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Thermal Degradation and Fourier Transform Infrared Spectroscopy Characterization of Polypropylene under Multiple Mechanical Recycling Cycles

Siti Hajar Zakaria[1], Saliza Asman[1]*

[1] *Department of Physics and Chemistry, Faculty of Applied Sciences and Technology, UTHM Kampus Cawangan Pagoh, Hab Pendidikan Tinggi Pagoh, KM 1, Jalan Panchor, 84600 Pagoh, Muar, Johor, MALAYSIA.*

*Corresponding Author: salizaa@uthm.edu.my
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Abstract

This study investigates the effect of repeated mechanical recycling on the thermal and chemical properties of polypropylene (PP). Virgin PP (v-PP) was subjected to three recycling cycles (r1-PP, r2-PP, r3-PP) through repeated melting. Thermal degradation behavior was analyzed using thermogravimetric analysis (TGA), while chemical structural changes were examined using Fourier transform infrared spectroscopy (FTIR). TGA result showed a single step degradation for all samples. The onset degradation temperature increased from 418.73°C (v-PP) to 440.64°C (r2-PP), indicating improved thermal stability after initial recycling cycles, but decreased to 414.62°C in r3-PP due to stabilizer depletion and chain scission. Weight loss remained high (~97–98 wt%) with minimal residue (~1–2 wt%). FTIR analysis revealed increased intensity of C–H, C–C, and C=O functional groups in recycled samples, indicating oxidative degradation and structural modification. These findings suggest that limited recycling cycles do not significantly degrade PP performance, while excessive recycling reduces thermal stability.

1. Introduction

Polypropylene (PP) is one of the most widely produced thermoplastic polymers due to its cost-effectiveness, mechanical strength and chemical resistance, making it essential for applications such as packaging, automotive components and household goods [1][2]. However, the increasing global demand for PP has led to a significant rise in plastic waste, creating a need for more effective recycling strategies [1]. Mechanical recycling is the most widely used method because it is simple and cost-effective. However, repeated melting and reprocessing of the polymer can negatively affect its structure and stability through thermal and thermo-oxidative degradation mechanisms, leading to changes in its molecular structure and properties [4][5]. Thermal analysis using Thermogravimetric Analysis (TGA) provides important data on mass loss behaviour, including onset temperature (Tonset), endset temperature (Tendset) and maximum degradation rate, which are commonly used to evaluate the thermal stability of virgin and recycled polymers [5][7]. In addition, Fourier Transform Infrared (FTIR) spectroscopy is widely used to detect chemical changes in polypropylene, such as the formation of carbonyl (C=O) groups, unsaturation (C=C), and changes in CH_2/CH_3 vibrations. These changes indicate oxidative degradation, chain scission, and structural modification caused by the recycling process [2][7]. A comparative analysis of virgin PP (v-PP) and recycled PP (r1-PP, r2-PP, and r3-PP) is carried out to evaluate the changes in thermal stability and chemical structure across recycling cycles. This study aims to systematically evaluate the effect of repeated recycling cycles on the thermal stability and chemical structure of polypropylene. However, limited studies

provide a combined evaluation of thermal degradation behaviour and chemical structural evolution of polypropylene across multiple recycling cycles using both TGA and FTIR techniques.

2. Material and Methods

2.1 Instruments

The Thermogravimetric analysis (TGA) was conducted using a Discovery TGA 550 TGA instrument and chemical structure was analysed by Agilent Cary 630 Fourier Transform Infrared (FTIR) Spectroscopy.

2.2 Materials

The material used was commercial polypropylene (PP) granules with a particle size of approximately 5 mm, representative of industrial PP feedstock used in mechanical recycling and property evaluation studies.

2.3 Sample Preparation

The sample preparation and reprocessing steps for polypropylene are illustrated in Fig. 1.

(a) (b) (c) (d) (e) (f)

Fig. 1 Sample Preparation Steps (a) weight for melt; (b) setup oven; (c) melt; (d) cut; (e) weight for analysis; (f) lable

Virgin polypropylene (PP) granules (v-PP, ~20g) were weighed and placed on a metal tray lined with aluminium foil to prevent sticking, following procedure used to stimulate repeated processing in oven-aging or remolding studies [8][9]. The granules were then heated in a conventional oven at 200-220 °C for 20 minutes until fully melted to form a uniform PP sheet (r1-PP), comparable to temperature used for melt processing and accelerated degradation of PP [10].

After cooling at room temperature for 30 minutes, the sheet was cut into flakes (~1cm^2) and further trimmed to ~5 to 10mg for analysis, consistent with typical TGA and FTIR sample masses [11]. To stimulate multiple recycling cycles, the r1-PP sheet was remelted under the same condition to obtain r2-PP, followed by cooling and

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flake preparation as described above, analogous to multiple-extrusion or multi-cycle recycling protocols used to study thermo-mechanical degradation of PP[4][5]. A third melting of r2-PP sheet produced r3-PP which was again cooled and cut into flakes. All flakes were properly labelled (v-PP, r1-PP, r2-PP and r3-PP) and stored in dry containers to minimize moisture uptake and additional ageing prior to analysis [11].

2.4 Thermogravimetric Analysis

The thermogravimetric analysis (TGA) instrument setup for thermal analysis is illustrated in Fig. 2.

Fig. 2 TGA instrument setup for thermal analysis

For thermal analysis, ~5-10 mg of v-PP, r1-PP, r2-PP, and r3-PP were placed in a platinum pan and analysed using a thermogravimetric analysis system under a nitrogen atmosphere, in line with standard protocols for studying thermal degradation and thermostability of virgin and recycled PP [5]. A single heating program from 30 °C to 700 °C at 10 °C/min was applied, with nitrogen as purge gas at a controlled flow rate, consistent with conditions used to study thermal degradation kinetics and stability of waste and recycled PP [10][11]. Data were collected at 0.1s intervals to ensure high resolution mass and temperature monitoring. The parameters were recorded including the onset temperature of degradation (Tonset), the endset temperature of degradation (Tendset), peak degradation, weight loss and the residual mass percentage as indicators of thermal stability and degradation behaviour for virgin and recycled PP [11][19].

2.5 Fourier Transform Infrared Spectroscopy Analysis

The Fourier Transform Infrared (FTIR) Spectroscopy Analysis instrument setup for identifying chemical changed is illustrated in Fig. 3.

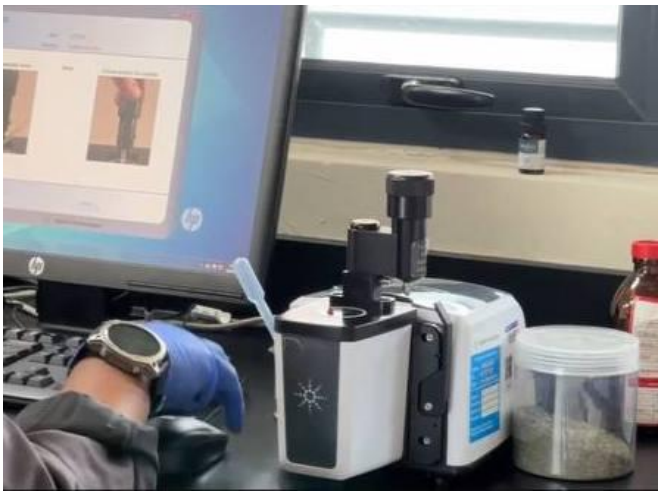

Fig. 3 FTIR instrument setup

FTIR analysis was performed on virgin and recycled PP samples (v-PP, r1-PP, r2-PP, r3-PP) using ATR mode. Fig. 3 shows the method of analysis of the samples by Attenuated Total Reflectance (ATR) mode with Agilent Cary 630 [11][13]. FTIR was configured using spectra wavenumber range of 4000–650 cm^{-1}, resolution of 4 cm^{-1}, 32 sample scans and 8 background scans. The analysis aimed at determining changes of chemicals related to thermal degradation, such as the development of carbonyl groups (C=O) at 1700 cm^{-1}, the appearance of C=C bonds at 1600 cm^{-1} and the change in the CH_2/CH_3 vibrations in 2800–3000 cm^{-1}.

3. Results and Discussion

3.1 Visual Observation

Visual observation is a simple method to show changes in the physical appearance of materials after processing. This approach offers an initial means of identifying colour shift or contaminant that can arise during recycling on thermal degradation process [14][15].

(a) (b) (c)

Fig. 4 Visual appearance of recycled PP (a) r1-PP; (b) r2-PP; (c) r3-PP

The visual appearance of polypropylene flakes after each recycling cycle is shown in Fig. 4. The virgin polypropylene (v-PP) was originally bright red, and r1-PP remained visually similar. However, r2-PP exhibited a darker shade after the second recycling cycle, while r3-PP appeared as a darker burgundy, indicating progressive colour change with repeated thermal processing. The colour change observed across recycling cycles reflects the cumulative effect of thermal reprocessing on the polymer. The observed colour darkening is attributed to thermo-oxidative degradation, leading to the formation of chromophoric groups such as carbonyl compounds due to oxidation and chain scission of the polypropylene backbone. Similar behaviour has been reported for mechanically recycled polypropylene under repeated processing conditions [14][16].

3.2 TGA Analysis

Thermogravimetric analysis is a widely used method to evaluate the thermal stability and degradation behaviour of polymers by measuring weight changes as a function of temperature [17][18]. Fig. 5 presents the TGA and DTG curves of virgin polypropylene (v-PP) and recycled polypropylene samples (r1-PP, r2-PP and r3-PP).

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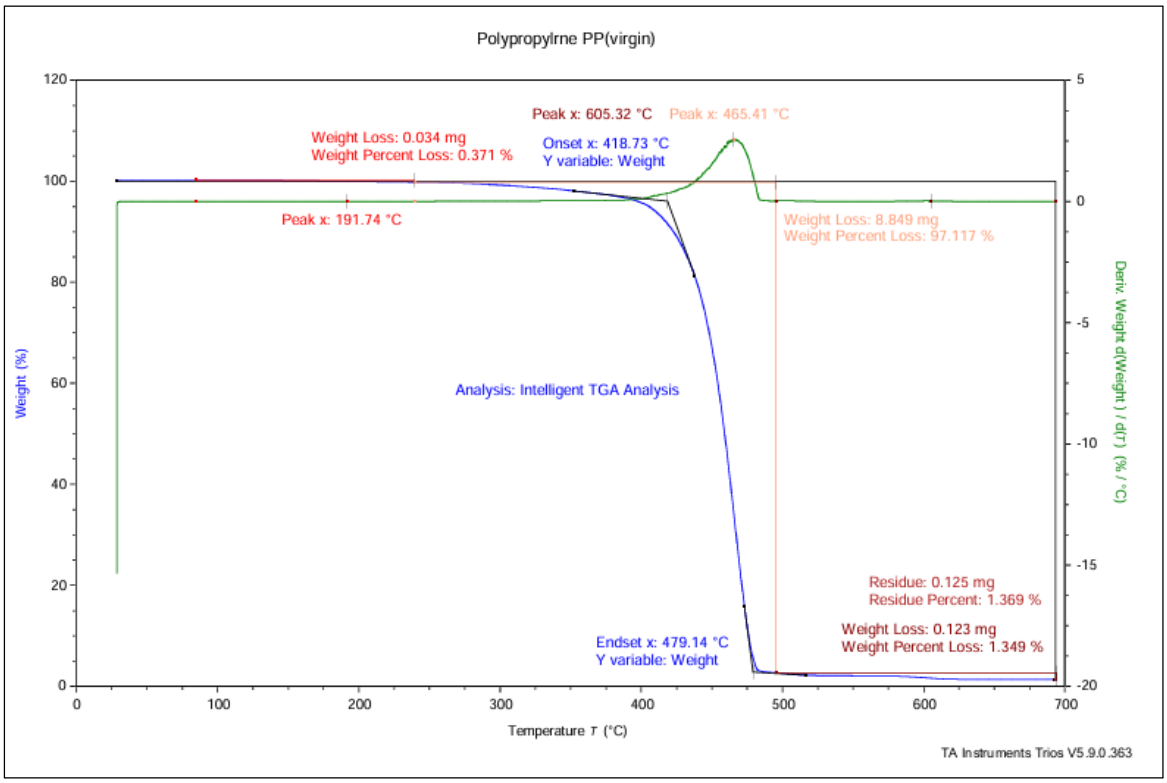
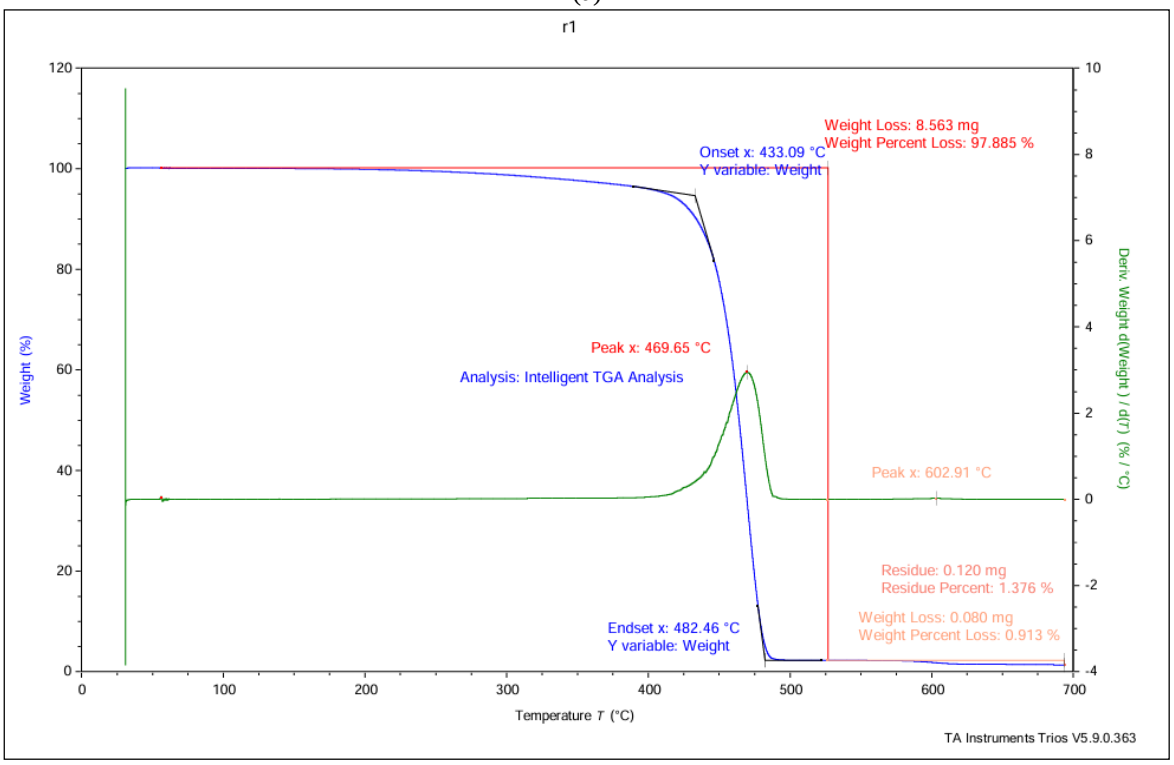
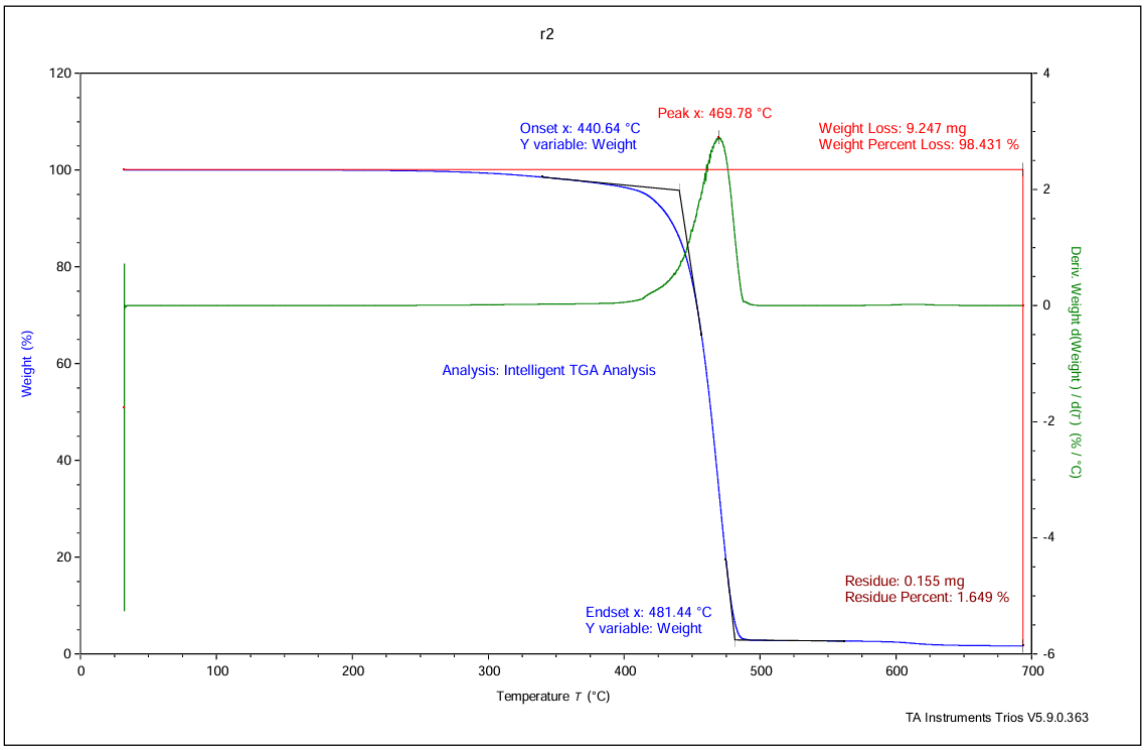


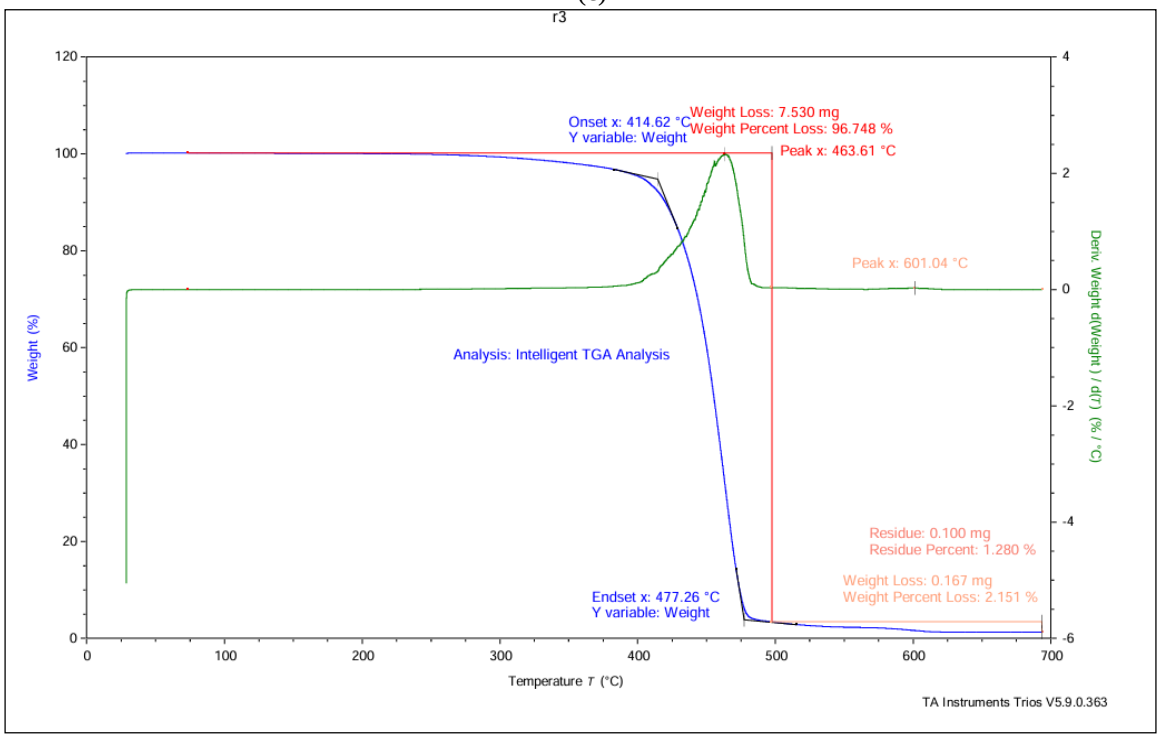
(a)



(b)



(c)



(d)

Fig. 5 TGA and DTG curves (a) v-PP; (b) r1-PP: (c) r2-PP; (d) r3-PP

All samples exhibit a single thermal degradation profile which is characteristics of polypropylene decomposition, in agreement with prior studies reporting one-step, random chain scission degradation for virgin and recycled PP [17][18][19].

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Table 1 summarizes key thermal degradation parameters of virgin and recycled polypropylene.

Table 1 Thermal Degradation Parameter of v-PP, r1-PP, r2-PP and r3-PP

Sample	T_{onset} (°C)	T_{endset} (°C)	Peak Temp (°C)	Weight loss (%)	Residue (wt%)
v-PP	418.73	479.14	465.41	97.117	1.369
r1-PP	433.09	482.46	469.65	97.884	1.376
r2-PP	440.64	481.44	469.78	98.431	1.649
r3-PP	414.62	477.26	463.61	96.748	1.280

3.2.1 Influence of Recycling Cycles on Degradation Temperatures

The onset degradation temperature (T_{onset}) of v-PP is 418.73°C while higher T_{onset} are observed for r1-PP (433.09°C) and r2-PP (440.64°C), indicates a slight improvement in thermal resistance after limited recycling cycles [20]. This enhancement may be attributed to the retention of stabilizers or additives during mechanical recycling, which can delay the initiation of thermal degradation [18][19]. However, the onset temperature decreases to 414.62°C for r3-PP, suggesting that excessive recycling cycles lead to the depletion of stabilizers and increased chain scission, resulting in reduced thermal stability [17]. Overall, PP exhibits an optimum thermal stability after one to two recycling cycles, behind which further reprocessing negatively affects the polymer's resistance to thermal degradation.

The endset degradation temperature (T_{endset}) of all samples remains within a narrow range of approximately 477-482°C, indicating that polypropylene maintains a similar overall degradation window despite multiple recycling cycles [17][18]. The slightly increase in T_{endset} v-PP (479.14 °C) to r1-PP (482.46 °C) and r2-PP (481.44 °C) suggest that mildly recycled PP can withstand high temperatures marginally longer once degradation has initiates, which is commonly attributed to residual stabilizers, pigment or contaminant that degrade at higher temperatures and extend the tail of the TGA curve [11][18]. In contrast, r3-PP exhibits a lower T_{endset} (477.26°C), together with reduced T_{endset} and peak degradation temperature, indicating earlier completion of degradation. Overall, the limited variation in T_{endset} up to r2-PP suggests that the fundamental thermal degradation mechanism of PP remains essentially unchanged, while the decrease observed for r3-PP confirms that excessive mechanical recycling accelerates thermo-oxidative degradation and reduces high-temperature endurance [17][18].

The DTG curves show a slight shift in peak degradation temperature among the samples. The peak temperature of v-PP is 465.41°C, while r1-PP and r2-PP show higher values of 469.65°C and 469.78°C, respectively. However, r3-PP exhibits a lower peak temperature of 463.61°C, slightly below that of v-PP. This indicates that thermal stability improves after one to two recycling cycles but decreases after the third cycle due to progressive degradation and stabilizer depletion. The presence of a single DTG peak in all samples confirms that polypropylene undergoes a single-step degradation mechanism, and recycling does not alter the fundamental degradation pathway [17][18][19].

3.2.2 Mass Loss and Residual Content

Weight loss remained consistently high across all samples (96.7–98.4%), indicating complete thermal degradation of polypropylene. This action can be linked to chain scission and molecular weight loss in recurring thermal treatment which results in shorter chains of polymer which is more volatile in heating. Additionally, the growth of content of residues in recycling cycles implies the concentration of inorganic impurities, filler of degradation by the product in repeated processing [17][19].

Thermogravimetric Analysis (TGA) residues of polypropylene (PP) were low for all recycling cycles with values ranging between 1.28 to 1.65 wt%. The virgin PP sample had slightly high values of 1.40 wt% and 1.65 wt%, respectively. On the other hand, r3-PP had the lowest residue at 1.28 wt%. Each of the samples showed only one primary step of degradation in the presence of nitrogen, and no other steps of degradation could be identified in the temperature range considered [17][21].

The outcome of mechanical recycling of plastic results in alteration of the polypropylene structure, however, based on TGA results, recycled polypropylene maintains or even increases thermal stability [17][19]. Nonetheless, further degradation and yield at subsequent recycling periods implies gradual change of the polymer structure that may limit progressive transformation of the polymer structure to the tough uses [21].

3.3 FTIR Analysis

Fourier Transform Infrared (FTIR) Spectroscopy was used to analyse the chemical structure of recycled polypropylene. The chemical structure of PP in Fig. 6 consists of hydrocarbon backbone, with repeating -CH2-

CH(CH3)- units, whose C-H stretching and bending vibration give rise to the characteristics PP bands in the FTIR spectrum [23].

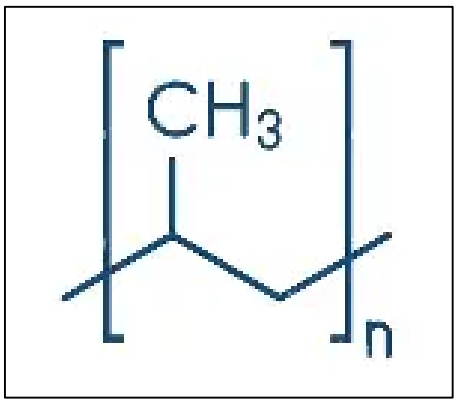

Fig. 6 Chemical Structure of Polypropylene, PP

Including this chemical structure helps interpret the FTIR spectra by linking observed absorption bands to specific functional groups and bonding arrangements in the polymer [23]. This provides a reference to identify whether recycling has caused any chemical modifications, oxidation, or chain rearrangements in the samples.

The FTIR spectra of virgin polypropylene (v-PP) and recycled polypropylene (v-PP, r1-PP, r2-PP, r3-PP) recorded in the 4000-600 cm^{-1} range exhibit the characteristic absorption band of PP as presented in Fig. 7, confirming that the typical polymer structure is preserved after multiple recycling cycles [22][23].

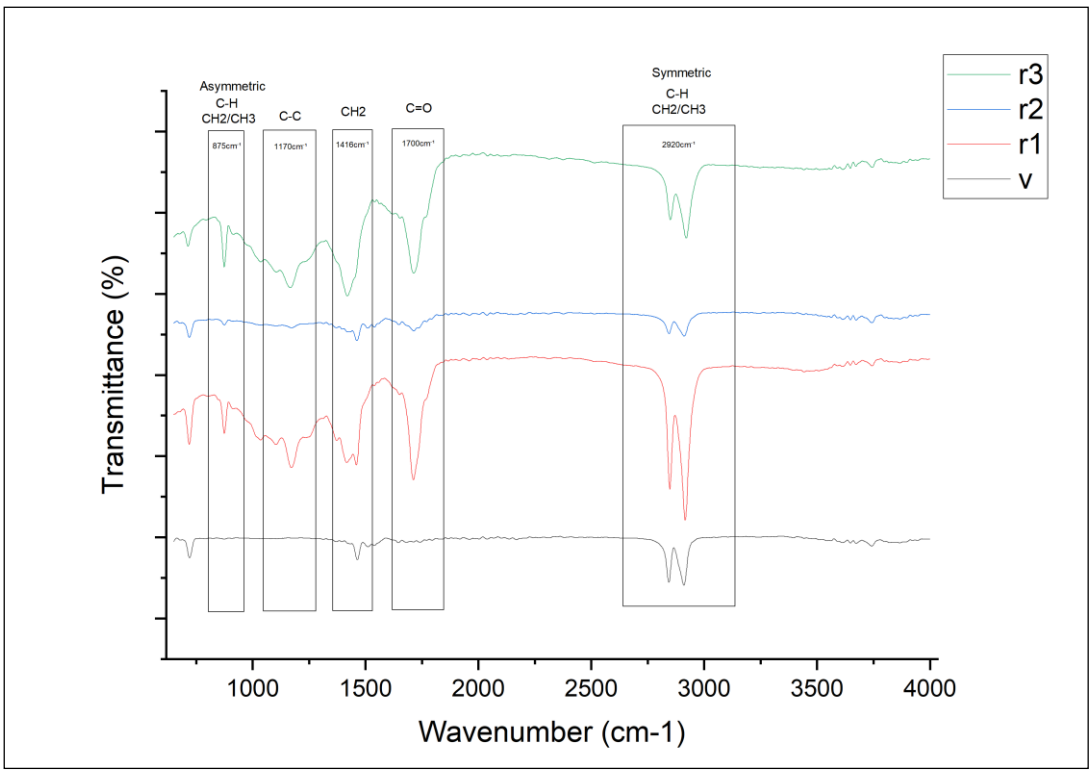

Fig. 7 FTIR Spectra of v-PP, r1-PP, r2-PP and r3-PP

Fig. 7 shows the FTIR spectra of r1-PP, r2-PP, r3-PP compared with v-PP (control sample) in the range of 4000–650 cm^{-1}. The comparison aims to identify changes in functional groups after treatment or reaction by observing the appearance, disappearance, or intensity changes of characteristic absorption bands relative to the control

3.3.1 Changes in C–H Functional Groups

The control sample (v-PP) shows very weak-intensity or barely detectable absorption bands corresponding to C-H stretching and bending vibrations indicates a low presence of aliphatic hydrocarbon groups. In contrast, r1-PP, r2-PP and r3-PP exhibit distinct band at 2920 cm^{-1}, which is a symmetric C–H stretching of CH_2/CH_3 in saturated

aliphatic chains, as typically observed in polymeric materials [23][24]. Others peak approximately at 875 cm^{-1} indicate asymmetric C-H as well as other confirmation of the existence of C-H out of plane or bending vibrations of aliphatic or vinyl type group in the treated [23][25]. This formation of these bands relative to v-PP shows the formation or introduction of alkyl chain due to the process of reaction and modification.

3.3.2 Formation of Carbon Backbone (C-C and CH_2)

The absorption band at 1170 cm^{-1}, which can be attributed to C-C linkage of the carbon backbones, this is weak in v-PP but is easily detectable in r1-PP, r2-PP and r3-PP which is consistent with the reported C-C region in modified polymers [23][25]. On the same way, the band at 1450 cm^{-1}which is associated with the CH_2/CH_3 bending vibrations has a more pronounced in r1-PP, r2-PP and r3-PP as is generally found in aliphatic chains in polymer and oil. These observations indicate that recycled sample have been modified in term of structure by the formation or development of carbon-based structure relatives to v-PP [23][24].

3.3.3 Appearance of Carbonyl (C=O) Group

The major distinction between the recycled samples and the control is observed at approximately 1700 cm^{-1}, which corresponds to C=O stretching vibrations. This band is weak or absent in v-PP but becomes more evident in r1-PP, r2-PP, and r3-PP, indicating the formation of carbonyl groups as a result of oxidative degradation during recycling [25][26]. The carbonyl peak is associated with oxygen-containing functional groups such as ketones, aldehydes, and carboxylic acids, which are commonly formed during thermo-oxidative degradation of polypropylene [23][25].

Overall, the FTIR spectra shows that recycled samples exhibit higher intensities of characteristic bands compared to v-PP. The appearance and increased intensity of C–H (2920, 1450, ~875 cm^{-1}), C–C (~1170 cm^{-1}), and C=O (~1700 cm^{-1}) bands in r1-PP, r2-PP, and r3-PP indicate chemical structural changes induced by repeated recycling.

4. Conclusion

In this research, simple oven melting, Thermogravimetric Analysis (TGA), and Fourier Transform Infrared (FTIR) spectroscopy were used to investigate the effects of repeated thermal recycling on polypropylene. TGA results showed that virgin PP and samples subjected to three recycling cycles underwent a single-step degradation with high weight loss and low residue, as expected for polypropylene under a nitrogen atmosphere. After one and two recycling cycles, the onset, endset, and peak degradation temperatures shifted slightly to higher values, indicating a marginal improvement in thermal stability, likely due to residual stabilizers and pigments. However, a decrease in these temperatures was observed after the third cycle, suggesting reduced thermal stability due to stabilizer depletion and increased chain scission. FTIR analysis confirmed chemical changes in recycled polypropylene compared to virgin PP. The increased intensity of C–H, C–C, and C=O absorption bands in r1-PP, r2-PP, and r3-PP indicates structural modification and oxidative degradation resulting from repeated recycling. Overall, polypropylene can withstand a limited number of recycling cycles with minimal degradation in its thermal and chemical properties. However, excessive recycling or more severe processing conditions may reduce thermal stability and increase oxidative degradation. Therefore, recycled PP performance should be carefully controlled when used in high-performance or long-term applications.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design****: Siti Hajar Zakaria;* ***data collection:*** *Siti Hajar Zakaria;* ***analysis and interpretation of results****: Siti Hajar Zakaria;* ***draft manuscript preparation:*** *Siti Hajar Zakaria contributed to* ***manuscript formatting, editing, and laboratory booking arrangements, including the preparation of related documentation; supervision and review****: Saliza Asman. All authors reviewed the results and approved the final version of the manuscript.*

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