



INTRODUCTION

Industrial development has led to the widespread use of polymeric materials, significantly increasing the issue of waste accumulation. Due to their longevity and negative environmental impact, it is crucial to develop effective recycling strategies. Despite environmental regulations, polymer recycling is hindered by the inconsistent quality of recyclates and degradation caused by processing conditions. This study investigates the impact of multiple mechanical recycling cycles on waste PE-HD packaging material. Using FT-IR spectroscopy and thermogravimetric analysis, the effect of extrusion cycles on the structure and thermal properties of PE-HD was examined.

Waste HDPE packaging from a cosmetic product labeled "100% recycled plastic" and virgin HDPE (Sigma Aldrich, USA) were used. The rPE-HD sample was a mixture of shredded bottle bodies labeled as F, O, C and R (5×5 mm). Recycling was performed in five extrusion cycles using a single-screw extruder, with samples labeled based on cycle count (r1PE-HD, r2PE-HD, etc.). Material properties were analyzed, revealing significant thermal stability deterioration after five cycles. To enhance thermal stability, r5PE-HD was blended with 30 wt.% and 50 wt.% virgin PE-HD, forming rPE-HD 30/70 and rPE-HD 50/50, extruded under the same conditions. Extrusion was performed using a Dynisco LME 230 extruder at 175°C and a screw rotation speed of 60 rpm. FT-IR analysis was used to identify the initial sample and assess the impact of extrusion cycles on the extrudate structure. The UATR technique with a diamond crystal was applied using a Spectrum Two FT-IR spectrometer (PerkinElmer, USA). Spectra were recorded in the 4000–400 cm⁻¹ range with a 4 cm⁻¹ resolution. Dynamic thermogravimetric analysis was performed using the thermogravimetric analyzer TGA 8000 (PerkinElmer, USA) under a nitrogen flow of 40 cm³/min in the temperature range 30–600 °C at a heating rate of 10 °C/min.

EXPERIMENTAL

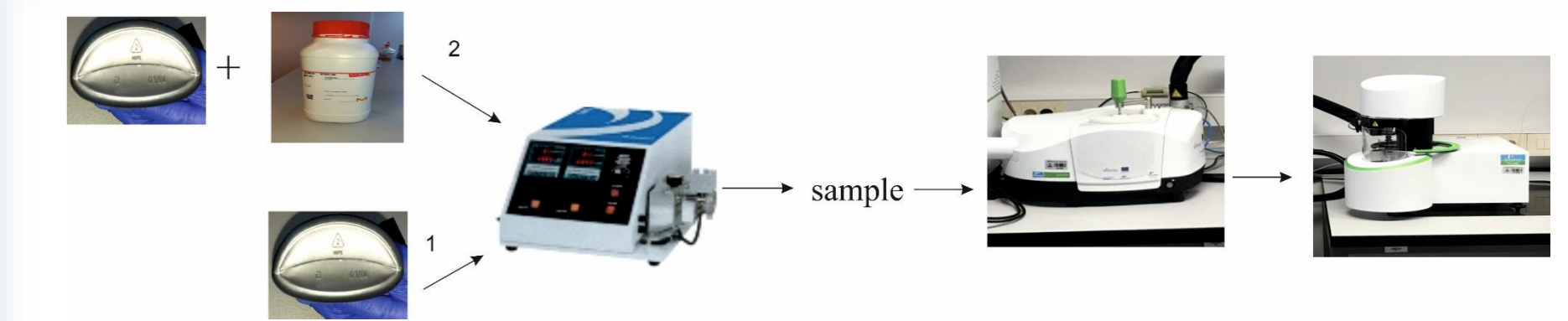


Figure 1. Schematic image of experimental procedure

RESULTS

FOURIER TRANSFORM INFRARED (FT-IR) SPECTROSCOPY

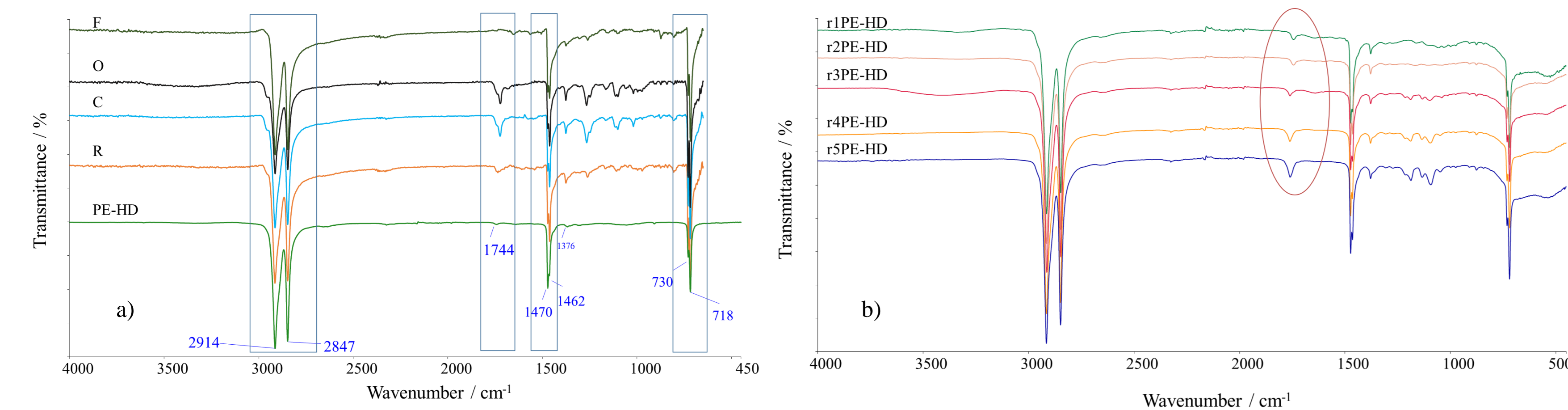


Fig. 2. Normalized FTIR spectrum of : a) bottle body from collected packaging and original PE-HD, b) recycled samples and c) samples rPE-HD 30/70 and rPE-HD 50/50

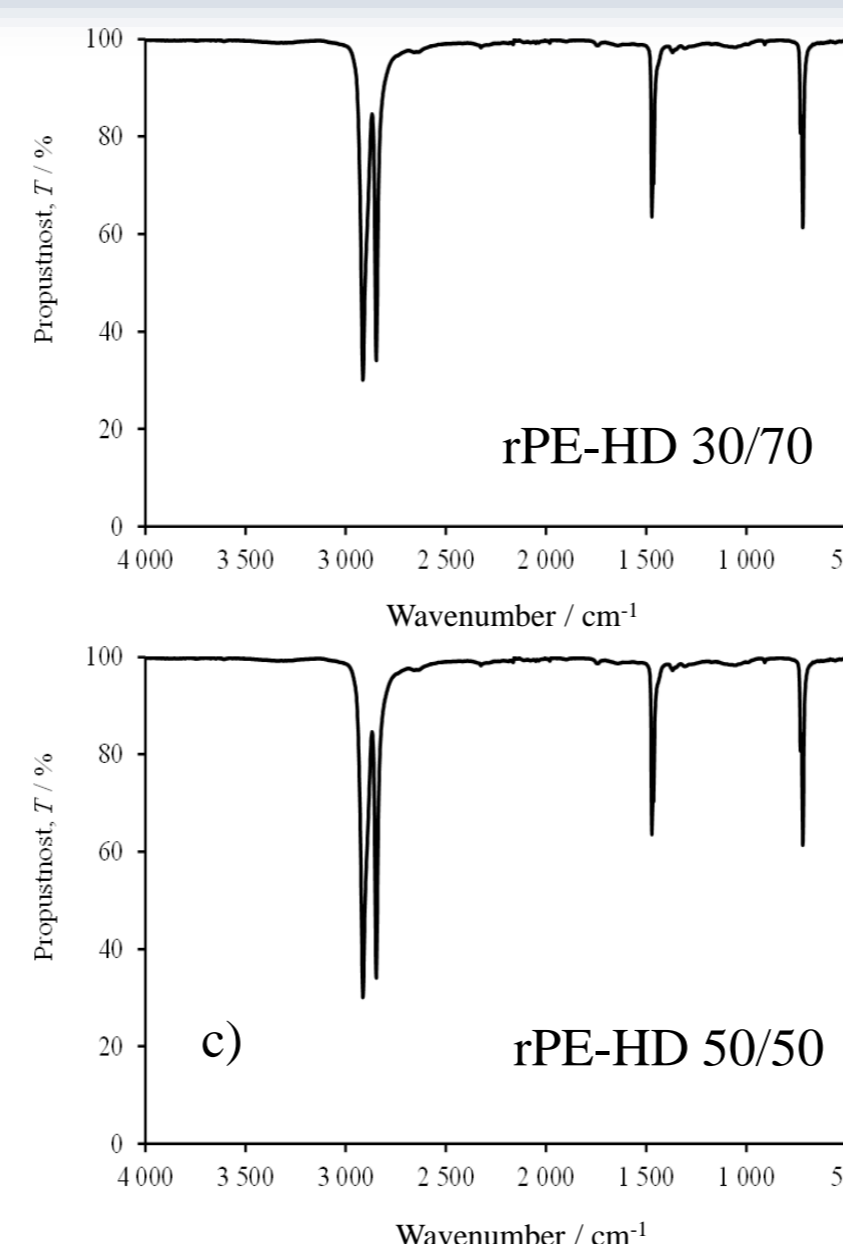


Table 1. Wavenumbers of characteristic absorptions peaks of recycled samples

Sample	CH ₂ asymmetric stretching	CH ₂ symmetric stretching	C=O stretching	CH ₂ bending	CH ₂ bending	CH ₃ bending	C-O-C stretching	CH ₂ rocking	CH ₂ Rocking
r1PE-HD	2915	2848	1742	1472	1460	1376	1161	732	718
r2PE-HD	2914	2848	1744	1472	1463	1377	1160	730	718
r3PE-HD	2913	2848	1752	1472	1462	1377	1185	731	718
r4PE-HD	2916	2849	1760	1472	1465	1378	1185	730	718
r5PE-HD	2915	2849	1760	1472	1462	1377	1185	729	718
rPE-HD30/70	2916	2849	1760	1473	1463	1370	1185	730	719
rPE-HD50/50	2915	2849	1760	1472	1462	1377	1185	730	718

THERMOGRAVIMETRY (TGA)

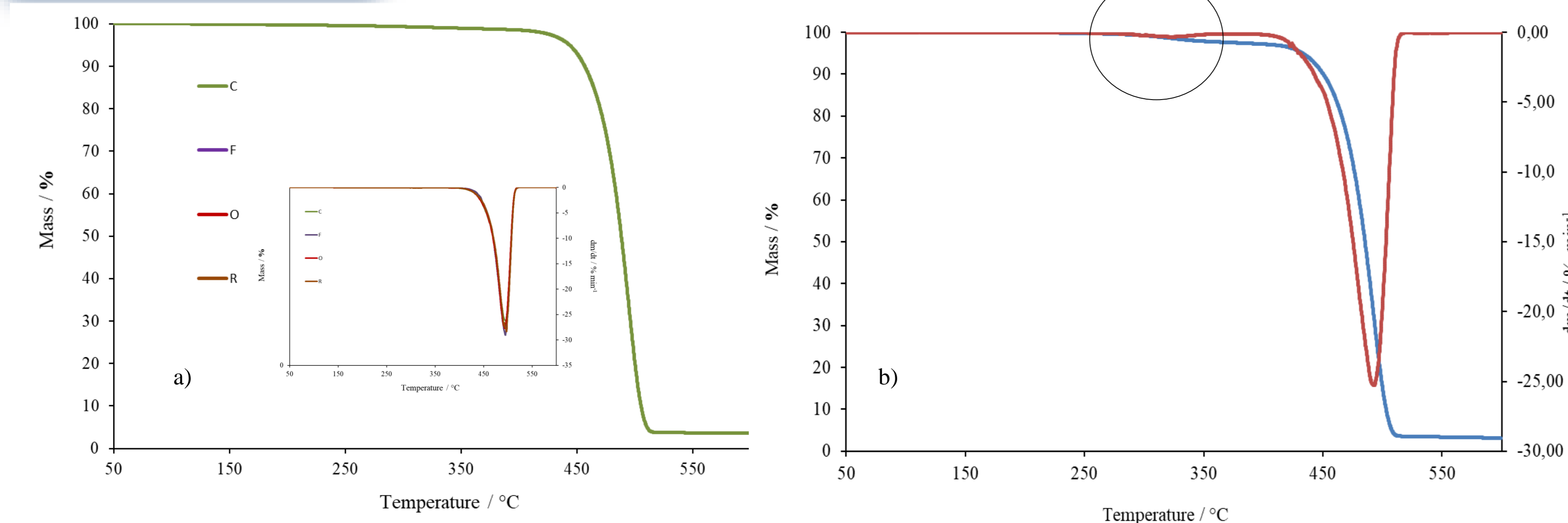


Fig. 3. TG and DTG curves of the non-isothermal degradation of : a) bottle body from collected packaging, b) sample r3PE-HD and c) samples rPE-HD 30/70 and rPE-HD 50/50

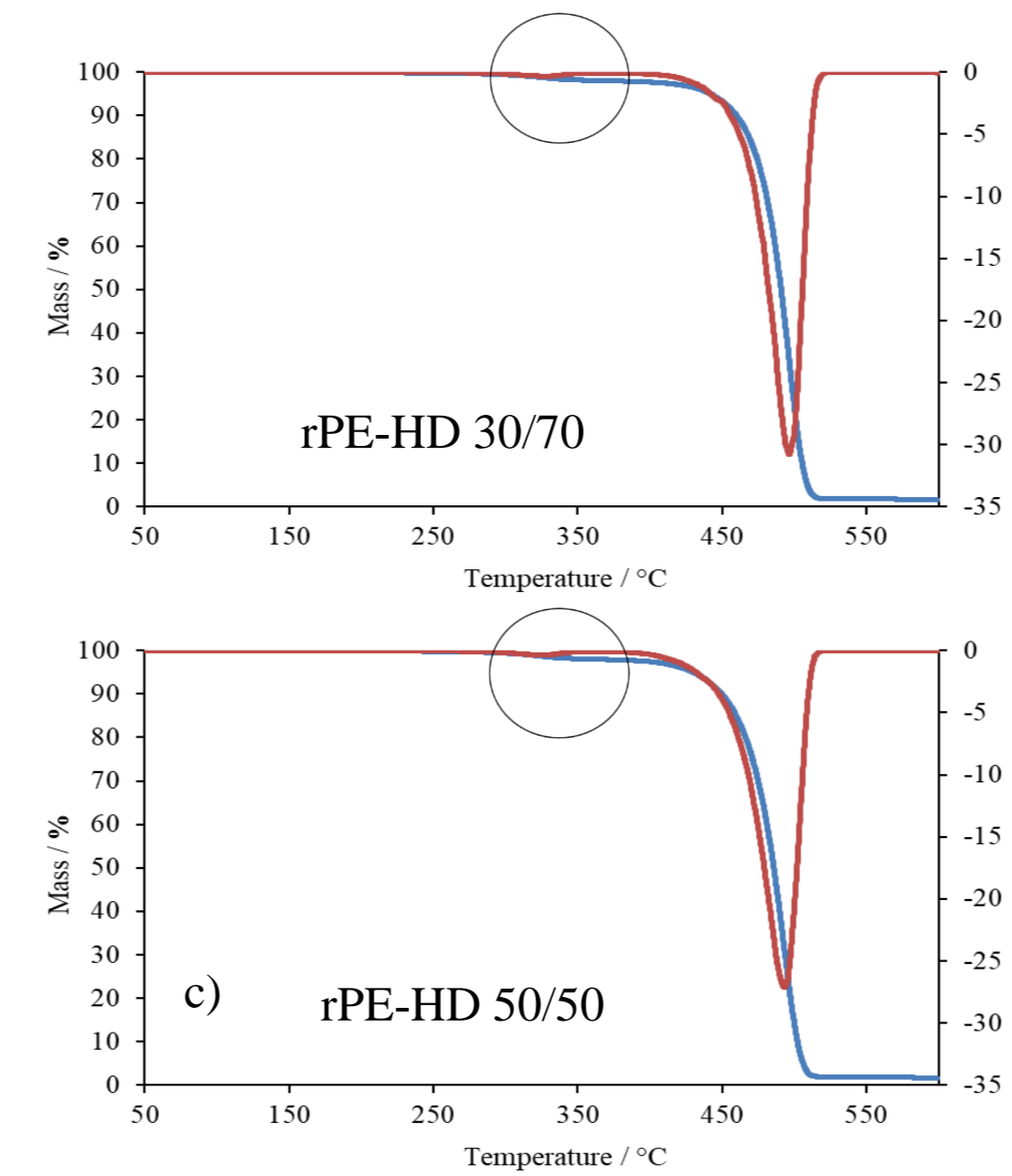


Table 2. Characteristics of non-isothermal degradation of recycled samples

Sample	T ₁ ^o / T ₂ ^o / °C	T _{max1} / T _{max2} / °C	R _{max1} / R _{max2} / %/min	Δm ₁ / Δm ₂ / %	m _f / %
r1PE-HD	-/472	-/497	-/28,1	-/96,7	3,3
r2PE-HD	-/470	-/492	-/27,5	-/96,9	3,0
r3PE-HD	297/470	329/493	0,3/25,2	2,8/94,1	3,2
r4PE-HD	311/472	332/497	0,7/26,2	3,9/93	3,1
r5PE-HD	309/472	327/497	0,6/27,1	4,1/92,7	3,1
rPE-HD 30/70	305/475	327/497	0,3/30,7	2/96,3	1,7
rPE-HD 50/50	305/467	329/493	0,4/26,4	1,8/96,7	1,5

T^o - the onset degradation temperature, T_{max} - the temperature at the maximum degradation rate, R_{max} - the maximum degradation rate, Δm - mass loss at a specific degradation stage, m_f - residual mass

CONCLUSIONS

This study investigated the thermomechanical effects of multiple recycling cycles on the structure and thermal stability of waste high-density polyethylene (HDPE) packaging material. FT-IR analysis showed that material structure depends on the number of extrusion cycles. Significant oxidation and degradation processes were observed after the third extrusion cycle. After the third extrusion cycle, bands indicating oxidation and polymer degradation process significantly increased in the intensity and new ones appeared. Adding virgin HDPE to the fifth-cycle recyclate reduces the intensity of these bands, demonstrating the potential for further recyclate utilization. Thermogravimetric data suggest that two extrusion cycles do not reduce the thermal stability of HDPE. Significant deterioration in thermal stability was recorded in recyclates after the third extrusion cycle. Degradation occurred in two stages, with lower onset degradation temperatures compared to the original material. TG analysis showed that adding virgin HDPE to the fifth-cycle recyclate positively impacts its thermal stability, as reflected in the reduced contribution of the first degradation stage to the overall material degradation process.