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Mechanical, Chemical, Thermal, and Rheological Properties of Recycled PA6/ABS Binary and PA6/PA66/ABS Ternary Blends

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ABSTRACT: The effects of multiple injection molding cycles on the chemical and mechanical properties of PA6/ABS and PA6/PA66/ ABS blends are investigated. The chemical structures of both PA6/ABS binary and PA6/PA66/ABS ternary blends do not alter after recycling process. For PA6/ABS binary blend, it is found that the tensile strength, strain at break, elastic modulus, impact strength, flexural strength, and modulus of recycled blend decrease by 6.49%, 15.19%, 21.00%, 9.41%, 7.09%, and 8.25%, respectively, while MFI increases by 23.59% as compared with the virgin blend. After five recycling process for PA6/PA66/ABS ternary blend, the tensile strength, strain at break, and impact strength of recycled blend decrease by 18.00%, 50.80%, and 87.27%, respectively. However, flexural strength and modulus of PA6/PA66/ABS blend increase slightly. For virgin PA6/PA66/ABS blend, MFI value was 7.7 g/10 min and with recycling this value showed an important increase to 31.56 g/10 min after five cycles. © 2014 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2014**, *131*, 40810.

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INTRODUCTION

Polyamide 6 (PA6), polyamide 66 (PA66), and acrylonitrilebutadiene-styrene (ABS) are extensively employed commercial polymers and each of them has some superior properties providing these polymers for using different application areas. Polyamide (PA) is a semicrystalline polymer and has good chemical resistance and abrasion resistance. However, PA has poor dimensional stability and high cost. There are many types of PA such as PA6, PA12, and PA66. PA6 has a molecular chain made of a base unit with six carbon atoms and is synthesized by ringopening polymerization of *ɛ*-caprolactam monomer. PA6 has good chemical resistance, good abrasion resistance and mechanical properties however, possesses dimensional instability and high water affinity. PA6 is employed in the production of automobile parts and textile fibres. PA66 is a semicrystalline polyamide and is used in bearings and gears owing to its good abrasion resistance and self-lubricating properties.¹ PA66 is extensively used in engineering applications since its high mechanical properties and good processability.² ABS consists of an amorphous phase of styrene-acrylonitrile copolymer (SAN) and a polybutadiene (PB) rubber phase. ABS is processed easily, has low cost and has high impact and chemical resistance. However, the recycling of ABS polymer is difficult since oxidative degradation of the butadiene rubber phase during service time,

decreasing its impact strength that is its most important property.³ Therefore, ABS polymer is blended with polymers having a good mechanical and thermal resistance like PA to recycle.⁴ ABS is extensively used in the automotive industry and electronic equipments.

Every polymer has not only superior properties but also some drawbacks therefore undesirable influence of polymers can be eliminated by the blending of polymers with each other. Polymer blends is one of the cheapest method to design new polymeric materials and they meet the desirable properties that cannot be provided by the currently available commercial polymers. Therefore, the purpose of PA6/ABS blends is to improve the properties with respect to the neat polymers. PA6/ABS binary blend was characterized by high impact strength, good chemical resistance, and excellent flow properties.⁵

Properties of plastics such as lightness, easiness to shape, resistance to corrosion, low cost, and electrically insulating result in higher demand for plastic materials and consequently the consumption of plastic is increasing. The increasing of the plastic consumption induces the plastic waste. It is known that plastic materials are nondegradable and the breakdown of plastic takes a long time, up to hundreds of years.⁶ Therefore, the environment is affected negatively by the plastic waste. In recent years, growing environmental awareness has resulted in new approaches in the

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Table I. Extrusion Conditions for Blending

Screw rotation speed	rpm	350
Die temperature	°C	220-260

use of plastic materials. The use of recycled plastic is one of the effective way⁷ to reduce the negative effect of plastic on environment. Some authors have investigated the effect of recycling on the mechanical, thermal and/or rheological properties of PA6,^{8–14} PA66,^{15–18} and ABS^{19–21} polymers. Virgin and recycled ABS/PA6 blends were prepared with the addition of the compatibilizer and a core-shell rubber impact modifier.²² The recycled materials were taken from dismantled cars²² and the number of recycling was not considered. Not only virgin form of PA6/PA66/ABS ternary blend but also recycling of PA6/PA66/ABS ternary blend were not investigated. PA6/ABS binary and PA6/PA66/ABS ternary blends are used especially in the automotive industry for exterior components such as wheel covers.

The aim of this study was to determine the effect of recycling process of PA6/ABS binary and PA6/PA66/ABS ternary blends on the mechanical, chemical, thermal, and rheological properties. In the literature, recycled PA6/ABS binary blend was used²²; however, the number of recycling was not taken into consideration. Therefore, this is the first study investigating the influence of multiple recycling process for PA6/ABS binary and PA6/PA66/ABS ternary blends.

EXPERIMENTAL

Materials and Process Conditions

PA6, PA66, and ABS polymers were provided by Volgamid, Radici, and Dow companies, respectively. Prior to injection molding process, PA6, and ABS polymers were extruded to obtain PA6/ ABS binary blend and PA6, PA66, and ABS polymers were extruded to obtain PA6/PA66/ABS ternary blend according to the conditions given in Table I. Compositions of PA6/ABS binary blends were 40/40 (wt/wt) and 20 wt % linear triblock copolymer compatilibizer (Kraton) was added to PA6/ABS binary blends. The percentage weight of PA6, PA66, and ABS polymers in ternary blends was the same and 20 wt % linear triblock copolymer compatilibizer (Kraton) was added to PA6/ PA66/ABS ternary blends.

Before injection molding process, PA6/ABS binary and PA6/ PA66/ABS ternary blends were dried in order to eliminate air bubbles occurring during the molding process. PA6/ABS binary blend was dried at 100°C for 2 h and PA6/PA66/ABS ternary blend was dried at 90°C for 2 h. A plastic injection machine (YIZUMI-UN90A2) which had a maximum clamping force of 900 kN and an injection pressure of 222 MPa was employed to mold test samples. A four-cavity mold (double cavity for tensile samples, one cavity for impact sample and one cavity for flexural sample) manufactured by using computer numerical control (CNC) machine was utilized to produce tensile, impact, and flexural samples. Injection molding conditions were kept constant during the experiments (Table II). PA6/ABS binary and PA6/PA66/ABS ternary blends were recycled five times to determine the effect of the recycling number on the chemical, mechanical, and rheological properties. First, virgin binary and ternary blends were molded and a lot of samples were produced. From these samples, five samples were employed for the tensile, impact, and flexural tests of virgin PA6/ABS binary and PA6/PA66/ABS ternary blends. After that, the remaining plastic was shredded into small pieces for a new injection cycle and reground plastic was molded again to produce 1st recycled sample. These procedures were repeated to manufacture 2nd, 3rd, 4th, and 5th recycled samples.

Measurements

In this study, the effect of recycling number on PA6/ABS binary and PA6/PA66/ABS ternary blends was investigated by measuring chemical, mechanical, and rheological properties. The change in the chemical structure of binary and ternary blends during multiple recycling process was determined by fourier transform infrared (FTIR) spectroscopy. The infrared spectra of the virgin and recycled binary and ternary blends were taken on a Perkin-Elmer Spectrum 100 spectrophotometer with an attenuated total reflectance (ATR) objective and the samples achieved from the injection molded tensile specimens were utilized in the tests. To determine the miscibility and melting behavior of binary and ternary blends differential scanning calorimetry (DSC) was conducted by using a Mettler-Toledo DSC 822 calorimeter. Each blend was heated from room temperature to 275°C at a rate of 10°C/min under an argon atmosphere and held for 5 min to erase the thermal history, then cooled to 25°C at 10°C min⁻¹, and finally heated to 275°C at 10°C min⁻¹. Thermal gravimetric analysis (TGA) was utilized to determine the degradation behavior and thermal stability of binary and ternary blends by using a Mettler-Toledo TGA/STDA 851 thermogravimetric analyzer with a heating rate of 20°C min⁻¹ from room temperature to 700°C. The fractured surfaces of the samples were examined by employing scanning electron microscopy (SEM, Philips XL30 SFEG). SEM micrographs were taken at 1000 \times magnification.

Tensile, impact, and flexural test specimens were molded in accordance with ISO 527,²³ ISO 180,²⁴ and ISO 178²⁵ standards, respectively. Tensile test specimen was the shape of dog bone and dimensions were 175 mm length, 20 mm width, and 4 mm thickness. Impact specimens were molded in the form of prismatic and dimensions were 80 mm length, 10 mm width and 4 mm thickness, then notch (2 × 45°) was produced at the impact test

 Table II. Injection Molding Conditions for PA6/ABS Binary and PA6/ PA66/ABS Ternary Blends

Injection parameters	Units	PA6/ABS	PA6/PA66/ABS
Melt temperature	°C	250	250
Mold temperature	°C	45	60
Injection pressure	bar	80-85	50
Holding pressure	bar	8	35
Injection speed	mm/s	50-60	30
Holding time	S	0.7	3.5
Cooling time	S	25	25





Figure 1. FTIR spectra of virgin and recycled specimens of (a) PA6/ABS binary and (b) PA6/PA66/ABS ternary blends. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

samples for PA6/ABS binary blend. INSTRON 5569 testing device was employed to conduct the tensile and three-point flexural tests. 50 mm min⁻¹ and 5 mm min⁻¹ tensile test speeds were used for PA6/ABS binary and PA6/PA66/ABS ternary blends, respectively. Three-point flexural test for both PA6/ABS binary and PA6/PA66/ABS ternary blends was carried out at the speed of 10 mm min⁻¹. The impact test was carried out by using INS-TRON Ceast 9050 testing device. Samples with notched were employed for impact tests of PA6/ABS binary blend while unnotched impact samples were used for PA6/PA66/ABS ternary blend. 2.75 J and 5.5 J of the impact hammers were utilized for PA6/ABS binary and PA6/PA66/ABS ternary blends, respectively. All mechanical tests were repeated five times and average of these values were employed for further analysis.

Melt flow index (MFI) of both PA6/ABS binary and PA6/PA66/ ABS ternary blends was measured by using a Devotrans-EA3 testing device in accordance with ISO 1133 standard.²⁶ Samples for MFI measurements were obtained by crushing the molded plastics. In MFI tests, PA6/ABS binary blend was preheated at 250°C and standard weight (5 kg) was applied onto the piston; however, temperature of 275°C was used for PA6/PA66/ABS ternary blend. The measurements were repeated five times and the average value was taken.

RESULTS AND DISCUSSION

Physical and Chemical Structures

FTIR results for PA6/ABS binary and PA6/PA66/ABS ternary blends are given in Figure 1(a,b), respectively. From FTIR results, it was concluded that the characteristic bands of both binary and ternary blends showed no significant change even after five recycling process. No difference between spectra of recycled and virgin binary and ternary blends implied that the chemical nature of PA6/ABS binary and PA6/PA66/ABS ternary blends remained unchanged with multiple recycling process and recycled samples did not suffer from any significant degradation. No difference of chemical nature with recycling process was also reported other polymers or polymer blends such as PA6.²⁷



Figure 2. DSC curves for virgin and recycled specimens of (a) PA6/ABS binary and (b) PA6/PA66/ABS ternary blends. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]



Figure 3. TGA curves for virgin and recycled specimens of (a) PA6/ABS binary and (b) PA6/PA66/ABS ternary blends. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

The peaks in the range of $2800-3150 \text{ cm}^{-1}$ were assigned to C—H stretching bands of the polymers [Figure 1(a)]. The characteristic peaks at 3300 cm⁻¹, 1635 cm⁻¹, and 1542 cm⁻¹ represents N—H, C=O (amide I band), and C(O)NH stretching vibration (amide II band) of PA6 polymer, respectively.²⁸

DSC results for PA6/ABS binary and PA6/PA66/ABS ternary blends are depicted in Figure 2(a,b), respectively. Melting temperature of PA6 and the glass transition temperature of ABS were found as about 220°C and 110°C, respectively [Figure 2(a)] and Goitisolo et al.¹³ found the temperatures of PA6 and ABS as 220°C and 106°C, respectively. For PA6/ABS binary blends, the positions of the peaks were not shifted to lower or higher temperature with the recycling process.

Melting peaks around 220°C and 260°C were observed for PA6 and PA66 polymers, respectively³ and the glass transition temperature of ABS was found as 100°C from DSC measurements.²⁹ The glass transition temperature of ABS was found as about 108°C, melting temperatures of PA6 and PA66 were found as 210°C and 255°C, respectively from DSC graphs of PA6/PA66/ABS ternary blend [Figure 2(b)].

The thermal stabilities of PA6/ABS binary and PA6/PA66/ABS ternary blends are shown in Figure 3(a,b), respectively. For PA6/

ABS binary blends, the main degradation started at \sim 360°C and finished at about 490°C [Figure 3(a)]. The degradation of virgin and recycled PA6/ABS binary blends was ended in a single step. It was also observed that TGA curves of all recycled samples were not significantly different from virgin samples. However, carbonaceous residues were found to be different. For PA6/ABS binary blends, carbonaceous residue was between 1.48 and 4.23% at the end of the degradation process at 700°C.

For PA6/PA66/ABS ternary blends, the main degradation started at about 370°C and ended in a single step at about 490°C [Figure 3(b)]. For PA6/PA66/ABS ternary blends, carbonaceous residue was between 1.38 and 1.97% except for R3 sample. For R3 sample, carbonaceous residue was 6.74%.

The fractured surfaces of virgin and recycled samples were examined by using SEM at a magnification of $1000 \times$ and SEM micrographs are depicted in Figure 4. Fibrillar morphology was seen in the structure of virgin and recycled PA6/ABS binary blends. Fibrillar structure was observed with PA6 polymer³⁰ and ABS/PA6 blend^{22,31} and the mechanism for generating this fibrillar structures explained as follows:^{22,31} PA6 phase initially had a certain continuity, it did not fracture together with ABS during the slow crack propagation, then it was elongated and pulled out from ABS phase, leaving fibrils in the fracture surface. For PA6/ABS binary blend, it was found that the morphology of fracture surface did not significantly change by increasing recycling process.

SEM micrographs of virgin PA6/PA66/ABS ternary blend showed a smooth fracture surface with few holes caused by particles detachment. This indicated that there was low adhesion between phases. However, a good distribution of the dispersed particles was observed. A smooth fracture surface was also seen for ABS polymer in the literature.³² Increasing recycling number, holes became larger and a slightly more heterogeneous distribution of the dispersed particle was observed. Larger particles of the dispersed phase with irregular shapes, in many cases elongated were observed at the fracture surface of 5th recycled PA6/PA66/ABS ternary blend. The roughness of the fracture surface increased in 5th recycled PA6/PA66/ABS ternary blend due to plastically deformed rubber particles and this surface was typical for rubber toughened crazing polymers like ABS.⁵

Mechanical and Rheological Properties

Tensile strength and strain at break values obtained from tensile tests are given in Figure 5 for both PA6/ABS binary and PA6/PA66/ABS ternary blends as a function of recycling number. For PA6/ABS binary blend, tensile strength did not change significantly by the multiple recycling process and a slight decrease was observed with recycling. The same behavior was found for tensile strength of PA6 polymer as a function recycling process, showing a slight drop in tensile strength value.¹⁴ The recycling process induced a decrease in the tensile strength of PA6/PA66/ABS ternary blends. For PA6/PA66/ABS ternary blend, there was a decrease of 18% in the tensile strength after five recycling process. The decrease in the tensile strength with increasing recycling number was due to the decrease in the molecular weights. It was found that the deterioration at the tensile strength for PA6/PA66/ABS ternary blend was much higher than that for





Figure 4. SEM micrographs for fracture surfaces of PA6/ABS binary and PA6/PA66/ABS ternary blends (at ×1000 magnifications).

PA6/ABS binary blend after five recycling process. For PA6 polymer, a decrease of 28% in the tensile strength was found after 3 recycling process in the literature.¹¹ The decrease in the tensile strength with recycling was found for also PA66^{15,33} and ABS³⁴ polymers.

It was also found that the tensile strength of PA6/ABS binary blends increased with the addition of PA66 polymer, similar to that found other polymer blends.³⁵ It was reported that an appropriate content of PA66 in polymer blends resulted in a continue PA66 phase and the polymer blends displayed a co-continue morphology. Stress could be transmitted effectively



Figure 5. Influence of the number of processing cycles on the tensile strength and strain at break for PA6/ABS binary and PA6/PA66/ABS ternary blends. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

and as a result, the mechanical performance like tensile strength improved. $^{\rm 36}$

Strain at break values of both PA6/ABS binary and PA6/PA66/ ABS ternary blends decreased with recycling process (Figure 5). This meant that the ductility of both PA6/ABS binary and PA6/ PA66/ABS ternary blends decreased after five injection cycles. It was reported that virgin ABS polymer was more ductile than that of recycled ABS.³⁷ For PA6/ABS binary blend, 15.19% decrement in the strain at break after five recycling process was found. Strain at break decreased by 50.80% with increasing recycling process for PA6/PA66/ABS ternary blend. Virgin PA6/ PA66/ABS ternary blend presented an important decrease in strain at break and this proved an important drop in molecular weight phenomenon as a result of degradation. It was known that recycling process resulted in a continuous thermomechanical degradation caused by chain scissions.³⁸ The strain at break results were in good agreement with the literature related to the recycling of PA6^{10,13} and ABS polymers.³⁴

The addition of PA66 in PA6/ABS binary blend decreased the strain at break values and it was reported that the stiffening effect provided by PA66 induced a significant decrease of the strain at break values.¹

The elastic modulus of PA6/ABS binary and PA6/PA66/ABS ternary blends versus the number of processing cycles is shown in Figure 6. The elastic modulus of PA6/ABS binary blend decreased by 21% after five recycling process. The decrease in elastic modulus might be due to the decrease in molecular weight induced by recycling process. The decrease in the elastic modulus indicated that the stiffness of PA6/ABS binary blend deteriorated with the recycling process. For PA6 polymer, a



Figure 6. Influence of the number of processing cycles on the elastic modulus for PA6/ABS binary and PA6/PA66/ABS ternary blends. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

decrease of 11% in the elastic modulus was found after 3 recycling process.¹¹ The decrease in elastic modulus of PA66¹⁵ and ABS³⁴ polymers was found with recycling process due to the chain scission. The elastic modulus of PA6/PA66/ABS ternary blend increased after 1st recycling process then decreased with increasing recycling process. However, 5th recycled PA6/PA66/ ABS ternary blend gave higher elastic modulus value than that of virgin PA6/PA66/ABS ternary blend.

Elastic modulus of PA6/ABS binary blend increased with the addition of PA66 polymer and the increment in elastic modulus with the addition of PA66 polymer was observed for other polymers.^{1,39} Granado et al.³⁹ explained the increment in elastic modulus of PC/PA66 blends with highly fibrillar morphology of the dispersed phase and the significant decrease in specific volume.

Figure 7 shows the effect of the number of processing cycles on the impact strength for PA6/ABS binary and PA6/PA66/ABS ter-



Figure 7. Influence of the number of processing cycles on the impact strength for PA6/ABS binary and PA6/PA66/ABS ternary blends. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

nary blends. Impact strength of PA6/ABS binary blend did not show any significant change with recycling process. The decrement in impact strength for PA6/PA66/ABS ternary blend was found to be 87.27% after five reprocessing cycles. The reduction of impact strength with recycling process could be owing to the drop of molecular weight of PA6/PA66/ABS ternary blend. The drop in impact strength with recycling process was explained with the behavior of molecular chain scission. Shorter molecular chain and broader chain length distribution resulted in poor chain entanglements, consequently the impact strength decreased with recycling.^{12,14} It was found that the impact strength of PA6 polymer showed a decrease of 16% after three recycling process.¹¹ The impact strength of PA66³³ and ABS^{19,34} polymers decreased with increasing number of recycling due to the thermomechanical degradation.

The addition of PA66 into PA6/ABS binary blend resulted in an increment of impact strength up to 3rd recycling process and then it induced a decrement of impact strength values for 4th and 5th recycled samples. It was reported that the addition of PA66 to PP polymer increased the impact strength.⁴⁰

The influence of the number of processing on flexural strength and modulus for PA6/ABS binary and PA6/PA66/ABS ternary blends is plotted as depicted in Figure 8. Flexural strength and modulus of PA6/ABS binary blend decreased with increasing recycling process. Flexural modulus of virgin PA6/ABS binary blend decreased from about 194 MPa to 178 MPa as the injection molding cycle increased. Flexural modulus of ABS polymer decreased with recycling process.³⁴ Flexural strength and modulus of PA6/PA66/ABS ternary blend slightly increased with recycling process and similar increment was found for PA6 polymer.¹²

Flexural strength of ternary blends was higher than that of binary blends. This behavior could be attributed to the effect of the third component (PA66). Similar to our observation, it was found that the addition of PA66 to PP polymer increased the flexural strength.⁴⁰



Figure 8. Influence of the number of processing cycles on the flexural strength and modulus for PA6/ABS binary and PA6/PA66/ABS ternary blends. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]



Figure 9. Influence of the number of processing cycles on the MFI for PA6/ABS binary and PA6/PA66/ABS ternary blends. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Flexural modulus of PA6/ABS binary blend increased with the addition of PA66 polymer and similar increment in flexural modulus with the addition of PA66 polymer was observed for other polymer in the literature.⁴¹

MFI results for PA6/ABS binary and PA6/PA66/ABS ternary blends are given as a function of the number of processing cycles as shown in Figure 9. Generally, it was found that MFI values of both PA6/ABS binary and PA6/PA66/ABS ternary blends increased with the recycling process in accordance with other studies about recycling of PA6.12 The increment in MFI values was due to the fact that polymer chain lengths reduced during recycling process and this reduction induced a decrease in the molecular weight of the polymer. MFI values of PA6/ABS binary blend increased a total of 23.59% after five injection cycles. For virgin PA6/PA66/ABS ternary blend, MFI value was 7.7 g/10 min and with recycling this value showed an important increase to 31.56 g/10 min after five injection cycles. The increase of the MFI with recycling was observed for ABS polymer and this result might be attributed to the consumption of antioxidants and stabilizers during recycling process.³⁴

The addition of PA66 into PA6/ABS binary blend resulted in a decrement of MFI values up to 3rd recycling process, then it induced an increment of MFI values for 4th and 5th recycled samples.

CONCLUSIONS

This study was carried out to investigate the recycling possibility of PA6/ABS binary and PA6/PA66/ABS ternary blends. To determine the recycling performance of these binary and ternary blends, virgin blends were recycled at five times and the effect of the number of recycling on the mechanical, chemical, thermal, and rheological properties was investigated. FTIR results showed that the chemical nature of these binary and ternary blends did not change with multiple recycling. From DSC analysis it was concluded that melting temperatures did not significantly change with increasing recycling process. It was observed that all TGA curves of the virgin and recycled blends were close to each other.

In general, tensile strength, strain at break, elastic modulus, flexural strength, and flexural modulus of PA6/ABS binary blend decreased as a function of the number of recycling, while MFI value increased. Impact strength of PA6/ABS binary blend did not show any significant change with multiple recycling process. It was concluded that the recycled PA6/PA66/ABS ternary blends gave lower tensile strength, strain at break, and impact strength than virgin ternary blends. Flexural strength, flexural modulus, and MFI of PA6/PA66/ABS ternary blend increased with recycling process. From the mechanical results, it could be concluded that both PA6/ABS binary and PA6/PA66/ABS ternary blends did not suffer any significant deterioration in mechanical properties except for impact strength of PA6/PA66/ABS ternary blend.

It was also found that the tensile strength, elastic modulus, flexural strength, and flexural modulus of PA6/ABS binary blends increased with the addition of PA66 polymer while the strain at break values decreased with the addition of PA66. The addition of PA66 into PA6/ABS binary blend resulted in an increment of impact strength and a decrement of MFI values up to 3rd recycling process, then it induced a decrement of impact strength and an increment of MFI values for 4th and 5th recycled samples.

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