

The influence of the number of recycling cycles on the thermal and physical properties of polypropylene

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Abstract

Plastic recycling has become one of the approaches used to reduce environmental pollution caused by plastic waste. This study explores the influence of the number of recycling cycles on the physical and thermal properties of Polypropylene (PP) plastic, which is commonly found in plastic waste. Test samples were prepared by melting waste PP in various melting cycles, including 1, 3, 5, and 10 times. The thermal and physical properties of the test samples were observed using Differential Scanning Calorimetry (DSC) and its viscosity, while its microstructure was observed using a Scanning Electron Microscope. The results showed that the higher the number of recycling cycles, the lower the melting temperature (T_m) and glass transition temperature (T_g) of the PP samples. This indicates a decrease in the crystallinity properties of PP as the number of recycling cycles increases. Additionally, the flowability of PP increases with an increase in the number of recycling cycles, which can be attributed to changes in the material's structure and physical properties. The results of this study can help PP manufacturers and users optimize the recycling process and improve the quality of the resulting products. Moreover, this study also contributes positively to energy and material resource conservation as well as reducing production costs.

Keywords:

Recycle, polypropylene, DSC, melting temperature, flowability.

1 Introduction

Incineration and landfilling are commonly used methods for plastic waste management. The incineration method has negative impacts on the environment as it can release harmful gases and compounds such as nitrogen oxides, sulfur oxides, particulate matter, dioxins, and other toxins [1]. Landfilling of waste is constrained by the limited capacity of the Final Disposal Sites (FDS) to accommodate plastic waste, which has a higher resistance to degradation, even though the volume of plastic waste disposal is increasing each year. Plastic waste recycling has become an alternative method in waste management that reduces the direct negative impacts of environmental pollution caused by incineration and landfilling. Recycling plastic waste can also contribute to energy conservation and material conservation by reducing the need for refining fossil fuels into pure plastic [2]. However, there are numerous challenges in establishing a cost-effective plastic waste recycling system due to the complexities associated with polymer separation and processing processes.

Polypropylene (PP) is currently one of the most widely used types of plastic, accounting for 16% of global plastic production [3]. Nearly 55 million tons of PP have been produced in a single year, and this figure continues to grow rapidly due to the popularity of its applications [4]. This material is used in various industries such as packaging, construction, automotive, and sports. Automotive parts such as bumpers, body panels, dashboards, and door panels are made from Polypropylene (PP). While in terms of chemical resistance and thermal endurance, PP material still falls below certain types of thermoset materials, such as when compared to epoxy resin [5]. Most of the PP plastic waste is recycled using primary recycling techniques, where the waste plastic is melted and transformed into plastic pellets. Recycled plastic pellets are then used as a mixture in the production of products with similar features to the original ones. Generally, industries that use recycled plastic pellets have noted differences between pure PP and recycled PP. The properties of pure PP are provided by plastic manufacturers, but the same is not true for recycled PP. Recycled PP from plastic waste can undergo recycling processes in multiple cycles, but there is no scientific research investigating the influence of the number of recycling cycles on the thermal and physical properties of this material. Both of these properties are crucial for reference in the plastic product manufacturing process. To address this information gap, a comprehensive study evaluating the thermal and physical properties of recycled PP as a result of the number of recycling cycles should be conducted.

PP is a recyclable polymer. Recycled PP can be used as a blend with pure PP, with an ideal maximum percentage of around 30%, as indicated by several studies [6][7]. In the production process, whether using pure PP or recycled PP, the material typically undergoes a melting process. During this process, the molten plastic experiences complex stress and temperature changes, as well as variations in frontal geometry. These factors are related to the melt viscosity and polymer processing ability [8], [9].

Incarnato et al. [10] have studied the influence of repeated recycling cycles on the rheological and mechanical properties of pure PP. Polymer viscosity decreases with an increase in the number of recycling cycles, which is attributed to the reduction in the Molecular Weight (MW) of PP after recycling and the narrowing of the polymer's Molecular Weight Distribution (MWD).

The processing ability of polymer materials in a molten state is highly dependent on their rheological properties. Therefore, measuring the rheology and obtaining viscosity data of recycled plastic materials is necessary [11]. Certainly, the data acquired from rheological measurements serve as crucial parameters for quality control in polymer processing. They also aid in identifying appropriate processing variables, as seen in injection molding and compression molding, for instance [12]. Rheological data provide valuable insights into the flow behavior, viscosity, shear rate sensitivity, and other key properties of the polymer material. This information enables manufacturers to optimize processing conditions, such as temperature, shear rate, and shear stress, to achieve the desired product characteristics and ensure consistent quality during production.

Measurement with a Melt Flow Indexer (MFI) is a simple and popular method used in the plastics industry to determine the viscosity of materials. Szczęsna et al. [13] utilized MFI to assess the flowability of PP. Pimentel et al. [9] employed this method to evaluate the homogenization level of PP/recycled virgin PP blends during injection molding.

Various studies have also shown the potential for reusing PP without significant loss of performance in the final product, based on the determination of the mechanical and thermal properties of the pure material throughout multiple recycling cycles [14] [15].

The crystallinity of PP is influenced by factors such as molecular weight, molecular weight distribution, comonomer content, and thermal history [16]. The crystallization in recycled PP materials is affected by the level of degradation, both in terms of degree and crystal form. The condition of PP from plastic waste differs from recycled PP that has undergone artificial degradation because it has experienced various phenomena, including thermal and mechanical loads during manufacturing, filling, transportation, storage, usage, and disposal in municipal recycling facilities (TPA). Additionally, there are contaminant factors that can also impact its thermal properties [17].

The objective of this research is to evaluate the thermal properties, crystallinity, and flowability of recycled PP with varying recycling cycles.

2 Research Methods

The material used is Polypropylene Trilene HI10HO plastic manufactured by PT. Chandra Asri Indonesia [18] which was sorted from the waste of the injection molding production process. The properties of Polypropylene are indicated in Table 1.

Table 1. The properties of polypropylene

Properties	Unit	Value
Melt flow rate	g/10 min	10
Density	g/cm ³	0.9
Yield strength	MPa	35
Flexural modulus	MPa	1500
Melting temperature	°C	163
Recommend processing temperature	°C	220-250

The plastic material was then shredded using a plastic shredding machine to reduce its size. The shredded plastic was washed with water and dried in a circulating hot air oven at 100°C, after washing, the polypropylene needs to be thoroughly dried. This activity ensures that the shredded waste polypropylene is free from contaminants and moisture, making it suitable for further processing. Polypropylene is a non-hygroscopic type of plastic, so it is not affected by moisture content. Moisture content up to 0.2% does not affect the physical properties of polypropylene in the mold, so it generally does not require drying processes [19]. In this case, drying is only needed to remove surface moisture from the washing stage.

The granulation process of the shredded plastic was performed using a screw extruder, where the temperature in the feed zone was set at 180°C, and the temperature in the metering zone was set at 230°C [7]. The melting cycle was set at different variations: 1, 3, 5, and 10 cycles (represented by R1, R3, R5, and R10, respectively). Fig. 1 illustrates the sequence of research steps from shredding to testing.

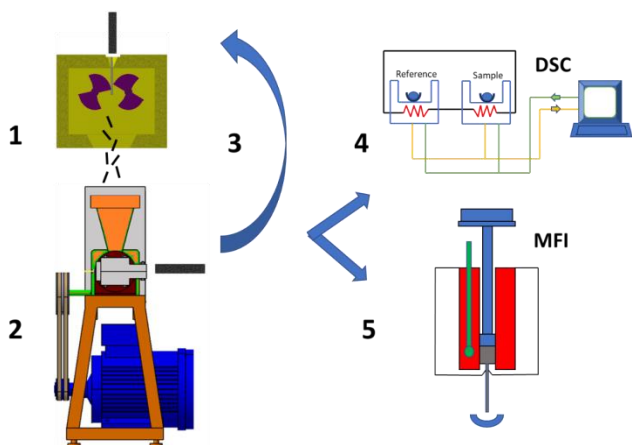


Fig. 1. The research steps: 1) shredding of waste material, 2) remelting, 3) variation of recycling cycles, 4) DSC testing, 5) MFI testing.

The TA instruments TMDSC model 2920 Differential Scanning Calorimetry (DSC) instrument was used to observe the thermal properties [20]. The observed thermal properties include melting temperature, glass transition temperature, crystallization temperature, and degree of crystallinity [21]. An intercooler cell was operated with nitrogen as the heat transfer gas at a flow rate of 100 mL/min. Helium at a flow rate of 25 mL/min was used as the purging gas. The sample was heated and cooled in the temperature range of 20°C–250°C at a heating and cooling rate of 20 °C/min, with a modulation period of 40 seconds and a modulation amplitude of 0.3°C [22]. PP samples weighing 5–10 milligrams were used for DSC testing. Each sample was heated and cooled in a cycle consisting of 1st heating – 1st cooling – 2nd heating. The 1st heating cycle was used to obtain the thermal history of the sample, while the 2nd heating cycle was used to obtain information regarding the structure and intrinsic properties of the polymer [23].

The degree of crystallinity for PP was calculated using the enthalpy of fusion of 100% crystalline PP, which is typically taken as 207 J/g [24][25]. Eq. 1 was used to calculate the degree of crystallinity [26].

$$\chi_c = \frac{\Delta H_i}{\Delta H_0} \times 100 \quad (1)$$

Where ΔH_0 is the specific heat of melting of the plastic material with 100% crystallinity, while ΔH_i is the measured value obtained from DSC. The reaction enthalpy can be determined through DSC measurements by integrating the peak area of the reaction and the baseline interpolated between the start and end of the reaction.

The physical properties of the recycled material are indicated by its flowability. The Melt Flow Index (MFI) functions as a convenient indicator of material processability and serves as an indirect measure of polymer molecular weight. The molecular chains within recycled material are commonly shorter in comparison to those found in virgin material. This phenomenon results in a reduction of molecular weight within the blends as the proportion of recycled material increases, consequently contributing to an observable rise in MFI values [27]. The measurement of the melt flow rate refers to the ISO 1133 standard at 230°C, with a load of 2.16 kg [13]. For each recycling variation, measurements were conducted on 5 samples, and then the average values were taken.

3 Results and Discussion

3.1 DSC Observations

The results of the DSC measurement for the degree of crystallinity during the 1st heating mode are presented in Table 2. Fig. 2 shows the thermogram curves obtained from the 1st heating DSC analysis of both virgin PP and recycled PP samples from different recycling cycles. By calculating the area under the peak and considering the constant heating rate of 20 °C/min, the values of ΔH_m (melting enthalpy) and the degree of crystallinity can be determined. The 1st heating mode provides information about the thermal properties of the material after the melting process, including thermal history and other thermal characteristics influenced by processing, crystallinity, aging, heat treatment, etc [28].

The small peaks before the melting point (indicated by red circles) in Fig. 2 indicate the presence of thermal history in the test sample. Thermal history refers to the history of heat treatment or thermal processes experienced by the material. Thermal history includes temperature, time, and other conditions that have influenced the structure and thermal properties of PP. Common thermal processes in PP include heating, cooling, melting, and crystallization. Each stage in the thermal history can affect the

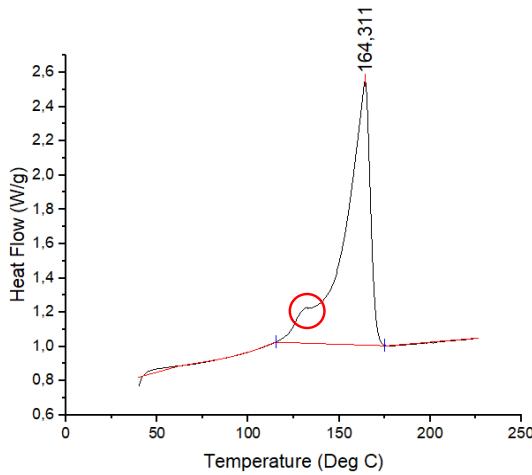
molecular structure, degree of crystallinity, and physical properties of PP. The number and sequence of thermal cycles also play a crucial role in the thermal history. If PP undergoes repeated cycles of heating and cooling, such as in recycling processes, the structure and thermal properties of PP can change. Recycling cycles can result in a decrease in molecular weight, thermal degradation, and polymer chain scission [29].

The recycling process in PP can also affect the melting enthalpy and degree of crystallinity. Melting enthalpy represents the energy required to melt the crystals at a certain temperature. The melting enthalpy of the first recycling is 25.6 J/g, this value increases during the third recycling to 26.4 J/g. In the fifth recycling, a melting enthalpy of 25.62 J/g is obtained, while the highest value is found during the tenth recycling, which is 27.97 J/g. In the case of PP, melting enthalpy increases with an increase

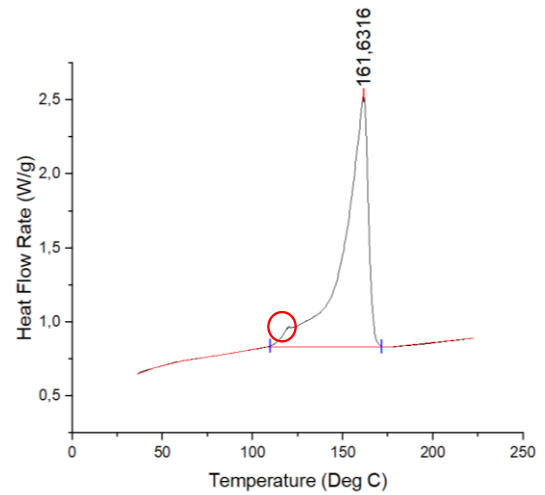
in the degree of crystallinity. The degree of crystallinity is a measure of the amount of crystalline structure formed in the material.

Table 2. The degree of crystallinity can be calculated based on the 1st heating

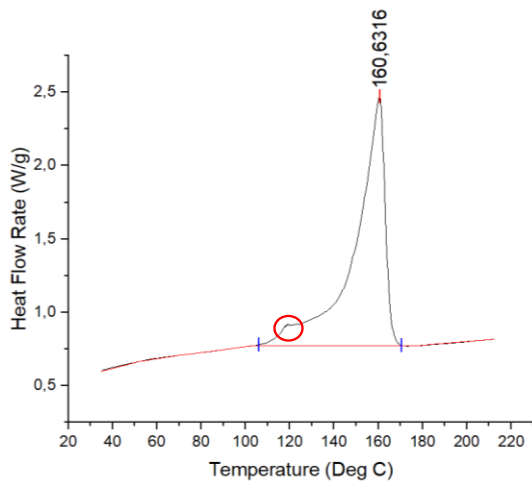
Number of recycling cycles	Heating			X_c (%)
	Area under peak curve (W °C/g)	Heating rate (°C/s)	ΔH_m (J/g)	
R1	25.595	0.333	76.86	37.1
R3	26.42		79.33	38.3
R5	26.62		79.93	39.19
R10	27.96		83.96	40.56



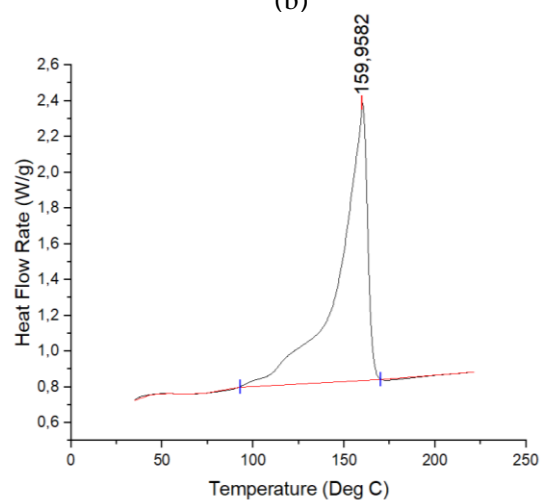
(a)



(b)



(c)



(d)

Fig. 2. DSC 1st heating: (a) R1, (b) R3, (c) R5, (d) R10.

From Table 3, the melting enthalpy shows an increasing trend with an increasing number of recycling cycles. Similar results can be observed in the determination of the degree of crystallinity. With an increasing number of recycling cycles, the crystallinity of the samples increases from 37.1% in the 1st recycling to 40.56% in the 10th recycling. The increase in crystallinity in recycled PP can be attributed to a decrease in molecular weight and shorter polymer chains due to multiple cycles of thermal processing [30]. Indeed, this can also be observed from the DSC measurements in Fig. 3, where the peaks with the same shape and size only shift, indicating that the changes are caused by chain scission rather than changes in the size of the formed structure. The shift in the peaks suggests that the thermal degradation during the recycling process leads to the breaking of polymer chains, which affects the

crystalline structure and properties of the PP material. It is an indication that the repeated recycling cycles result in molecular changes and potentially affect the overall performance of the recycled PP [31]. The lower molecular weight of PP contributes to higher chain mobility, which can result in increased crystallinity. When the molecular weight of PP is reduced through thermal degradation or recycling processes, the chains become shorter, allowing them to move more freely and rearrange into a more ordered crystalline structure. This increased chain mobility facilitates the formation of crystalline regions, leading to higher crystallinity in the recycled PP material. Higher crystallinity often corresponds to improved mechanical and thermal properties of the polymer [32]. The increase in crystallinity can be attributed to the fact that larger molecules with lower molecular weight act as

nucleating agents, promoting the crystallization of semicrystalline polymers by facilitating chain folding and the formation of larger crystal structures. When the molecular weight decreases, the chains become shorter and more mobile, allowing them to more easily arrange into ordered crystalline regions. The presence of nucleating agents facilitates the formation of nuclei for crystal growth, leading to increased crystallinity in the polymer. Therefore, a decrease in molecular weight often increases crystallinity [33] [34].

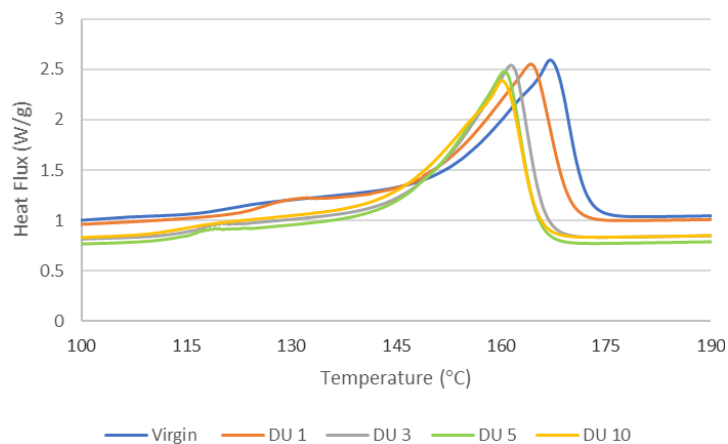
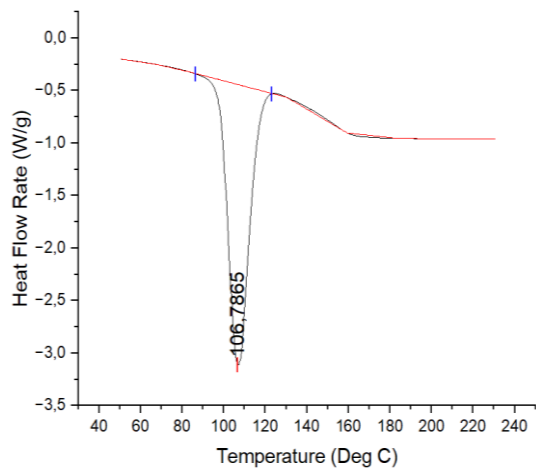
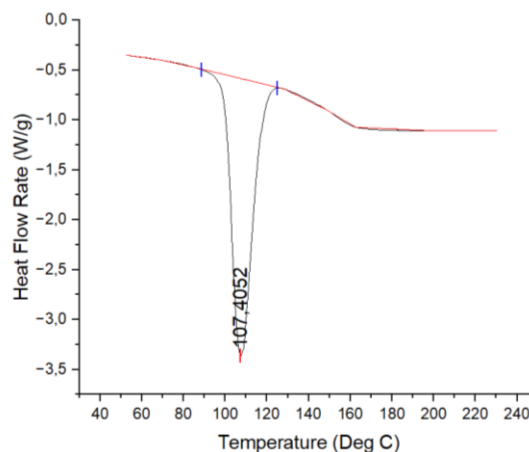


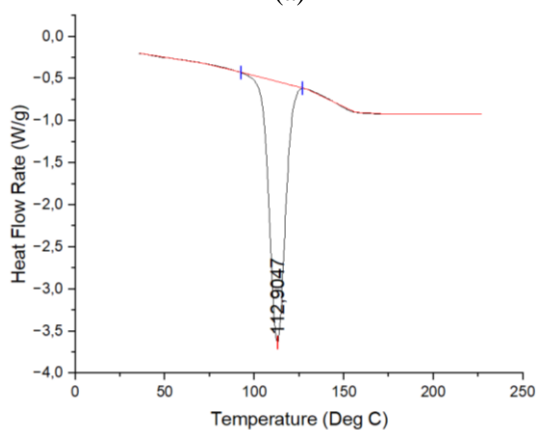
Fig. 3. DSC thermogram for melting temperature, 1st heating method.



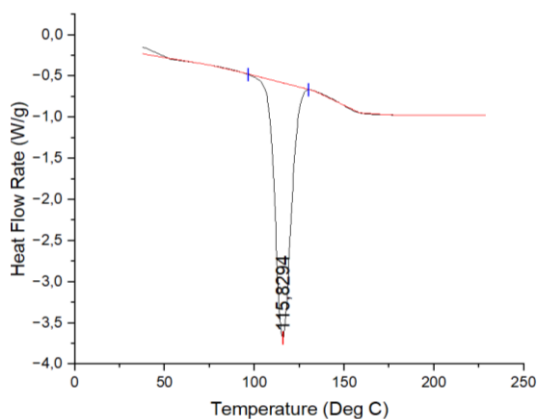
(a)



(b)



(c)



(d)

Fig. 4. DSC 1st cooling: (a) R1, (b) R3, (c) R5, (d) R10.

The first heating step performed in the previous stage eliminates the thermal effects from the previous processing history and provides information about the thermal properties of the recycled PP. Subsequently, in the second heating, the DSC curve of the recycled PP provides additional valuable information by showing the intrinsic properties of the material without any influence from previous processing. As observed in Fig. 5, the

The results observed in Table 3 indicate that as the number of recycling cycles increases, it leads to a decrease in T_m .

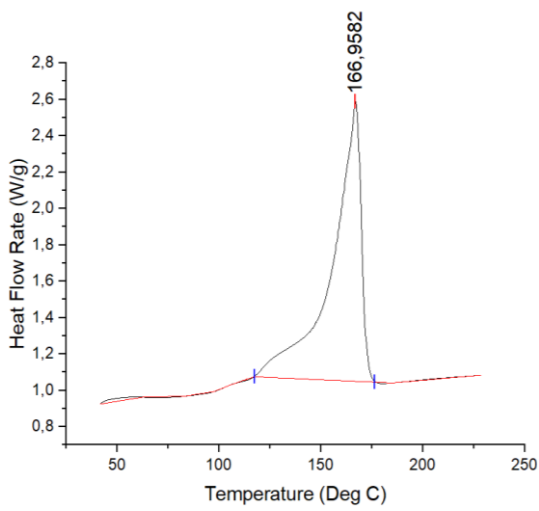
Table 3. Melting temperature and crystallization temperature, 1st heating and 1st cooling

Number of recycling cycles	T_m (°C)	T_c (°C)
Virgin	166.96	109
R1	164.31	106.8
R3	162	107.4
R5	160.6	112.9
R10	160	115.8

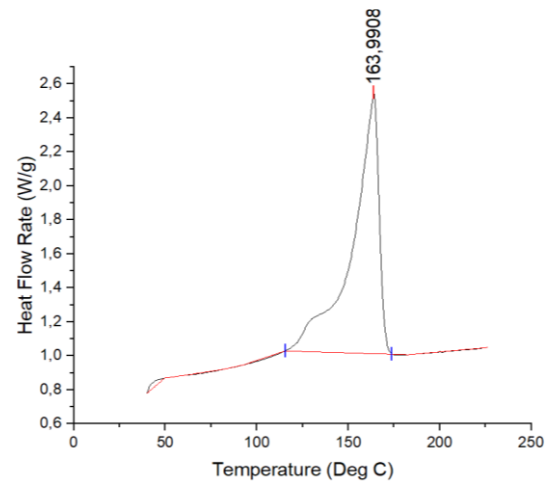
For the determination of crystallization temperature (T_c), the tested samples were subjected to controlled cooling at a rate of 20 °C/minute. The observation of crystallization temperature is shown in Fig. 4. It can be observed that due to the decrease in molecular weight in the recycled material, the crystallization temperature increases with recycling cycles. The figure indicates that the main exothermic peaks for recycling 1 and 2 decrease compared to the virgin material while recycling 5 and 10 exhibit an increase above the crystallization temperature of pure PP. The crystallization temperature tends to increase in recycled PP. These results indicate that thermal recycling promotes the formation of the crystalline phase [35].

second heating curve does not exhibit small peaks like the first heating curve. Therefore, the data presented below is suitable for indicating the thermal properties of the recycled material.

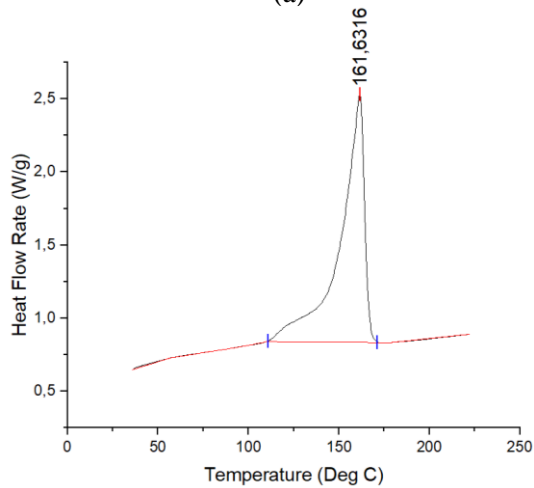
Table 4 and Table 5 represent the results of the calculation of the degree of crystallinity and melting temperature from the second heating. These values indicate the pure properties of the recycled PP material.



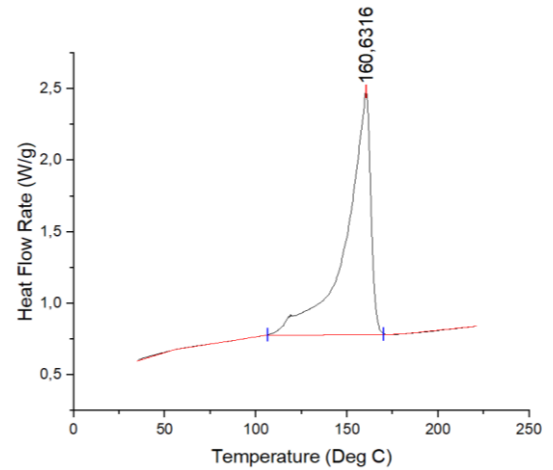
(a)



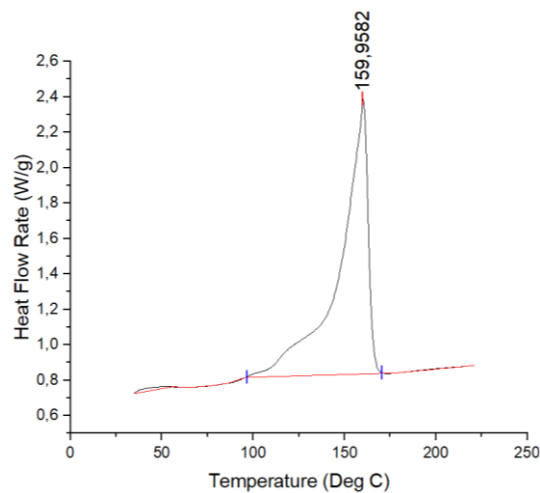
(b)



(c)



(d)



(e)

Fig. 5. DSC 2nd heating: (a) Virgin, (b) R1, (c) R3, (d) R5, (e) R10.Table 4. Calculation of the degree of crystallinity based on 2nd heating

Number of recycling cycles	Heating			X_c (%)
	Area under peak curve (W °C/g)	Heating rate (°C/s)	ΔH_m (J/g)	
1 kali	25.25	0.333	75.82	36.6
3 kali	25.55		76.73	37.06
5 kali	26.07		78.29	37.82
10 kali	26.59		78.85	38.57

The heating rate in Differential Scanning Calorimetry (DSC) measurements of recycled PP is kept constant at 0.333 °C/s. Maintaining a constant heating rate ensures consistency and

reproducibility in the experimental results. When the heating rate is consistent, it becomes easier to identify and characterize various thermal transitions, such as melting points and crystallization temperatures, which are important for understanding the material's behavior.

Table 5. Melting temperature, 2nd heating

Number of recycling cycles	T_m (°C)
Virgin	166.95
R1	164
R3	161.63
R5	160.63
R10	159.96

3.2 Flowability Observation

Table 6 and Fig. 6 present the results of Melt Flow Index (MFI) measurements for several cycles of PP processing. It can be observed that the MFI increases with the number of recycling cycles. These results indicate a decrease in melt viscosity and molecular weight during each processing cycle [36]. Molecular weight is directly related to the length of polymer chains, and it can be concluded that a reduction in polymer chain length occurs. Chain scission can be caused by thermal degradation, oxidative degradation, and mechanical factors during the PP re-melting process. Thermal degradation occurs due to the thermal oscillation of molecules at high temperatures, leading to the breaking of molecular bonds along the chains and the formation of two free radicals. Lower molecular weight is associated with shorter molecules that release bonds more quickly and, therefore, exhibit a higher frequency of entanglements, while longer molecules take longer to release bonds and display more liquid-like behavior at lower frequencies.

Table 6. MFI measurements

Number of recycling cycles	MFI (g/10 min)					Avrg
	1	2	3	4	5	
Virgin						10
R1	10.7	11.2	12.5	12.3	11.8	11.7
R3	11.2	11.6	12.8	13.4	12.9	12.4
R5	14.1	14.6	15.3	15.8	14.9	14.9
R10	16.3	15.9	16.7	17.2	17.2	16.7

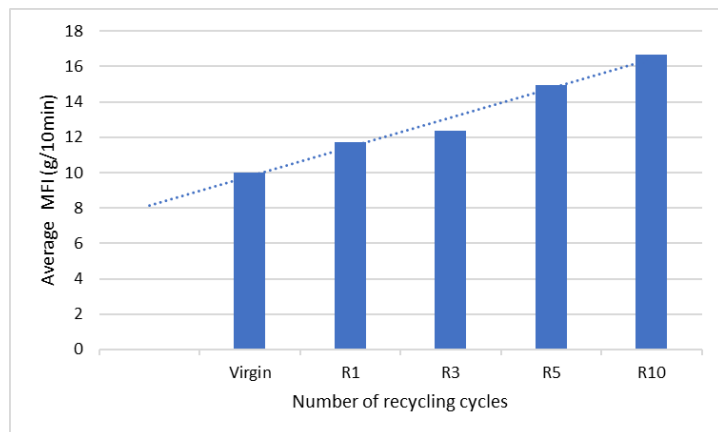


Fig. 6. The trend of increasing flowability in recycled materials.

4 Conclusion

The study's findings emphasize the impact of extensive PP recycling on flow and thermal properties. Flow properties, evaluated through the Melt Flow Index (MFI), revealed a consistent increase with recycling cycles, indicating a reduction in the molecular weight of the recycled PP. Concurrently, the thermal properties assessed using Differential Scanning Calorimetry (DSC) unveiled significant shifts. Notably, the melting temperature displayed a decreasing trend, in contrast to the increasing trend observed for the crystallization temperature. The dominant degradation mechanism during PP recycling was identified as chain scission, as evidenced by minimal changes in microstructure according to the DSC thermogram. Intriguingly, the degree of crystallinity exhibited a rising pattern from the initial to the tenth recycling cycle, suggesting that the presence of shorter polymer chains facilitated the development of crystalline regions. In summary, this study elucidates the intricate interplay between recycling cycles and the resulting alterations in flow and thermal properties, shedding light on the molecular and structural modifications occurring within the recycled PP material.

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