increase in recent years. In Europe, 35% of post-consumer plastic waste was recycled in 2020; thus, the recycling rate of these wastes increased by 117% from 2006 to 2020 (5). Moreover, the use of recycled plastics has continued to grow every year in many industrial areas, especially in the automotive sector, which is one of the largest end-use markets for plastic materials (1,12). According to the European End-of-Life Directive, at least 95% of a vehicle mass must be reused or recovered (13). In the last decade, the amount of plastics used in vehicles has increased, and the current amount has reached 15–17% of the total weight and 50% of the volume of an average modern car since the decrease in weight leads to a reduction in fuel consumption and CO_2 emission (14,15). Therefore, recycling or reuse of plastics in the automotive industry has become inevitable today due to its great contributions to the environment, resource conservation, economy, and sustainability (16).

Polyamide 66 (PA66) is one of the most widely used engineering thermoplastics in the automotive industry in many interior and exterior components such as engine parts, bumpers, gears, bearings, fuel systems, electrical components, trims, and door panels (12,15,17,18) as it exhibits high mechanical strengths and stiffness, good resistance to heat, chemicals, lubricity and wear, and good processability (10,17,19). However, there is always a need to further improve the properties of PA66 for some of its specific uses in under-the-hood applications. The reinforcement of PA66 with glass fibers allows for stabilizing the polymer matrix and superior mechanical properties, increase in heat distortion temperature, dimensional stability, satisfactory corrosion resistance and low cost of the final material (20–23). Although the glass fiber content can be varied depending on different uses, application requirements, and design features, most of the reinforced PA66 components used in under-the-hood applications are injection-molded composite materials with 30–40% short glass fiber content by weight, contributing to improved engine efficiency and reduced energy consumption and emissions (18,24). As well as the fiber content, the length, distribution, diameter and orientation of the fibers and the interfacial interactions between the polymer matrix and the fibers are crucial parameters that determine the final performances exhibited by the injection molded short glass fiber reinforced (SGFR) PA66 composites (14,25–28).

Another critical aspect is the recycling of SGFR PA66 composites. They can be recycled through different processes such as mechanical, chemical and thermal (15,29). Mechanical recycling, consisting of the steps of regranulation, remelting and reinjection molding, respectively, is the most used approach for this type of composites because it is an efficient, high output and technically and industrially feasible method, can replace virgin material production, and has acceptable costs and the least adverse environmental impacts, comparing to other recycling methods (11,24,29–31). However, it is worth emphasizing that there are some limitations to the mechanical recycling of the fiber-reinforced composites because of the reprocessing operations. During the recycling processes, fiber breakage and thermomechanical degradation of the polymer matrix occur due to exposure to repeated grinding, high temperatures, and shear forces, resulting in the deterioration of some important mechanical properties and reduced durability of the final composites as well as affecting the melt flow behavior of the polymer mixture (14,32–35). Therefore, it is of great importance to understand the behavior of fiber–reinforced thermoplastic materials during the recycling process, to reveal the effect of reprocessing operations on the properties of composites and to achieve efficient recycling and reuse of these composites.

In the literature, there are some studies on the mechanical recycling of glass fiber–reinforced PA66 composites. Eriksson et al. investigated the effects of fiber shortening due to grinding followed by injection molding on the tensile and impact strength of PA66 reinforced with 30 wt% of short glass fiber samples. Comparing the virgin sample with the eight times operated material, it was observed that the average fiber length, tensile strength, and impact strength decreased by 57%, 31%, and 39%, respectively (32). In another study, they studied fiber length changes and matrix and fiber–matrix interface degradation after recycling processes of the same composite. They found that fiber shortening predominates during compounding and the first injection molding cycle, with more regrinding and remolding having lesser of an effect and reprocessing had a negligible effect on the strength for both the fiber–matrix interface and the matrix of this system (36). Bernasconi et al. conducted similar experimental studies on test samples obtained by adding mechanically recycled 35 wt% glass fiber reinforced PA66 granules, PA66 GF 35 to virgin composite at different ratios. It was found that both the elastic modulus and the tensile strength decreased with the increase of the recycled material content, while the strain at break increased (37). They also investigated the effect of reprocessing on the fatigue behavior of PA66 GF 35. The processed material at different ratios, 25%, 50%, and 100%, was blended with the virgin material. They confirmed that fiber shortening from the injection molding process is responsible for reducing stiffness and strength, and the fatigue strength decreased by the presence of the reground material (38). Pietroluongo et al. mechanically recycled the end-of-life radiator part based on PA66 reinforced with short glass fiber of 35.7% three times and examined their mechanical properties. The tensile and bending strength values of the end-of-life sample mechanically recycled three times decreased by 41% and 35%, respectively, compared to the unused reference sample, as a result of the shortening of the fiber length caused by the grinding and molding steps in the recycling process (39). Licea-Claverie et al. examined the effect of reprocessing on the microstructure and mechanical properties of injection molded hybrid PA 6.6 short glass/carbon fiber composite. Up to five recycling cycles, an increase in tensile strength, Young modulus and impact strength was observed, while molecular weight and average fiber size decreased. They also determined that the matrix fractures occurred by increasing fiber pullout detected from Scanning Electron Microscopy (SEM) images, and the mechanical properties decreased after five cycles (40).

In this study, the main purpose is to reveal the effective recycling and efficient reusability of wastes generated during the injection molding process of PA66 reinforced with 30 wt% of short glass fiber (PA66-GF30) used in the production of engine fasteners in the automotive industry. That is, it was primarily aimed to reduce the amount of plastic waste released into the environment by recycling the high amounts of waste produced by the production of PA66-GF30, which is widely used in the automotive industry, to use raw material resources more efficiently and to get a sustainable and more economical production by reincorporating waste into production in this way. In this context, we focused on restraining the degradation during processing, incorporating recycled materials into production without losing product properties, and also being environmentally and economically advantageous applied processes. The recycled composites were prepared by mechanical recycling processes, including regranulation and reinjection molding steps, respectively. Due to the worsening of some key properties of products manufactured from recycled plastics, the recycled PA66-GF30 composites obtained through multiple reprocessing cycles were blended with the virgin composite at different weight ratios to overcome this problem. To get a useful recycled material that retains high-quality product properties, it is important to know how many times to recycle the composite and how much the optimum amount of recycled material is in it. First, PA66-GF30 was subjected to the three recycling cycles, and the recycled materials obtained in each of these cycles were included at the ratios of 15, 20, 25, and 30 wt% to the virgin composite. Then, the effects of the recycling cycle and recycled material content on the thermal, microstructural, melt flow, and mechanical properties of PA66-GF30 products were extensively investigated and compared with those of the virgin sample.

2 Materials and methods

2.1 Materials

In this study, a PA66-based polymer composite material, including 30 wt% of short glass fiber additive (average fiber length of 300–350 μm) with a density of 1.37 g·cm^{-3}, designed to increase the resistance to automotive coolant and suitable for use in injection molding method, was used. This material, with the trade name Technyl A 218 V30 Black 34 NG and the trade code PA66-GF30, was obtained from Solvay Türkiye.

2.2 Preparation of recycled PA66-GF30 composite samples

Recycled PA66-GF30 composites were prepared by mechanical recycling processes consisting of regranulation followed by reinjection molding steps in three different recycling cycles and at different recycled material ratios of 15, 20, 25, and 30 wt%, their sample codes and formulations are given in Table 1. The following process steps were applied in order to obtain these composite samples.

2.2.1 Preparation of first recycling cycle samples

The injection molding process of virgin PA66-GF30 (R0) granules and the steps of the applied mechanical recycling process are shown in detail in Scheme 1. As seen, the injection molded samples were produced in two different forms: automobile engine fasteners and plates for the preparation of mechanical test samples. Before the injection processes, the granules of all samples were dried in an air-circulating oven at 85°C ± 10°C for 4 h. To obtain the engine fastener products, the virgin 30 wt% glass fiber–reinforced PA66 composite granules (R0) were molded using a DEMAG

Table 1: Compositions of recycled PA66-GF30 composite samples

Sample code	Material content
R0	Original virgin granules of PA66-GF30
R0*	Granules obtained after injection molding of R0 followed by grinding
R15	15 wt% R0* + 85 wt% R0
R20	20 wt% R0* + 80 wt% R0
R25	25 wt% R0* + 75 wt% R0
R30	30 wt% R0* + 70 wt% R0
RR15	15 wt% R15 + 85 wt% R0
RR20	20 wt% R20 + 80 wt% R0
RR25	25 wt% R25 + 75 wt% R0
RR30	30 wt% R30 + 70 wt% R0
RRR15	15 wt% RR15 + 85 wt% R0
RRR20	20 wt% RR20 + 80 wt% R0
RRR25	25 wt% RR25 + 75 wt% R0
RRR30	30 wt% RR30 + 70 wt% R0

plastic injection machine with an injection pressure of 600 bar, a holding pressure of 900 bar, and an injection speed of 80 mm·min^{-1}. During the processes, mold and injection machine screw zone temperatures were set at 85°C and, 300°C, 300°C, 300°C, 300°C, and 285°C, respectively. On the other hand, the injection process of the plate samples to be used in mechanical tests was carried out at 400 bar injection pressure, 650 bar holding pressure, and 50 mm·min^{-1} injection speed, and all other conditions were kept the same. Thus, injection-molded products of R0 granules were obtained.

In order to perform the first recycling cycle, the engine fastener products obtained after the injection molding process were granulated in a crushing machine under atmospheric conditions. These granules were mechanically mixed with the virgin granules (R0) at the ratios of 15, 20, 25, and 30 wt% to get four different mixtures, and then, they were blended by injection molding process under the same process conditions detailed above to manufacture two different products, namely, the engine fasteners and the plates. Thus, the first recycling cycle was completed by applying all the processes shown in Scheme 1 step by step, and the samples containing 15, 20, 25, and 30% recycled material by weight were named R15, R20, R25, and R30, respectively.

2.2.2 Preparation of second recycling cycle samples

The injection-molded engine fastener products and plates containing 4 different ratios of recycled materials (R15, R20, R25, and R30) obtained in the first recycling cycle were regranulated separately in the crushing machine under atmospheric conditions. These granules in four different

Scheme 1: Schematic representation of the mechanical recycling process of the PA66-GF30 composite.

compositions were mechanically mixed with the R0 granules at the ratios of 15, 20, 25, and 30 wt%, and then the injection processes were applied again in the same way as the first cycle sample preparation to get injection molded products in the forms of engine fasteners and plates. After this regranulation followed by the reinjection process, the second recycling cycle was completed according to the process steps given in Scheme 1, and the samples with a recycled material ratio of 15, 20, 25, and 30 wt% were obtained, called RR15, RR20, RR25, and RR30, respectively.

2.2.3 Preparation of third recycling cycle samples

The injection molded engine fastener products and plates containing four different ratios of recycled materials (RR15, RR20, RR25, and RR30) obtained in the second recycling cycle were regranulated separately in the crushing machine under atmospheric conditions. These granules in four different compositions were mechanically mixed with the R0 granules at the ratios of 15, 20, 25, and 30 wt%, and then, the same injection processes were applied as in the preparation of the first and second recycling cycles samples, as shown in Scheme 1. After these regranulation and reinjection processes, the third recycling cycle was completed and the samples obtained with a recycled material ratio of 15, 20, 25, and 30 wt% were called RRR15, RRR20, RRR25, and RRR30, respectively.

2.3 Characterizations

Thermogravimetric analysis (TGA) of the virgin and the recycled composite materials was carried out using the SII Nanotechnology-SII6000 Exstar TG/DTA 6300 instrument at a heating rate of 10°C·min^{-1} in the temperature range of 25–800°C under N_2 atmosphere.

Differential scanning calorimeter (DSC) analyses of the samples were performed using a Seteram/Labsys instrument at a heating rate of 10°C·min^{-1} in the temperature range of 30–350°C under the N_2 atmosphere. Melting data, including peak melting temperature (T_m) and enthalpy of melting (ΔH_m) of the samples, were obtained from this analysis, and the degree of crystallinity (X_c) was also calculated by the Eq. 1 below:

$$X_c(\%) = \frac{\Delta H_m}{\Delta H_m^o\ (1-\alpha)} \times 100 \tag{1}$$

where ΔH_m^o is the melting enthalpy of 100% crystalline PA66 polymer, whose value is 196 J·g^{-1} (41), ΔH_m is the measured melting enthalpy from the DSC analysis, and α is the weight fraction of glass fibers in the PA66 composites.

Glass fibers were taken out from the composite matrix by the burning method. Incineration of composite samples according to ISO3451-1:2019 (42) standard was carried out in a muffle furnace at 600°C ± 25°C. Thus, glass fibers were extracted from the ash obtained. To determine the average fiber length of the fibers, they were dispersed on a glass microscope slide and photographed using a Nikon ECLIPSE MA100 optical microscope.

SEM analysis of fractured surfaces of tensile test samples of the composites was carried out using Zeiss EVO® LS 10 SEM instrument after the fractured surfaces of the samples were coated with a thin conductive layer of Au–Pd by applying spraying method.

Tensile test samples of all composites were prepared by cutting the plate-shaped composite products obtained by injection molding process in a CNC machine according to ISO 527-1BA standard (43). The tensile tests of the prepared samples were performed using an Instron 3369 Universal tensile test machine equipped with a 1 kN load cell *t* at a crosshead speed of 100 mm·min^{-1} according to ASTM D638 standard (44). The average value of three test samples was taken for each polymer composite.

Charpy notch impact test samples were prepared according to ISO 179-1 standard by applying the cutting process in a CNC machine from the plate-shaped composite products obtained by the injection molding process. Then, the Charpy notch impact tests of the samples, whose notches were created according to ISO179 – Type B standard, were carried out using a Devotrans impact tester according to ISO 179 standard (45). The average value of three test samples was taken for each polymer composite.

Shore-D hardness test of the composite samples was carried out according to the ISO 868:1985 standard (46).

Melt flow index (MFI) analysis of the composite samples was performed at 280°C ± 5°C under 5 kg load according to ISO 1133 standard (47) using an Instron MFI Tester.

3 Results and discussion

3.1 Thermal characterizations of recycled PA66-GF30 composites

TGA was used to investigate the effect of the recycling cycle and recycled material content on the thermal stability of virgin PA66-GF30 composite (R0). Thermal stability of the composites was evaluated through the TGA curves, which show the change in weight of the samples with temperature and the differential thermogravimetry (DTG) curves indicating the weight loss rate, which is a derivative of the

TGA chart. Figure 1(a)–(d) shows the TGA and DTG curves of virgin PA66-GF30 and recycled composites, and also the correlative data, the temperature of 5% weight loss (T_5), the temperature of 10% weight loss (T_{10}), the temperature of 50% weight loss (T_{50}), the temperature of maximum weight loss (T_{max}), and the residue percentage at 800°C are given Table 2. As seen in the TGA curves, all composites displayed a similar one-step thermal degradation process.

In the TGA and DTG curves of virgin PA66-GF30 composite, the initial weight loss of less than 0.5% occurred between 250°C and 270°C, corresponding to the volatilization of residual moisture or adsorbed water in the composite structure. The main degradation was between 355°C and 500°C with a maximum degradation temperature of 445.2°C. The weight loss in this temperature range was found to be 68.5% due to the degradation of the main chains of the PA66 matrix (39). The residue of the virgin composite at 800°C was 30.4%, indicating the amount of glass fiber reinforcer remaining in the composite material without decomposition, and this was equal to the amount of glass fiber in the PA66-GF30 composite with a theoretical weight ratio of 30% reported by the supplier.

The TGA and DTG curves of the recycled composites obtained as a function of the recycling cycle, and recycled material ratio showed no remarkable change as their thermal degradations occurred in one step, just as in the virgin composite, as shown in Figure 1(a). The initial decomposition temperatures (T_5 and T_{10}) of the recycled samples were lower than that of the virgin PA66-GF30 due to early polymer matrix degradation starting in recycling cycles (48). At the same time, there were slight differences between the T_{50} and T_{max} values of the virgin and

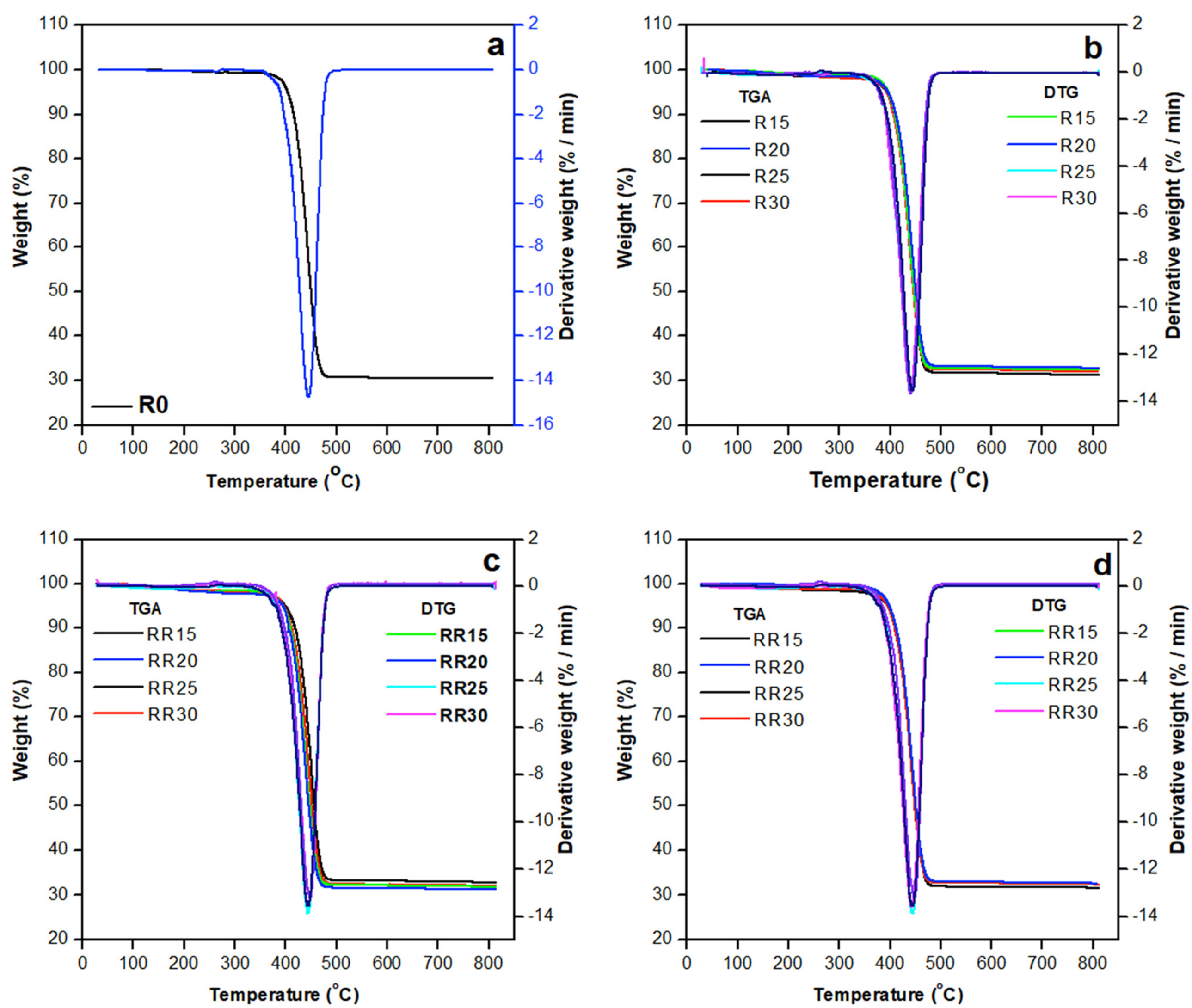

Figure 1: TGA and DTG curves of virgin and recycled PA66-GF30 composites.

Table 2: Thermal decomposition data of virgin and recycled PA66-GF30 composites

Sample (%)	T_5[a] (°C)	T_{10}[b] (°C)	T_{50}[c] (°C)	T_{max} (°C)	Char residue[d]
R0	404.1	416.1	450.2	445.2	30.4
R15	394.1	408.3	447.3	442.0	31.2
R20	394.4	407.2	446.7	440.3	31.9
R25	397.0	409.8	447.7	441.7	32.4
R30	396.2	411.9	451.0	444.4	32.8
RR15	398.5	418.9	456.7	444.3	32.9
RR20	396.3	411.9	452.2	447.3	32.2
RR25	394.5	409.3	447.4	447.3	31.9
RR30	394.1	405.7	445.5	444.1	31.3
RRR15	395.0	410.9	450.0	444.3	31.7
RRR20	395.3	409.8	449.0	442.9	32.5
RRR25	397.8	414.5	452.6	448.4	32.9
RRR30	398.5	412.5	451.1	444.2	32.7

[a, b, c]Temperature at which 5, 10, and 50 wt% loss is achieved.
[d]Char residue obtained at 800°C.

recycled composites (Table 2). The residues of the recycled composites at 800°C varied between 31.2% and 32.9%, slightly higher than the 30.4% of the virgin composite. The results indicated that the virgin PA66-GF30 composite protected its thermal stability against the applied mechanical recycling for up to three cycles and the presence of recycled material up to 30 wt% obtained from different recycling cycles in the composite. It can be concluded that the number of recycling cycles and recycled material ratio had no significant deteriorating impact on the thermal stability of the PA66-GF30.

Nonisothermal DSC analysis was carried out to determine the melting behavior of virgin and recycled PA66-GF30 composites. From this analysis, DSC curves that show the change in heat flow of the samples with temperature were obtained. The DSC melting curves of all composites for the temperature range of between 180°C and 300°C are given in Figure 2(a)–(d). The peak melting temperature (T_m), enthalpy of melting (ΔH_m), and degree of crystallinity (X_c) of the samples obtained from data of the endothermic peaks on the first heating DSC curves are displayed in Table 3. All composites exhibited almost the same behavior with a single melting endothermic peak between 235°C and 275°C, independently from the recycling cycles or the recycled material content, as shown in Figure 2.

For the virgin PA66-GF30 composite, T_m and ΔH_m values were found to be 264.6°C and 47.3 $J{\cdot}g^{-1}$, respectively. Also, its crystallinity degree was 34.5%. Incorporating recycled materials subjected to different recycling cycles into the virgin PA66-GF30 in varying weight ratios slightly decreased the T_m and ΔH_m values, ranging from 264.0°C to 261.7°C and 47.1 to 41.2 $J{\cdot}g^{-1}$, respectively. In addition, the crystallinity degree of the recycled composites was lower than that of virgin PA66-GF30 composite, regardless of the cycle number and recycled material content, and the value varied between 34.3% and 30.0%. During reprocessing of polyamide composites, polymeric chain scission probably occurs, resulting in shorter polymer chains with lower molecular weight. This can lead to an increase in the rate of crystallization and the formation of smaller-sized and defective crystals. In addition, increasing the crystallization rate may be caused by increased nucleation due to the existence of impurities introduced into the matrix during processing and memory effects related to histories of thermal and mechanical stresses remaining in the sample after injection molding (33,49). Consequently, these parameters may explain the slight reduction observed in the degree of crystallinity as well as the melting points of the recycled PA66-GF30 composites.

3.2 Microstructures of recycled PA66-GF30 composites

The fiber content, length, aspect ratio, orientation, and interfacial strength are the main microstructural parameters that determine the final mechanical and physical properties exhibited by injection-molded fiber-reinforced thermoplastic composites (25). Mechanical recycling processes and the recycled material content affect fiber length and fiber length distribution in these composites. During processing, the fibers are often broken, shortening the fibers and changing their length distribution (32,50). Therefore, both fiber shortening and orientation are important aspects to consider in evaluating the effect of a recycling process on the final properties of fiber-reinforced thermoplastic composites.

To determine the effect of the recycling cycle and recycled material content on the length of glass fibers and their length distributions in the recycled PA66-GF30 composites, the glass fibers were extracted from the polymer matrix by burning each sample obtained with different ratios of recycled material subjected to different recycling cycles. The extracted fibers for each composite sample were analyzed under an optical microscope, and the optical micrographs obtained for the virgin and recycled PA66-GF30 composites are given in Figure 3. By using these micrographs, the length of fibers was measured, and the frequency histograms of fiber length distributions of all composites were displayed in Figure 4. As seen in the optical micrograph of virgin composite, R0 (Figure 3), almost all fibers were of the same length with a longer

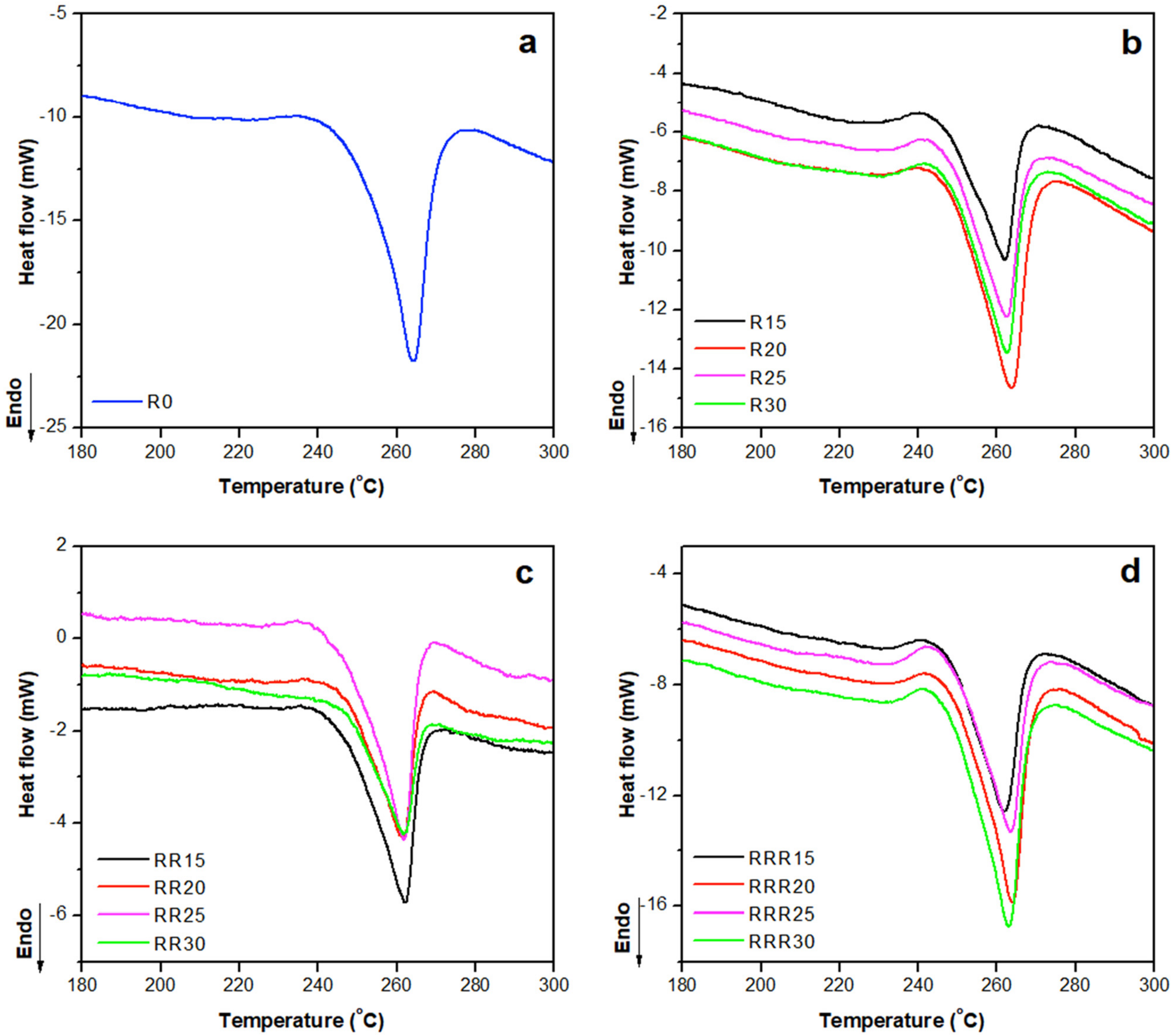

Figure 2: DSC melting thermograms of virgin and recycled PA66-GF30 composites.

Table 3: DSC data of virgin and recycled PA66-GF30 composites

Sample	T_m (°C)	ΔH_m ($J \cdot g^{-1}$)	X_c (%)
R0	264.6	47.3	34.5
R15	262.1	47.1	34.3
R20	263.6	43.9	32.0
R25	262.5	41.7	30.4
R30	262.5	42.8	31.2
RR15	262.0	41.2	30.0
RR20	261.7	43.2	31.5
RR25	261.8	43.1	31.4
RR30	261.8	44.1	32.1
RRR15	261.8	42.8	31.2
RRR20	264.0	42.5	31.0
RRR25	263.5	43.7	31.9
RRR30	263.0	44.3	32.3

size. Their average length was found to be around 300–350 µm, with a fiber length distribution between 150 and 600 µm. As can be clearly observed from Figures 3 and 4, the shortening of the glass fiber occurred after each recycling cycle, and more than 60% of the glass fibers were found to be between 100 and 300 µm in length for all recycled composites. The increase in the number of recycling cycles and the recycled material content in the composites at each cycle shifted the average fiber length to lower values, indicating that the glass fibers were exposed to breakage during each mechanical recycling process and injection molding (26,51,52).

In addition, a variation was observed in the fiber length distribution of the composite samples, depending on the recycling cycles and the recycled material ratios (Figure 4). The composites obtained after the first recycling

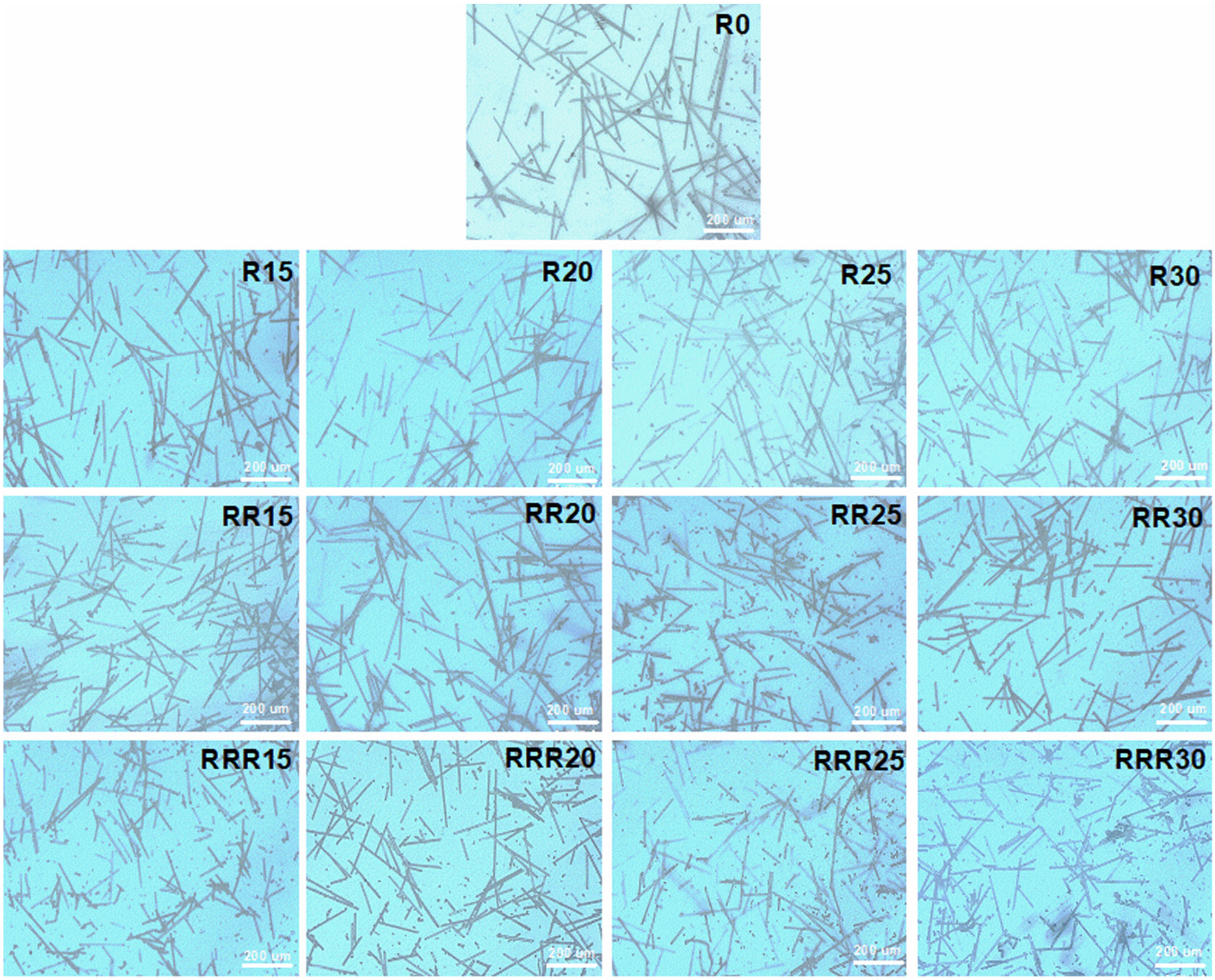

Figure 3: Optical microscope images of the glass fibers extracted from the PA66-GF30 composite samples.

cycle (R15, R20, R25, and R30) showed wider fiber length distribution than the virgin PA66-GF30 composite, R0. With the increasing number of recycling cycles, the length distributions narrowed slightly due to an increase in the number of short fibers (39,40,52). On the other hand, the increment in the amount of recycled material incorporated in the virgin composite in each cycle resulted in a broadening of the fiber length distributions corresponding to the increasing number of fibers in a wide variety of lengths, large and small.

SEM analysis was performed to examine the microstructure of the PA66-GF30 composites containing various amounts of recycled material obtained by subjecting different numbers of recycling cycles. Figure 5 exhibits the SEM micrographs of fractured surfaces of tensile test samples of the virgin and recycled composites obtained at a magnification of 500×.

For the injection molded fiber reinforced composites, the fibers close to the surface of the sample are highly oriented, aligned parallel to the injection flow direction compared to those at the center which tend to be transversely oriented (53,54). The SEM micrograph of virgin PA66-GF30 composite (R0), given in Figure 5, showed that the long glass fibers were randomly oriented, and there was a homogeneous dispersion of these fibers in the matrix as well as a good fiber–matrix adhesion. In addition, the fracture surface of this composite was rough, and the voids appeared as a result of pulling out some fibers from the matrix during the tensile test. On the other hand, its morphology showed a noticeable change by incorporating recycled materials, depending on the recycling cycle and composite composition.

As can be clearly seen from the SEM micrographs in Figure 5, as the number of recycling increased, the length

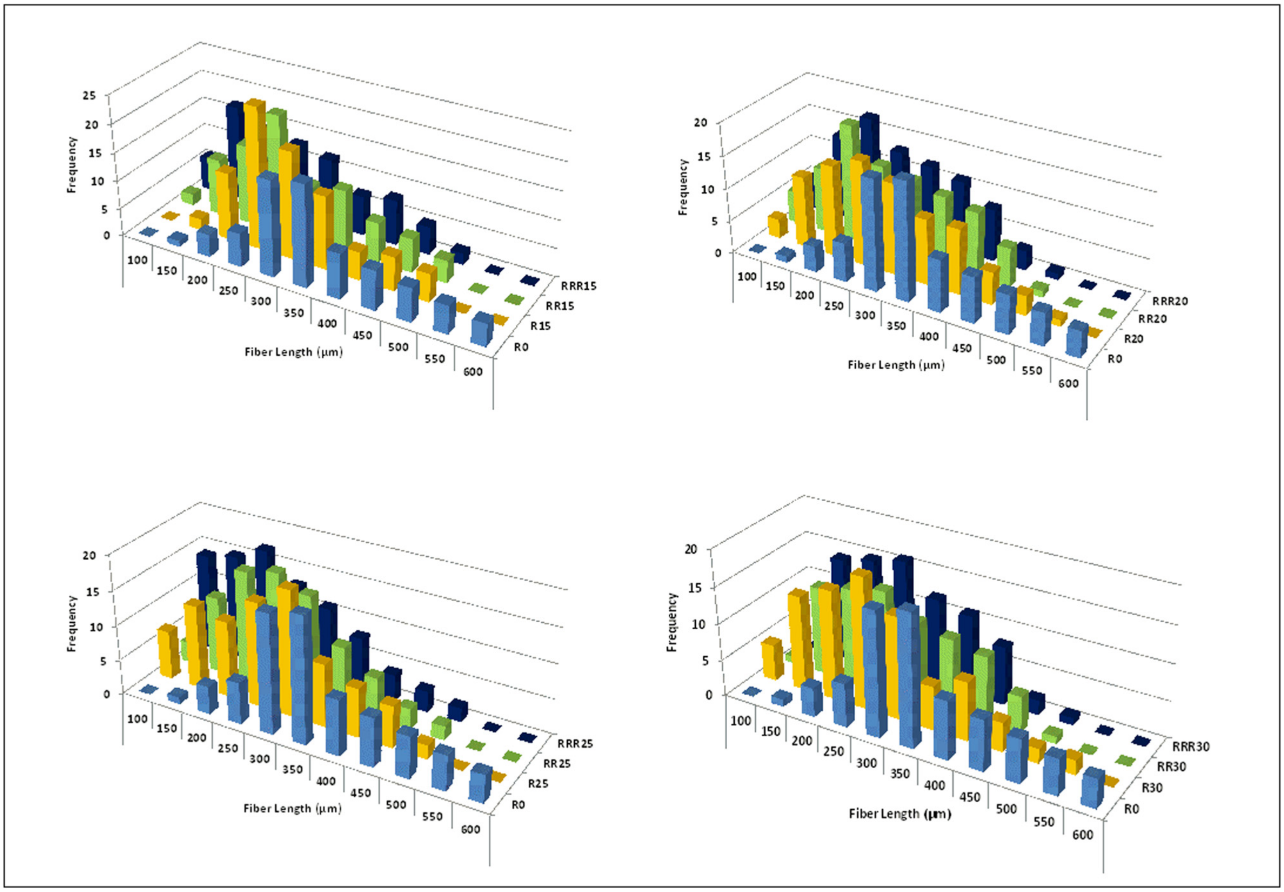

Figure 4: Fiber length distributions of the virgin and recycled PA66-GF30 composites.

of the fibers extending from the matrix became shorter and the diversity in fiber lengths increased. It was also observed that there was a more random fiber orientation and good interfacial adhesion between the fibers and polymer matrix, although the homogeneous dispersion of fibers was lost in some regions. Similar morphological behavior was revealed by increasing recycled material content in the composite with each recycling cycle. However, the fact that the recycled material in the composite exceeds a certain amount, such as more than 25 wt%, resulted in a heterogeneous dispersion of the fibers. Fiber bundles emerging in this heterogeneous distribution can be considered structural defects such as voids (24).

As mentioned in the literature, the length of the fibers also affects the failure mechanisms of the material, which may occur in the form of matrix fracture, fiber pullout, and fiber breakage. During fracture, in the presence of shorter fibers, the fibers are predominantly pulled out from the matrix, and thus, the formation of voids is even more pronounced (14,24). It was clearly observed that this effect significantly changed the morphology of the polymer phase, especially in composites where the recycling cycle was applied above a certain number. All recycled composites containing material that had been recycled three times exhibited a partial matrix deformation, crack formation occurred, and more voids were seen, compared to smoother polymer phases seen for composites obtained through the first and second recycling cycles. Despite these, it can also be assumed that a sufficient fiber–matrix adhesion has taken placed.

3.3 Melt flow property of recycled PA66-GF30 composites

The influence of incorporating the recycled material obtained in 3 different recycling cycles on the melt flow property of PA66-GF30 composite was studied by MFI analysis, which is a measure of melt fluidity or viscosity. The MFI values obtained as a function of the recycling cycle and recycled material ratio incorporated into the virgin composite are given in Figure 6. As can be seen, the obtained MFI values clearly revealed that the melt flow behavior of the PA66 composite was significantly affected by the applied mechanical recycling process,

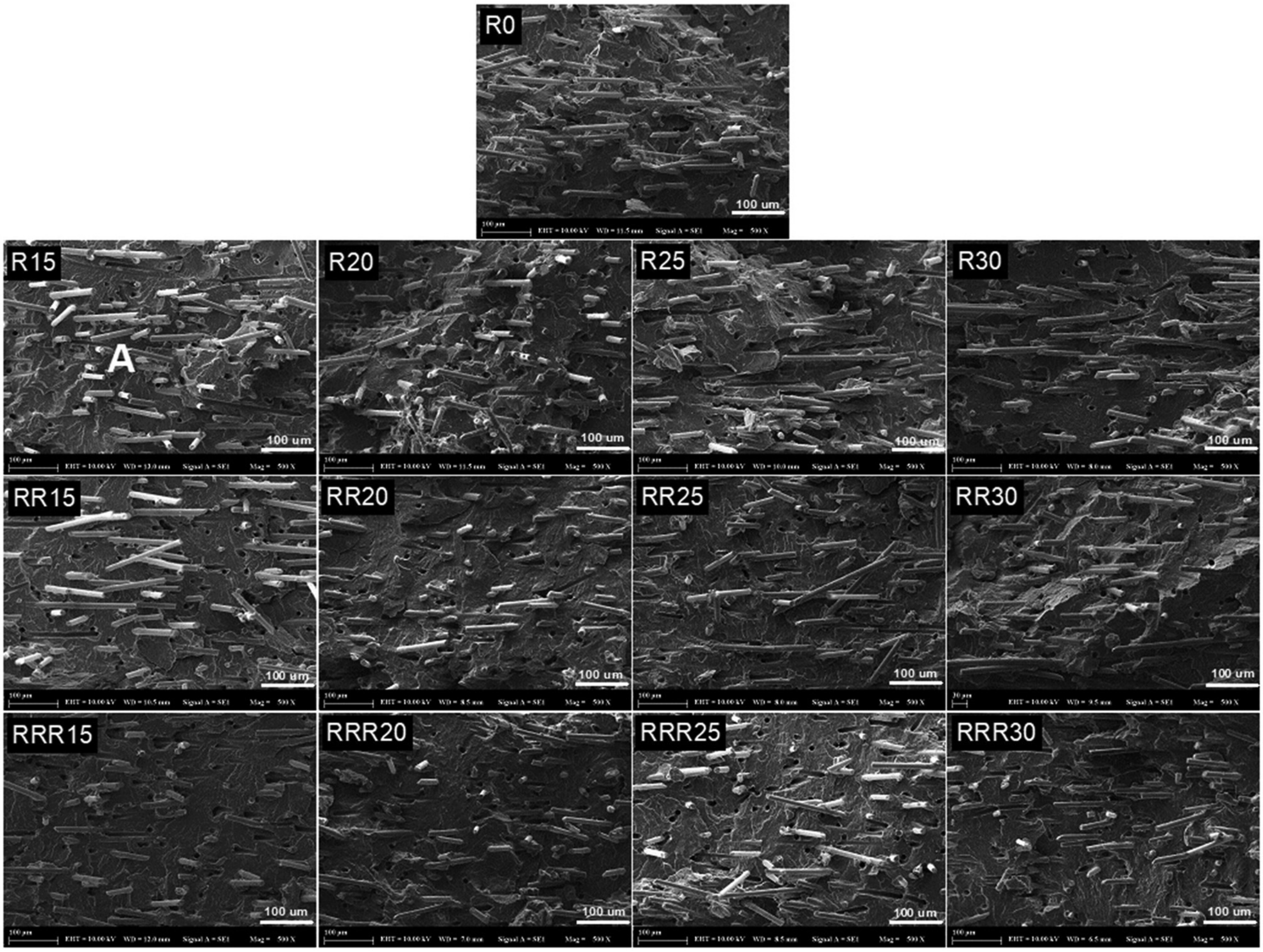

Figure 5: SEM micrographs of fractured surfaces of tensile test samples of the virgin and recycled PA66-GF30 composites (500× magnification).

the recycled material content in the composite, and the number of recycling cycles.

The MFI value of the virgin sample (R0) was 22.73 g·min^{-1}. When the PA66-GF30 composite was reinforced with the recycled material subjected to only the first recycling cycle at a ratio of 15 to 25 wt% (R15–R25), it was found that the MFI values of these samples were lower than those of the virgin sample. The same behavior was observed in composites consisting of 15–20 wt% recycled material subjected to the 2nd and 3rd times recycling (RR15-20, RRR15-20). The highest decrease in MFI values of approximately 9% was observed for R15 and R20 samples. This is often unexpected behavior for thermoplastics that have been mechanically reprocessed many times. However, the melt flow behavior of glass fiber–reinforced thermoplastics is controlled more by the incorporated glass fiber rather than by the matrix properties, and the amount and length of the

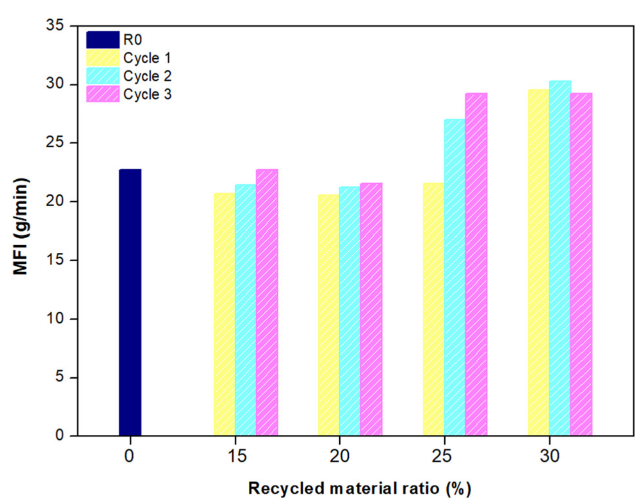

Figure 6: MFI test results of the virgin and recycled PA66-GF30 composites.

fibers affect the rheological properties of these composites. (30,55,56). The observed decrease in MFI values, in other words, the increase in melt viscosity indicates that the glass fibers effectively restrict the chain mobility of PA66 as a result of ensuring efficient fiber length and its distribution after the applied recycling processes (39,57). For these composites, the increased surface area with fiber breakage led to higher interfacial interactions between fibers and polymer chains, increasing the resistance to flow (57,58).

On the other hand, the use of 25 wt% of the recycled material obtained in the second and third recycling cycles and increasing this ratio to 30 wt% for all cycles resulted in a remarkable increase in MFI values. This can be summarized as the higher ratios of the recycled material subjected to the higher number of recycling cycles in the composite further increased the MFI value compared to the virgin and other recycled composites. The MFI value was 30.31 $g{\cdot}min^{-1}$ for the RR30 sample, and this was the highest increase of 33%. In the reprocessed fiber–reinforced thermoplastic composites, the increment in the MFI values, i.e., decrease in melt viscosity, is due to the thermomechanical degradation of polymer matrix during the recycling process as well as fiber shortening. As a result of repeated melt processing and regrinding cycles, polymer chain scissions can occur, and therefore, the molecular weight is reduced, increasing the MFI value (48,59). In addition, the presence of very short fibers with the increasing number of recycling cycles and recycled material content in the composite reduces their resistance to the flow of the matrix and the viscosity decreases. However, with repeated recycling processes, very short fibers are less likely to break again, and the degradation of the material becomes less and less important (39). This effect can also explain why the MFI value of the recycled composite, RRR30, was not much different. In addition, the observed change in MFI test results can be confirmed by SEM and optical microscope analysis findings.

3.4 Mechanical properties of recycled PA66-GF30 composites

The tensile test was applied to investigate the effect of the number of recycling cycles and recycled material content on the tensile strength, elastic modulus, and elongation at the break of PA66-GF30 composite, and the obtained results are displayed in Figure 7(a)–(c). For the virgin PA66-GF30, the tensile strength, elastic modulus, and elongation at break values were 107.6 MPa, 3.0 GPa, and 6.1%, respectively.

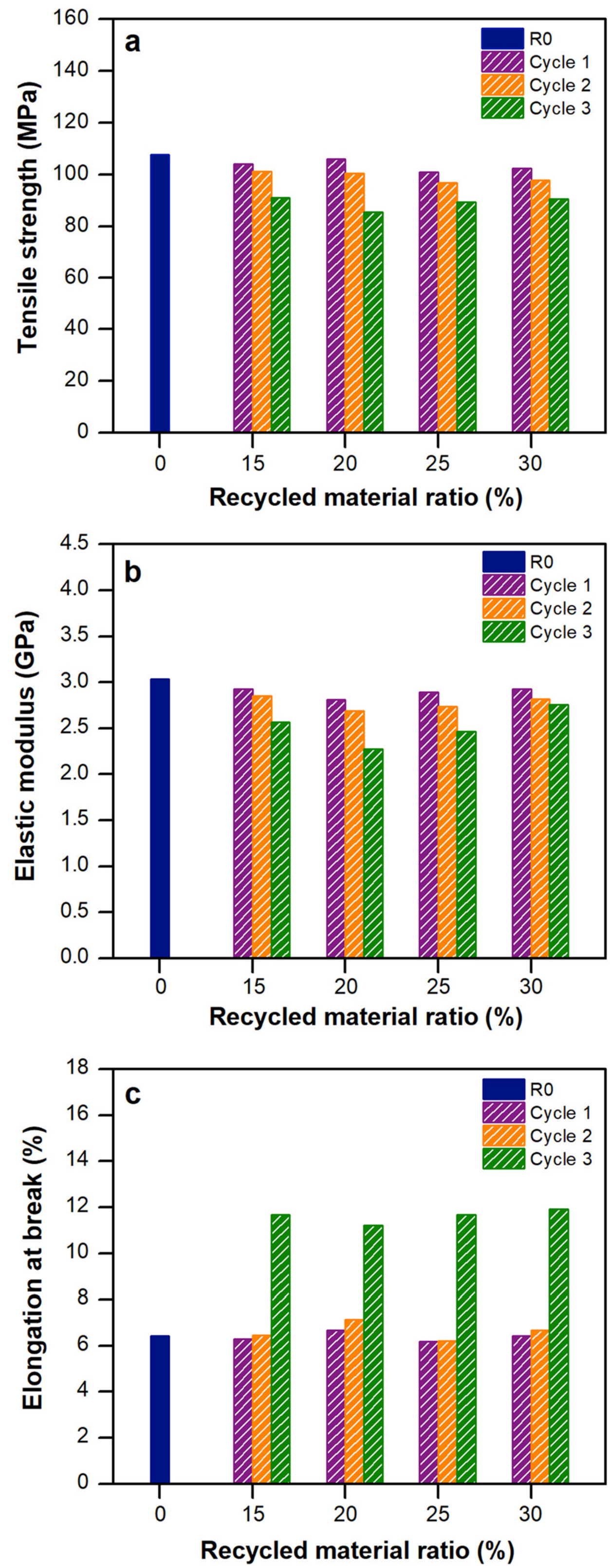

Figure 7: (a) Tensile strength, (b) elastic modulus, and (c) elongation at break results of the virgin and recycled PA66-GF30 composites.

With the incorporation of various ratios of recycled material subjected to different recycling cycles into the virgin sample, it was observed that the number of recycling cycles played an essential role in the tensile properties of the PA66-GF30 composite. As the number of recycling cycles increased, the tensile strength and elastic modulus values decreased while the elongation at break value increased. On the other hand, increasing the recycled material content in the composite at each recycling cycle slightly affected these properties.

The tensile strength values of the recycled composites subjected to the first, second, and third recycling cycles decreased by an average of 4.0%, 7.2% and 17.2%, respectively, compared with the R0 virgin sample (Figure 7a). For each cycle, the values varied with increasing recycled material content. In addition, the elastic modulus exhibited the same declining behavior as in the tensile strength after each recycling cycle (Figure 7b). The lowest values for both tensile strength and elastic modulus were 85.3 MPa and 2.28 GPa for the RRR20 sample. Furthermore, the fact that the composites contained the recycled material subjected to the first and second recycling cycles caused no notable change in the elongation at break values compared to the virgin composite. However, the elongation at break values of the composites obtained with the third recycling cycle changed significantly, with an increase of approximately 90% (Figure 7c).

To achieve an enhanced mechanical performance in the fiber-reinforced thermoplastic composites, the applied stress should be transferred from the matrix to the fibers. In these composites, the change in mechanical properties mainly depends on the fiber length, fiber length distribution, fiber orientation, and interfacial strength (60). For the mechanically recycled fiber-reinforced composites, Eriksson et al. reported that the potential effects of recycling on the fiber orientation, interfacial strength, and short-term matrix performance are small, and thus, the decrease in tensile properties as a function of recycling is almost entirely due to fiber shortening induced by the reprocessing. The fiber length should be as much as possible above the critical fiber length to ensure sufficient stress transfer (32). Also, Bernasconi et al. revealed that fibers make different contributions to tensile strength depending on their length distribution, and mainly, an increase in the number of fibers of subcritical length causes a decrease in tensile strength rather than a decrease in the average fiber length (37). Another study confirmed that increasing subcritical fiber length decreases the interfacial adhesion strength and thus the composite tensile strength (61). Moreover, Evens and co-workers reported that when the fibers shortened by mechanical recycling are of subcritical length, the fibers slip out of the matrix and the fiber pullout failure mechanism occurs, increasing strain at break (52). Kuram et al. explained the increase in the strain at the break of the carbon fiber–-reinforced composites seen after five reprocessing cycles by the poor interfacial adhesion between fibers and matrix and by the increasing mobility of fibers due to the easier debonding.

As can be clearly seen in Figure 7(a)–(c), the recycled composites produced by the first and second recycling cycles exhibited a much smaller reduction in the tensile properties compared with those of the virgin PA66-GF30. This behavior of these composites agrees with the results obtained from their microstructural and melt flow analyses. These composites had good interfacial adhesion and no matrix deformation except for some voids, and their average fiber length ranged from about 300–200 μm, resulting in satisfactory tensile properties. On the other hand, the worsening of the tensile properties that appeared after the third cycle is related to the shortening of the fibers. In the recycled composites obtained in this cycle (RRR15-30), the average fiber length shifted to the lower value in the 100–200 μm range, which is even below the critical value. The critical length for glass fibers in PA66 is known to be in the region of 180–230 μm (32). The subcritical length of most of the fibers in these composites is not sufficient in reinforcing the matrix and can no longer impart the same stiffness to the matrix, the ends of the fibers cannot fully withstand tensile stress, therefore their tensile strength and elastic modulus were reduced (39,51). As expected, this fact can also explain the significant increment of the elongation at break values. With the increase in fibers of subcritical length, fiber pullout easily occurred, resulting in voids and crack formation, and thus, an increase in elongation at break was observed.

The Charpy notch impact test was conducted to determine the amount of energy that the composite materials obtained as a function of the recycling cycle and the ratio of recycled material added to the virgin PA66-GF30 material can absorb under a dynamic load. The obtained impact energy values are shown in Figure 8. The impact energy value was found to be 0.19 J for the virgin R0 composite material. All recycled composites had lower values of the impact energy, regardless of the number of recycling cycles and the recycled material content. The increasing number of fiber ends can explain this decrease as a result of fiber breakage from reprocessing. An applied load causes stress concentration at the ends of fibers, which may initiate the formation of a matrix crack as the first failure stage before destructive crack propagation occurs (32). However, the amounts of impact energy absorbed by the recycled composites exhibited an increasing inclination with the rising cycle number. A similar behavior was also revealed, with

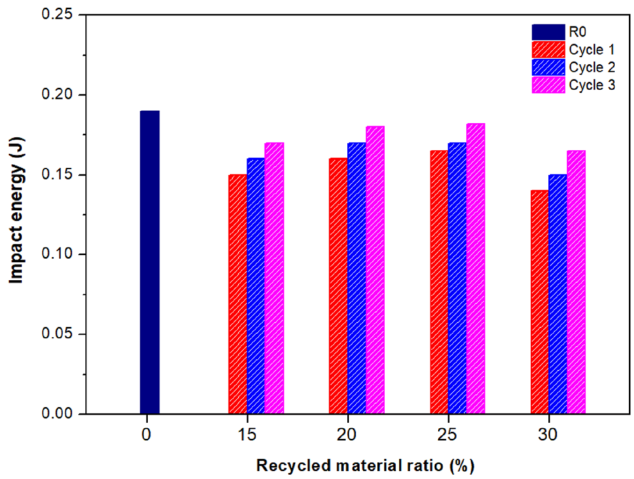

Figure 8: Charpy impact energy values of the virgin and recycled PA66-GF30 composites.

the recycled material content in the composite increasing by up to 25% at each recycling cycle. This result may be due to the increase in the number of shorter fibers broken with each recycling cycle and the broadening of the short fiber length distributions with the increasing amount of recycled material in the composite. Hassan et al. showed that shorter fibers tend to have more ductile fracture due to the fiber pullout mechanism and thus provide more fracture energy to the composite during crack propagation (62). In crack propagation, since fibers of subcritical length are pulled out from the matrix instead of breaking, the fiber pullout mechanism increases with the decrease of fiber length, resulting in an increase in fracture energy (32). Then, the values decreased with the addition of recycled material of 30 wt% in each cycle, and the lowest impact energy value was observed for the R30 sample, with a reduction of about 26% compared to the virgin R0 sample. In many studies, the decrease in impact energy after the critical fiber length is reached has been attributed to the matrix degradation that occurred as chain scission. With this degradation, the shortening of the polymer chains and broadening distribution of the chain lengths lead to loss of impact strength (40,48,58,63). Moreover, the higher MFI values obtained attributing the thermomechanical degradation of polymer matrix and the heterogeneous dispersion of the fibers observed in SEM images indicating poor fiber–matrix adhesion seem to be consistent with the lower impact energy results for these samples (R30, RR30, and RRR30).

In addition, the Shore D hardness of the composites was examined. For the virgin R0 sample, the hardness value was found to be 83. The values were slightly lower and varied between 81 and 83 for the recycled composites, regardless of the recycling cycle and the recycled material content. This may be due to the shortening of the glass fibers in the composite, which are stiffer than the matrix, from the recycling processes.

4 Conclusions

The recycling and reuse of thermoplastic wastes generated during production is important regarding the environment, economy, resource conservation, and sustainability. Also, great importance is attached to the efficient inclusion of these wastes into production without losing the final properties of the products. This study investigated the effective recyclability of wastes generated during the production of 30 wt% SGFR PA66 composite (PA66-GF30) products used as engine fasteners in the automotive industry. PA66-GF30 injection molded composite products were mechanically recycled, consisting of regranulation and reinjection molding steps, respectively. PA66-GF30 was subjected to the three mechanical recycling cycles, and the recycled materials obtained in each of these cycles were incorporated 15, 20, 25, and 30 wt% ratios to the virgin composite.

The effects of the recycling cycle and recycled material content on the final properties of PA66-GF30 products were extensively investigated by thermal, microstructural, melt flow and mechanical analyses. TGA analysis showed that the number of recycling cycles and recycled material content in the composite had no significant change in the thermal stability of the PA66-GF30 composite. The incorporation of recycled materials into the virgin PA66-GF30 resulted in a slight decrease in the degree of crystallinity, as well as the T_m and ΔH_m values, independent of the number of recycling cycles and recycled material content. With mechanical recycling, the fiber shortening occurred, and the average fiber length shifted to lower values of 150–250 µm, with the increasing number of recycling cycles and the recycled material content, compared with that of virgin composite, 300–350 µm. SEM analysis showed that the homogeneous dispersion of fibers was lost in some regions, and a partial matrix deformation appeared when the recycled material content in the composite exceeded 25 wt%, and the recycling cycle was applied three times. It was observed that the mechanical and rheological behaviors of the composite changed, especially under certain recycling conditions. For the sample obtained by the recycled ratio of 25 wt% in the 3rd cycle (RRR25), tensile strength, elastic modulus, and impact energy decreased by 17.0%, 28.7%, and 4.2%, respectively, while the elongation at break and

MFI increased by 91.9% and 28.6%, respectively. On the other hand, the recycled composites obtained in the first and second recycling cycles and containing up to 25 wt% recycled material showed similar properties with those of the virgin composite. Consequently, the findings of these investigations and the obtained properties of many recycled materials have been promising in terms of developing a more efficient and effective recycling of PA66-GF30 wastes generated during production and the potential to use these recycled materials as end products in automotive components.

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