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Blow molding of mechanically recycled post-consumer rigid polyethylene packaging waste

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Abstract

Recycled post-consumer rigid polyethylene (PE) packaging waste was washed and compounded on a pilot scale using different processing conditions and the processability of recycled materials with blow molding was assessed. Compared to virgin grade high-density PE, the recycled materials showed a lower crystallinity, a lower thermo-oxidative stability, a higher ash content, a lower viscosity and melt elasticity, and a lower melt strength and drawability. The thermo-oxidative stability varied due to the different washing media. Rheological characterization by frequency-sweep measurements indicated that the virgin grade PE had as expected more linear-polymer-like characteristics whereas the recycled materials showed chain branching or crosslinking related to the processing condition during compounding. The recycled materials were successfully blow molded into 4 L-containers where they showed less resistance to flow than the virgin grade PE. The recycled materials differed in color when different washing media were used. There were no significant differences in the mechanical properties of the 4 L-containers made of virgin grade and recycled PE.

Highlights

- Washing, compounding, and blow molding of rigid polyethylene packaging waste were studied.
- The degradation varied depending on the washing and compounding conditions.
- Rheological results indicated chain branching or crosslinking due to degradation.

KEYWORDS

blow molding, compounding, plastic recycling, rheological properties, rigid polyethylene packaging waste, washing

1 | INTRODUCTION

High-density polyethylene (HDPE) is one of the most frequently used polymers in packaging applications, for

example, milk, shampoo and detergent bottles, sun cream tubes, buckets, cosmetic containers, pharmaceutical bottles, and so on.^{1–3} To improve the manufacturing process, plastic base resins are usually prepared by adding

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compounds such as plasticizers, antioxidants, fillers, colorants, and heat and UV stabilizers.^{4,5} Labels with inks and adhesives, prints, caps, and lids are usually made of other polymers than the original product and other plastic/non-plastic components are often incorporated into the plastic that reaches the market. Collected plastic packaging is therefore very heterogeneous in composition, and contamination originating from the use phase, such as food or chemical residues, degradation products, and other dirt and greases is generally present. When the plastic waste arrives at the sorting plant and is sorted into different streams such as flexible polyethylene (PE), rigid PE, polypropylene (PP), or polyethylene terephthalate, there may be polymeric or nonpolymeric cross-contamination due to the original design of the product and as well as to faulty sorting. As a result, a variety of contaminants are generally found in sorted post-consumer packaging plastics, and these in turn affect the properties of recyclates and create problems in subsequent industrial applications.^{6–9} In mechanical recycling, washing of the sorted plastic waste is usually made with added caustic soda (NaOH) or other chemical washing aids and usually at high temperatures, 70–90°C.^{10–12} This washing also removes contaminants having higher densities, such as metals and minerals, in combination with hydrocyclone or other technologies.^{13,14} The sorted and washed plastic waste can then be compounded and regranulated for further use as a new feedstock. Although mechanical recycling is a well-established process, the recycling of polyolefins, that is, PP and PE, has not reached the same level as that of polyethylene terephthalate, due to a lack of collection schemes, heterogeneity of the products, difficulties in decontamination, and so on which require further development for these waste streams.^{15,16}

One of the main problems in the mechanical recycling process seems to be the degradation occurring during the washing with washing aids at high temperatures and during the compounding with a high temperature and exposure to oxygen.^{1,11,17–19} The common degradation mechanisms, chain scission, chain branching, and crosslinking, usually occur simultaneously during the thermo-oxidative degradation of HDPE, but one of the mechanisms tends to dominate depending on polymer composition, molecular structure and morphology, additives, catalyst residues, and processing conditions.^{11,19–25}

Ideally, recycled PE-rigid waste should have a potential for use in products similar to those in which blow molding, injection molding, and extrusion shaping of virgin grade HDPE are commonly used. Typical rigid packaging products are bottles, containers, and jars manufactured by blow molding where a so-called parison is formed by extrusion and then inflated inside a closed

mold to form a hollow product. During the blow molding process, a stable parison formation is of great importance, and there may be problems due to melting instabilities and other irregularities, sagging, and pleating, that is, buckling of the molten plastic. From a polymer perspective, the molecular weight, melt elasticity, elongational viscosity, melt strength, and drawability are important factors in parison formation.^{26–30}

The complex heterogeneous character of the PE-rigid waste may complicate the thermo-oxidative degradation process and lead to uncertainty with regard to the properties of recyclates and the subsequent shaping process, but rheological characterization by viscosity measurements, entrance pressure losses during capillary flow, rheotens measurements, and time-sweep and frequency-sweep rheometry can provide important information on the melt characteristics and the degradation-related changes in the polymeric structure.^{31–38} The rheological properties are usually sensitive to the molecular structure of the polymer such as branching type, topology, and amount which lead to different melt characteristics and behavior.^{33,39–49}

Our previous study explored the influence of laboratory-scale compounding conditions on laboratory-scale- and large-scale-washed post-consumer rigid PE packaging waste where rheological characterization was used to assess degradation-related changes in the properties.⁵⁰ The present work investigates the influence of the pilot-scale processing conditions on the properties of the mechanically recycled rigid PE stream from post-consumer plastic packaging waste, with a special focus on assessing degradation-related structural changes in washed and extruded recycled materials by means of rheological measurements. The recycled materials were blow molded into 4 L-containers and a commercial virgin grade HDPE was used as a reference. The color and mechanical properties of the blow-molded samples were also examined.

2 | MATERIALS AND METHODS

2.1 | Materials

Post-consumer rigid PE waste (hereinafter referred to as PE-rigid) from source-separated household plastic packaging waste in Sweden, was received from a large-scale sorting plant, Swedish Plastic Recycling. According to the supplier, the sorted PE-rigid bales contained 95% PE-rigid (high-density PE), 2% PE-film (low-density PE), 1% other films, 0.5% PP, 1% other rigid plastics, and 0.5% residues.

SABIC HDPE B5421, with a density of 0.954 g/cm³ and a melt mass flow rate at 190°C and 2.16 g of

0.16 g/10 min, was used as a reference HDPE typically used for blow molding to produce containers for detergents, cleaners, shampoos, and cosmetics. According to the supplier, this grade also contains antioxidants, but unclear in terms of type and amount.

2.2 | Processing

The preparation for mechanical recycling involved shredding, pre-washing, wet grinding, friction washing, centrifugation to remove excess water, batch washing, friction washing, centrifugation to remove excess water, hydrocyclone density separation, friction washing, and centrifugation to remove excess water. On receive, the PE-rigid waste was divided into three batches and washed under different conditions in the batch washing step, whereas the other steps in the washing procedure were kept the same, Figure 1A. The first batch was washed at 40°C with anti-fat detergent and defoamer, the second batch at 40°C with NaOH, anti-fat detergent, and defoamer, and the third batch at 80°C with NaOH, anti-fat detergent, and defoamer.

The washed flakes were compounded using a Coperton ZSK 58 Mc18 co-rotating intermeshing twin-screw extruder. The material was fed into barrel zone 5 and passed through barrel zone 13, followed by a melt filter with a screen size of 150 μm , a melt pump, and a pelletizer, as shown in Figure 1B. The screw configuration included some kneading and reverse elements in barrel zone 7 with mixing elements in barrel zone 10. The temperature profile used was 80–200–230–230–230–230–230–230–240–240–240–240–240°C including the discharge unit, melt filter, melt pump, and die head. All three types of washed material were compounded with a vacuum, 100 mbar, applied in barrel zone 11. For the materials washed at 40°C with NaOH and 80°C with NaOH, the compounding was also done with atmospheric degassing instead of vacuum in barrel zone 11. The material washed at 80°C with NaOH was also compounded with water stripping at a rate of 1 kg/h in barrel zone 10 followed by a vacuum of 100 mbar in barrel zone 11. The throughput rate was 200 kg/h in all compoundings with a screw rate of 300 rpm. The pelletizing was carried out with an underwater pelletizer BKG AH2000 having a die plate of 15 \times 2.8 mm (number \times diameter) with eight knives.

All six types of recycled materials as well as the virgin grade HDPE were blow-molded into a 4 L-container, Figure 1C, using a Uniloy Milacron UMS 12 s machine equipped with W. Müller's torpedo and spider-leg mandrel die head. The temperature profile was kept the same for all the materials where the feeding zone 1 was set to

30°C, zones 2–12 to 200°C, and the die head to 190°C. The screw rotation rate was adjusted for each material to produce bottles weighing 145 \pm 5 g.

A sample of the sorted PE-rigid waste was shredded into small flakes using a Rapid Granulator 300-45KU with a sieve size of 6 mm, and the unwashed flakes were then mixed in a Brabender internal mixer AEV 330 having counter rotating W50 screws at 40 rpm for 5 min at 190°C. The molten polymer was manually flattened and after solidification cut into small pieces.

The notations of the specimens produced are given in Table 1, indicating their processing histories.

All the materials were also injection molded into 25 mm discs using an Xplore Micro Injection Molder IM12. For each disc, the total cycle time was 10 s, with an injection pressure of ca. 450 bar and a holding pressure of ca. 695 bar at 190°C. The discs were characterized with rheometry and differential scanning calorimetry.

2.3 | Experimental

2.3.1 | Parisons

During the blow-molding, the operating window limits were investigated. All processing parameters were kept fixed except for the die opening which was varied between 0.4 and 8 mm, and the screw rotation rate which was varied between 10 and 40 rpm resulting in a pressure range, measured at the end of the extruder between 25 and 160 bar. The parisons shaped under different die openings and extrusion pressures were examined visually to observe pleating or other melt instabilities.⁵¹ The occurrence of pleating or melt instability was rated subjectively from 0 (none) to 3 (very severe), as shown in Figure 2, to give a relative comparison between the processing of virgin grade HDPE and recycled rigid PE.

2.3.2 | Color and gloss analysis

Color and gloss measurements were made simultaneously using a high-precision stationary Konica Minolta CM-36dG spectrophotometer with an integrated ISO-2813-compliant 60° gloss sensor. The illuminant was standard daylight (D65) with a 10° observer and the aperture used had illumination and measurement diameters of 11 and 8 mm, respectively. The geometry was diffuse illumination with a viewing angle of 8° and the wavelength range was 360–740 nm with a wavelength pitch of 10 nm. The color was measured simultaneously with specular-component-included (SCI) and -excluded (SCE) modes, and the gloss measurements were made

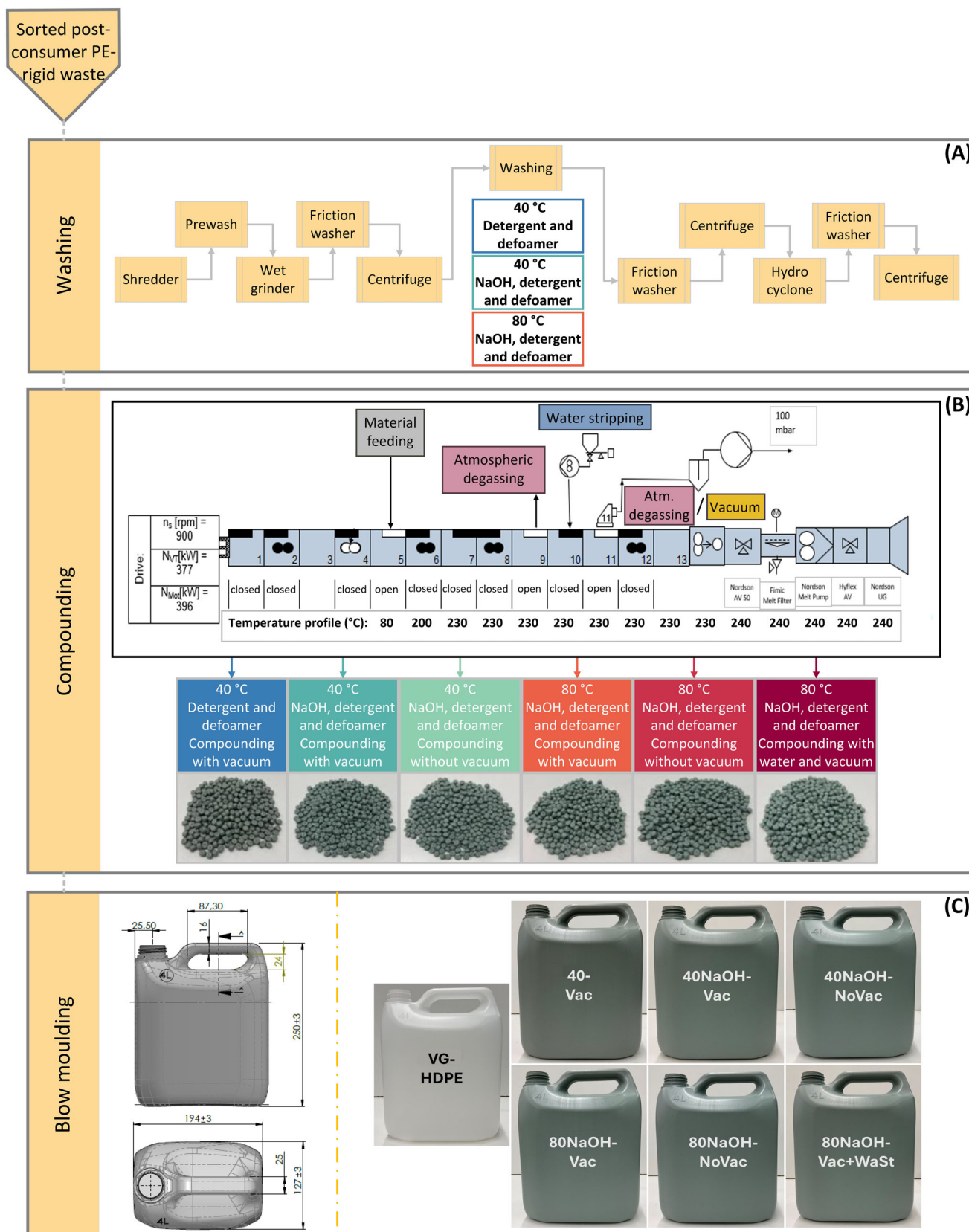


FIGURE 1 Pilot-scale processing (A) washing and (B) compounding. Industrial-scale blow moulding. (C) The 4 L-container (in mm) and examples of samples produced. HDPE, high-density polyethylene; PE, polyethylene.

TABLE 1 The notations and processing histories of the samples.

Material	Washing	Compounding	Sample code
Virgin grade HDPE	N/A	N/A	VG-HDPE
Post-consumer PE-rigid waste	With detergent and defoamer at 40°C	With vacuum	40-Vac
	With NaOH, detergent and defoamer at 40°C	With vacuum	40NaOH-Vac
	With NaOH, detergent, and defoamer at 40°C	Without vacuum	40NaOH-NoVac
	With NaOH, detergent, and defoamer at 80°C	With vacuum	80NaOH-Vac
	With NaOH, detergent, and defoamer at 80°C	Without vacuum	80NaOH-NoVac
	With NaOH, detergent, and defoamer at 80°C	With vacuum and water stripping	80NaOH-Vac + WaSt
Unwashed PE-rigid	N/A	N/A	UW

Abbreviations: HDPE, high-density polyethylene; PE, polyethylene.

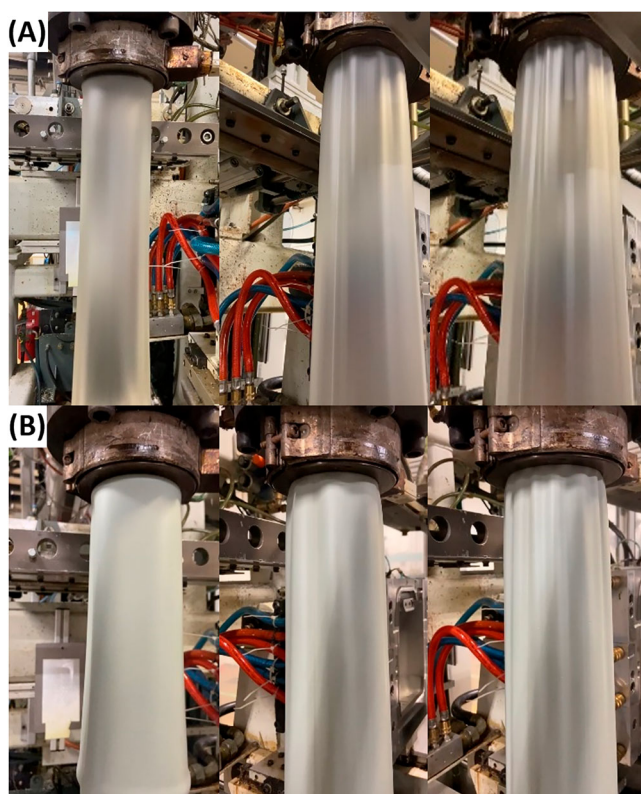


FIGURE 2 Pleating rate 0 (left), 1 (middle), and 3 (right) for (A) VG-high-density polyethylene and (B) 40NaOH-Vac.

with an incidence angle of 60°. The SCE mode represents the color seen by the observer and depends on the surface conditions, whereas the SCI gives the total color appearance independent of the surface conditions. The color was expressed in terms of the CIELAB $L^*a^*b^*$ system

with an L^* (lightness) range from 0 (black) to 100 (white); $+a^*$ being red and $-a^*$ being green; and $+b^*$ being yellow and $-b^*$ being blue. For the VG-HDPE samples, the measurements were made both with the sample holder as background (black) and with a white paper background, because of the translucency of the material, but for the other materials the sample holder background was used alone. The color and gloss measurements were made on a piece of a mixed UW material, on 4 L-containers of VG-HDPE and recycled PE-rigid and on injection-molded discs of all the materials. Five pieces of the mixed UW material were used and three measurements were made on each piece. Three different 4 L-containers were used and six measurements were made on each container. Five different injection-molded discs were used and a single measurement was made on each disc. The average L^* , a^* , b^* , and gloss values were reported.

2.3.3 | Thermal properties and thermo-oxidative stability

The melting characteristics of samples prepared according to ISO 11357-1:2016 from both pellets and injection-molded discs were assessed using differential scanning calorimetry (DSC), Mettler-Toledo DSC 5+. The measurements were performed in a nitrogen atmosphere with a purging rate of 50 mL/min and with heating and cooling rates of 10°C/min. Duplicate tests were made and the average results were reported for the first heating cycles. For the measurement of the heat of fusion (ΔH), the baseline was taken from 65 to 144°C. The degree of crystallinity (X_c) was

assessed using $X_c = (\Delta H / (w_{PE} \times \Delta H_0)) \times 100$ (%), where w_{PE} is the weight fraction of total PE (rigid and film) taken as 97% for the sorted waste and 100% for the virgin grade PE. The heat of fusion for 100% crystalline PE ΔH_0 was taken to be 293 J/g.⁵²

The oxidation-induction-temperature (T_{ox}) and -time (OIT) of samples prepared according to ISO 11357-6:2018 from injection-molded discs were measured with Mettler-Toledo DSC 5+. The T_{ox} measurements were made in air with a heating rate of 10°C/min. The OIT measurements were made at 200 and 240°C. The samples were first heated to the measurement temperature in a nitrogen atmosphere at a rate of 20°C/min, then allowed to rest for 3 min before the atmosphere was changed to air. The gas flow rate was 50 mL/min in all cases. The OIT of the injection-molded 25 mm discs was also measured by time-sweep rheometry using an Anton Paar MCR702 rotational rheometer with a 25 mm parallel plate geometry. The time-sweep rheometry was carried out in air at 200 and 240°C, with a shear strain of 1%, a frequency of 0.159 Hz, and a gap of 1 mm. The time between the loading of the sample and the beginning of the measurements, including the melting, compressing, and trimming of the sample was 9 min. The values of T_{ox} and OIT were retrieved from the intercept point determined by the tangent method, described in ISO 11357-6:2018, and the same was applied for the time-sweep rheometry using the plot of storage modulus (G') versus time. The steepest linear slope observed in the exotherm (DSC) or G' (time-sweep rheometry) was extrapolated by drawing a tangent line and its intercept with the extended baseline was taken as T_{ox} and OIT. Duplicate measurements were made and the average values were reported. The 4 L-containers of VG-HDPE, 40-Vac, 40NaOH-Vac, and 80NaOH-Vac were evaluated for a comparison of their thermo-oxidative stability using the same methods as described above using single measurements.

All the materials were ground into powder and 3.38 ± 0.07 mg samples were used to determine the ash content using a Mettler Toledo TGA/DSC 3+ by two different methods. In one method, the sample was heated from 25 to 650°C at a rate of 10°C/min with a flow rate of 50 mL/min in air. In the second method, the sample was first heated from 25 to 550°C at a rate of 10°C/min with a flow rate of 50 mL/min in nitrogen and then heated from 550 to 650°C at a rate of 20°C/min with a flow rate of 50 mL/min air. Duplicate measurements were made for each type of material and the ash content values were determined at 600°C.

2.3.4 | Rheological properties

The melt viscosity of the samples was evaluated using a Göttfert high-pressure Rheograph 20 capillary rheometer

at 200°C with shear rates between 1 and 10³ s⁻¹, using dies having a diameter (D) of 2 mm and aspect ratios (L/D) of 5, 10, and 15. The Bagley and the Weissenberg–Rabinowitsch shear-rate corrections (ISO 11443:2021) were applied and the graphs of corrected viscosity versus shear rate and of the entrance pressure losses based on the Bagley plots were given for the die with an L/D ratio of 10.

For the injection-molded 25 mm discs, the Anton Paar MCR702 rheometer with a 25 mm parallel plate geometry was used for dynamic-oscillatory shear flow measurements. The measurement gap was set to 1 mm and all measurements were made at 200°C in nitrogen. The linear viscoelastic region for each sample was first determined with oscillatory strain sweep tests performed at a constant frequency of 1 Hz in the shear strain range of 0.001%–100%. Frequency sweep tests were then carried out at a constant shear strain of 1% within the linear viscoelastic region in a frequency range of 100 and 0.0016 Hz. The storage modulus (G'), the loss modulus (G''), the complex modulus (G^*), the complex viscosity (η^*), and the phase angle (δ) were recorded and averages of duplicate measurements were reported.

The elongational properties of the melts were assessed using a haul-off unit, consisting of a strand wheel connected to a force transducer and a take-off wheel, coupled with the Rheograph 20 capillary rheometer. In accordance with ISO 16790:2021-02, the capillary die had a diameter of 2 mm with an L/D of 10 and the drawing length was 220 mm. The measurements were made at a melt temperature of 200°C in an environment of 24 ± 1°C and 22 ± 5% RH. The initial velocity (v_0) of the extruded strand at the exit of the capillary was kept constant at 13.5 mm/s and the starting tangential velocity of the take-off wheel was the same for all the samples except for the UW material for which it was set to 9.9 mm/s. The speed of the take-off wheel (v_1) was increased at a rate of 1.2 mm/s². The force required to draw the melt was recorded together with the velocity of the take-off wheel and the time until the extended strand broke. The strain (ϵ) was calculated as $\epsilon = (v_1 - v_0)/v_0$.⁵³ The mean values of the melt strength (force-at-break) and of the strain-at-break were based on five independent measurements.

2.3.5 | Mechanical properties

Mechanical properties were determined at a strain rate of 1 min⁻¹ at 23 ± 1°C and 31 ± 5% RH using a Zwick/Roell Z2.5 tensile tester equipped with a 2 kN load cell. Tensile specimens of type 5A in ISO 527-2:2012 were cut from three different 4 L-containers for each type of

material where four specimens were taken in the longitudinal direction (parallel to the air blown in the mold), four in the transverse direction and five from the bottom of the container including a weld-line from each 4 L-container. The thickness of the specimens was 1.5 ± 0.2 mm in the weld line regions and 1.2 ± 0.1 mm in the longitudinal and transverse directions. The UW material was injection-molded into tensile bars of type 1BA in ISO 527-2:2012 using Xplore Micro Injection Molder IM12, the thickness being 2 mm. All the samples were conditioned according to ISO 527-1:2012 before the measurements. The Young's modulus, stress- and strain-at-yield, and the stress- and strain-at-break were determined and averages of at least five independent measurements were reported.

3 | RESULTS AND DISCUSSION

3.1 | Parison observations

For the virgin grade HDPE, the pleating of the parison started at a combination of die opening below 4 mm and pressure above 50 bar whereas for the recycled PE-rigid it started at a combination of die opening below 4 mm and pressure above 30 bar. Figure 3 shows the points with fitted lines for the least severe pleating (rate-1) exhibited by the tested materials.

In general, as indicated in Figure 3, there is a shift to a lower pressure level at a given die-opening for the recycled materials compared to the virgin grade HDPE, which indicates that the melts of recycled materials showed less resistance to flow. Among the recycled

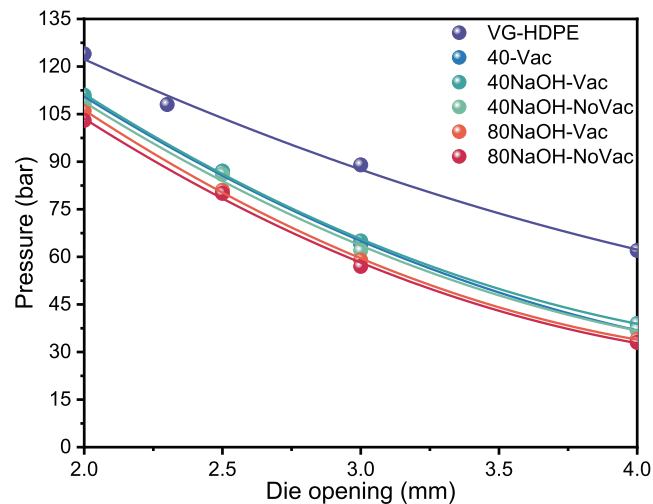


FIGURE 3 Pressure versus die opening (points and fitted lines) representing the pleating rate-1 of the parisons of the tested materials. HDPE, high-density polyethylene.

PE-rigids, those washed at a high temperature exhibited slightly lower pressure values under the same processing conditions. These differences in pressure value observed during the blow molding process paralleled the differences in viscosity and entrance pressure losses, hence melt elasticity and melt characteristics of the samples were subsequently observed. Nevertheless, it was possible to blow-mold 4 L-containers with the desired bottle mass of 145 ± 5 g using 100% recycled PE-rigid material with the same cycle time as the virgin grade HDPE.

3.2 | Appearance, color, and gloss

The 4 L-containers made of 40-Vac showed fisheye formations but the reason was not clear.

As shown in Figure 1, the color of the compounded pellets and blow-molded 4 L-containers varied and the samples were lighter when NaOH was used in the washing. Table 2 shows the average L^* , a^* , b^* , and gloss values of the 4 L-containers and IM-samples produced.

The visual observation was confirmed by color measurement. It was unfortunately impossible to obtain meaningful data for the translucent 4L_VG-HDPE and IM_VG-HDPE samples. For the opaque washed 4 L containers, the values for the samples measured with the SCI were very similar to and probably not statistically different from the values with the SCE, in accordance with the low gloss values. In the case of the washed IM samples, the gloss values were higher and the SCI values accordingly differed slightly from the SCE values. These samples were essentially gray, but after washing with NaOH, the a^* and b^* values indicated that they were slightly more green and slightly less yellow. The values for both 4 L containers and IM samples confirmed that the lightness L^* -value was indeed significantly higher when NaOH was present in the washing medium, confirming the visual observation. This difference in color might be due to different washing mediums causing different efficiencies of contaminant removal^{54,55} or inducing different levels of degradation which results usually in a darker and more yellow color.^{24,54}

3.3 | Thermal properties and thermo-oxidative stability

The first heating endotherm of the VG-HDPE pellets showed a peak at 132°C with a crystallinity of 62%. All the recycled PE-rigid pellets, as well as the UW material, had a main peak similar to that of the VG-HDPE at $131\text{--}134^\circ\text{C}$, corresponding to HDPE, and a very small peak at $150\text{--}170^\circ\text{C}$, attributed to the PP fraction, which

TABLE 2 The L^* , a^* , b^* , and gloss of the materials.

Sample	SCI			SCE			Gloss (GU)
	L^*	a^*	b^*	L^*	a^*	b^*	
4L_VG-HDPE (white)	83.6	-0.6	-4.9	83.4	-0.7	-4.8	7.5
4L_VG-HDPE (black)	59.5	-2.0	-8.6	59.2	-2.1	-8.5	6.9
4L_40-Vac	55.7	-3.7	3.5	55.0	-3.8	3.7	11.0
4L_40NaOH-Vac	60.0	-5.6	2.5	59.6	-5.7	2.6	9.0
4L_40NaOH-NoVac	61.0	-5.9	2.5	60.6	-6.0	2.6	7.3
4L_80NaOH-Vac	61.7	-5.9	2.8	61.1	-6.0	2.9	10.7
4L_80NaOH-NoVac	61.8	-5.8	2.4	61.2	-5.9	2.5	10.9
4L_80NaOH-Vac + WaSt	61.9	-6.0	2.7	61.4	-6.1	2.8	9.5
UW	50.0	-1.4	4.9	49.9	-1.5	5.0	3.3
IM_VG-HDPE (white)	78.0	-0.5	-4.0	76.8	-0.6	-4.1	38.8
IM_VG-HDPE (black)	59.5	-1.8	-7.5	57.6	-2.0	-7.8	40.0
IM_40-Vac	55.7	-3.5	3.3	53.4	-3.9	3.4	53.4
IM_40NaOH-Vac	60.2	-5.4	2.3	58.2	-5.7	2.4	50.0
IM_40NaOH-NoVac	60.6	-5.5	2.5	58.8	-5.9	2.5	46.6
IM_80NaOH-Vac	61.6	-5.6	2.6	59.6	-6.0	2.7	51.9
IM_80NaOH-NoVac	61.8	-5.5	2.4	59.9	-5.8	2.5	42.0
IM_80NaOH-Vac + WaSt	61.9	-5.7	2.4	59.7	-6.1	2.5	57.7
IM_UW	52.6	-1.4	5.0	50.3	-1.6	5.3	38.3

Abbreviations: HDPE, high-density polyethylene; SCE, specular-component-excluded; SCI, specular-component-included.

TABLE 3 Thermo-oxidative stability and ash content (in air) of the samples.

Sample	OIT, time-sweep (min)		OIT, DSC (min)		T_{ox} (°C)	Ash content (%)
	200°C	240°C	200°C	240°C		
VG-HDPE	118	6	210	3.3	250	1.0
40-Vac	15	7	5	0.7	220	1.8
40NaOH-Vac	25	3	12	1.0	231	2.0
40NaOH-NoVac	26	3	11	1.0	232	1.9
80NaOH-Vac	28	4	13	1.2	233	1.8
80NaOH-NoVac	27	4	13	1.0	232	1.6
80NaOH-Vac + WaSt	28	4	14	1.2	234	1.4
UW	30	4	18	1.4	236	2.2

Abbreviations: DSC, differential scanning calorimetry; HDPE, high-density polyethylene; OIT, oxidation-induction-time; T_{ox} , oxidation-induction-temperature.

was also reported in the composition of the sorted PE-rigid waste.⁵⁶ The estimated degree of crystallinity, based on the composition of the feedstock, was similar to that of the recycled PE-rigid and slightly lower than that of the VG-HDPE, between 56% and 58%. The IM-samples also showed melting peaks and crystallinity values similar to those of the pellets of each material.

The thermo-oxidative stability of the materials was characterized by the OIT determined by both time-sweep

rheometry and DSC measurements. The results are shown in Table 3. The time-sweep rheometry results were obtained from the plots of storage modulus in air versus time shown in Figure 4.

The OIT values obtained by DSC measurements were much shorter, but the pattern was the same. The greatest stability was shown by the VG-HDPE sample. In all cases, the stability was poorer at 240°C than at 200°C. Poh et al.³⁶ have suggested that the two-step increase in

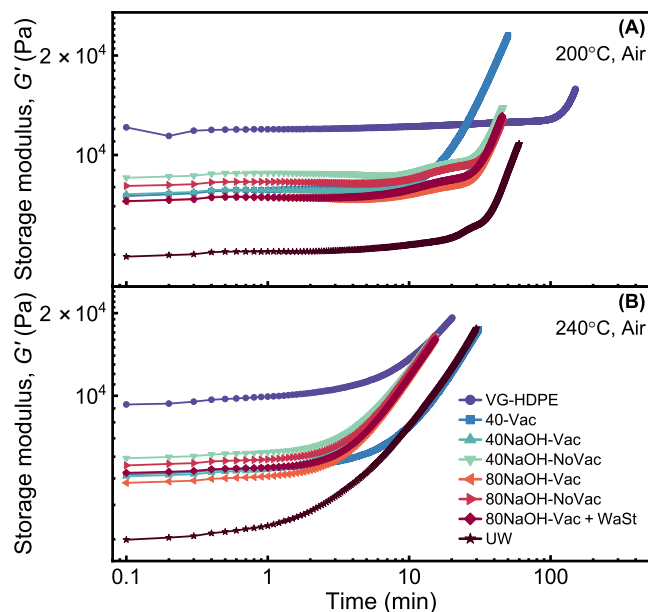


FIGURE 4 The storage moduli versus time in air at (A) 200°C and (B) 240°C. HDPE, high-density polyethylene.

storage modulus of the recycled PE-rigids and UW-material at 200°C is due to chain splitting followed by chain branching, whereas Dordinejad et al.³² consider the behavior to be due to active stabilizers in the materials, but the present study does not permit such conclusions to be drawn. The T_{ox} -temperatures followed the same pattern as the OIT values. The material washed without NaOH had the lowest T_{ox} -temperature, but the reason for this is not clear.

The OIT values based on the DSC measurements, Table 3 showed a similar pattern as the time-sweep rheometry. The oxidation temperatures (T_{ox}), given in Table 3, followed the same pattern as the time-sweep rheometry-OIT and DSC-OIT. All the samples had a T_{ox} above that of unstabilized PE, which was reported to be 210°C in one study⁵⁷ and $180 \pm 5^\circ\text{C}$ in another.⁵⁸ The material washed without NaOH at 40°C had the lowest T_{ox} , due perhaps to a different level of residual chemicals, stabilizers, and oxidative moieties.^{6,8,16,55} This was also observed in our previous study.⁵⁰ The compounding conditions had no significant influence on the thermo-oxidative stability of the samples. The thermo-oxidative stability of the selected 4 L-containers evaluated by time-sweep rheometry, OIT, and T_{ox} was similar to that of the IM samples, Figure 4 and Table 3.

The ash content was lowest for the VG-HDPE and highest for the UW material. The recycled PE-rigids had intermediate values of 1.4%–2.0% which were close to those reported in other studies and usually attributed to the presence of additives, fillers, and catalysts.^{59,60}

3.4 | Rheological properties

Figure 5 shows the melt viscosity (A) and the entrance pressure loss (B) as functions of the shear rate.

In general, VG-HDPE had the highest viscosity whereas the other samples were within a similar range with the UW material having the lowest viscosity. This difference between the viscosity of VG-HDPE and recycled PE-rigids paralleled the difference in pressure values observed for VG-HDPE and recycled PE-rigids during the blow-molding, Figure 3. Also here, the materials washed at the higher temperature showed slightly lower viscosity values than those washed at the lower temperature, in agreement with the small difference in the processing pressure.

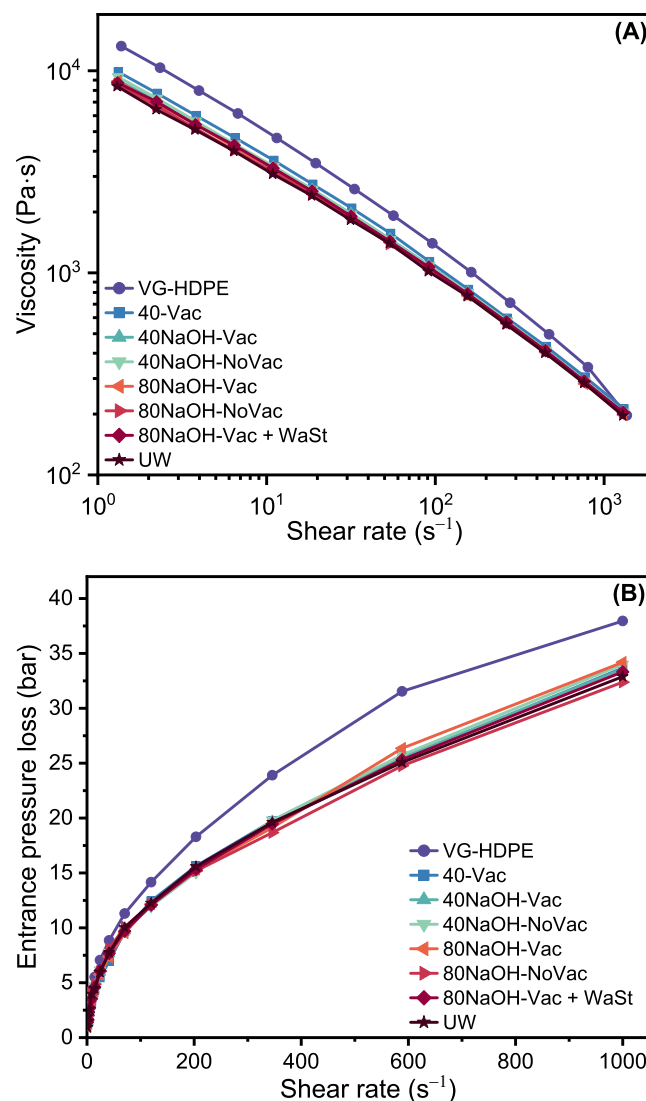


FIGURE 5 (A) The viscosity versus the shear rate at 200°C for all samples. (B) The entrance pressure losses versus the shear rate at 200°C for all samples. HDPE, high-density polyethylene.

The entrance pressure losses in capillary flow in Figure 5B show that the VG-HDPE exhibited the greatest pressure loss, especially at higher shear rates, indicating a higher melt elasticity.⁶¹ The other samples were in a similar range, the difference between them being max ±0.6 bar. These results mirrored the difference in pressure levels during blow molding, Figure 3, and showed similar melt elasticity values of the recycled PE-rigids.

Figure 6 shows the results of the oscillatory shear measurements in the form of plots of (A) phase angle versus complex shear modulus, known as a van Gurp–Palmen plot, (B) complex viscosity versus frequency, and (C) storage and loss moduli versus frequency.

As expected, the VG-HDPE showed linear-polymer-like behavior with a monotonic decrease in the vGP-plot shown in Figure 6A. The change in the shape of the curves for the recycled PE-rigids suggests that the level of branching was greater in those samples.^{33,39,40,48,49} This

observation agrees to some extent with the difference between VG-HDPE and recycled PE-rigids in crystallinity, as chain branching or crosslinking may inhibit the formation of crystalline regions.⁶² The UW material showed a behavior similar to that of VG-HDPE, but this may be because the low-temperature processing in a closed batch-mixer limited the access of oxygen, causing less degradation. Among the recycled PE-rigids, the samples compounded without vacuum exhibited slightly lower phase angle values in the vGP-plot, which indicates either an increase in long branches or other changes in the molecular structure.^{33,39,41,43} This was supported by the slightly higher values of both the complex viscosity and the storage modulus at lower frequencies for these materials, Figure 6B,C, indicating a more branched structure.^{33,49,63} When vacuum was not applied in the degassing zone, a greater rate of degradation and increased branching can be anticipated due to the greater access to

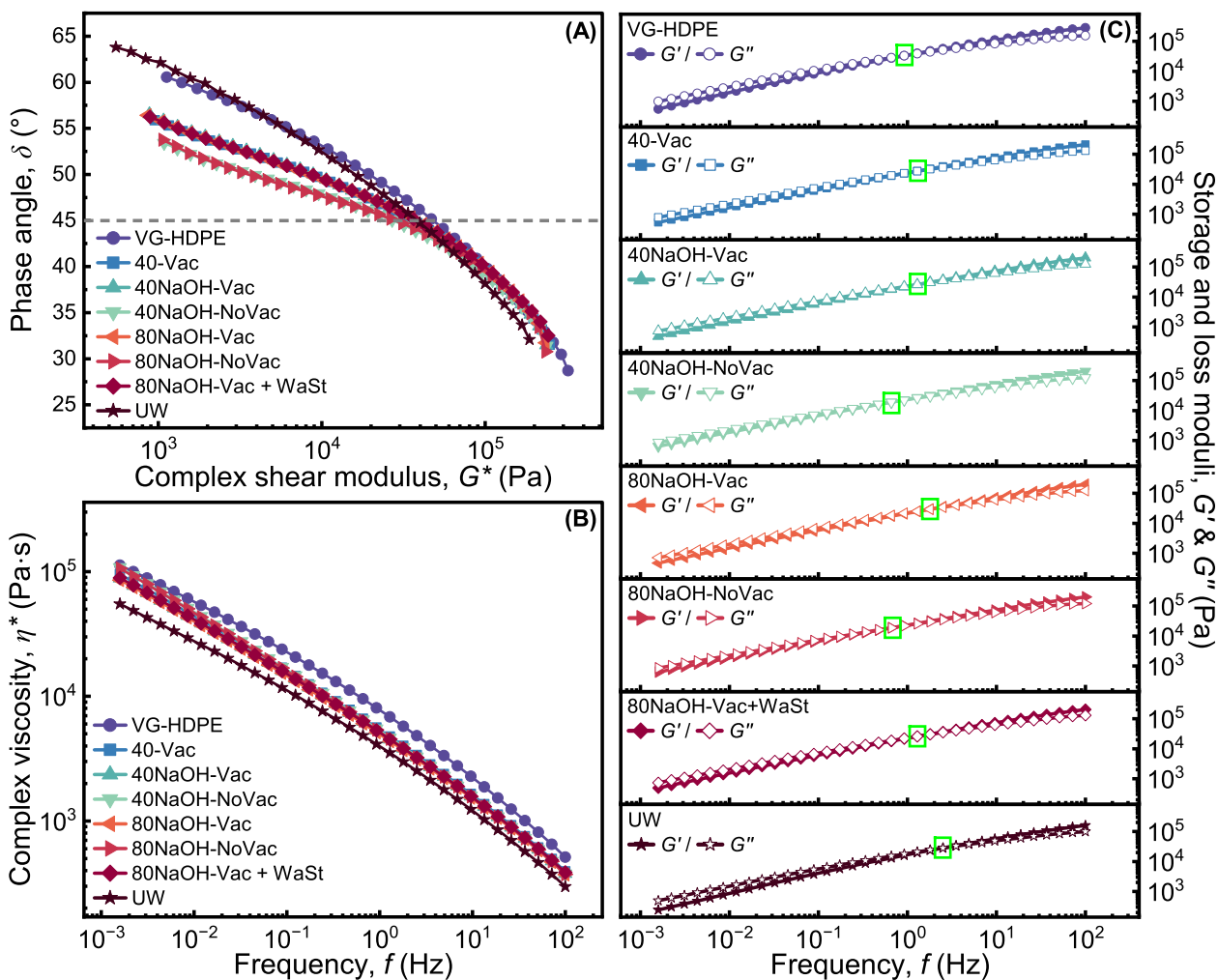


FIGURE 6 Dynamic rheological behavior of the samples in frequency sweeps at 200°C: (A) van Gurp–Palmen plots showing phase angle as a function of complex modulus, (B) complex viscosity as a function of frequency, and (C) storage and loss moduli as functions of frequency for each sample, with the crossover point framed by a green rectangle. HDPE, high-density polyethylene.

oxygen. The use of a vacuum in compounding can lead to structural differences and different levels of branching. The different compounding temperatures were affected in a similar way, inducing different levels of chain branching, as shown in our previous study.⁵⁰ All the samples exhibited a character that was more viscous than elastic, with regard to the reference line at the phase angle of 45° in the vGP-plot.⁴⁷ This was also reflected in the crossover points of G' and G'' , in Figure 6C, at a relatively high frequency for all the samples, indicating a more viscous character.⁴⁷ The use of the vGP method could be further supported by other properties, such as free volume, but this would require more detailed rheological characterization and applying the time-temperature superposition principle, which would provide further information regarding the complexity of the molecular structure.^{33,42,44,45}

The drawability and strength of the melts are shown in Figure 7. The melt properties of the recycled PE-rigids differed from those of the VG-HDPE but were in a similar range within themselves. Although the VG-HDPE

sample had only a slightly higher melt strength, it had a significantly higher melt strain-at-break than the PE-rigids, which implies that the recycled materials will have lower deformability at shaping than the studied VG-HDPE. The lowest values shown by the UW material were expected, due to the remaining impurities and rigid particles that probably caused early rupture. No major influence of the washing or compounding conditions was observed. The differences in melt properties between the VG-HDPE and recycled PE-rigids and the similarity of melt properties of the recycled PE-rigids, supported the observed differences in pressure levels during blow molding, Figure 3.

3.5 | Mechanical properties

Figure 8 shows the mechanical properties (Young's modulus, stress-at-rupture, strain-at-rupture, stress-at-break, and strain-at-break) of the samples, taken from the 4 L-containers and the UW material.

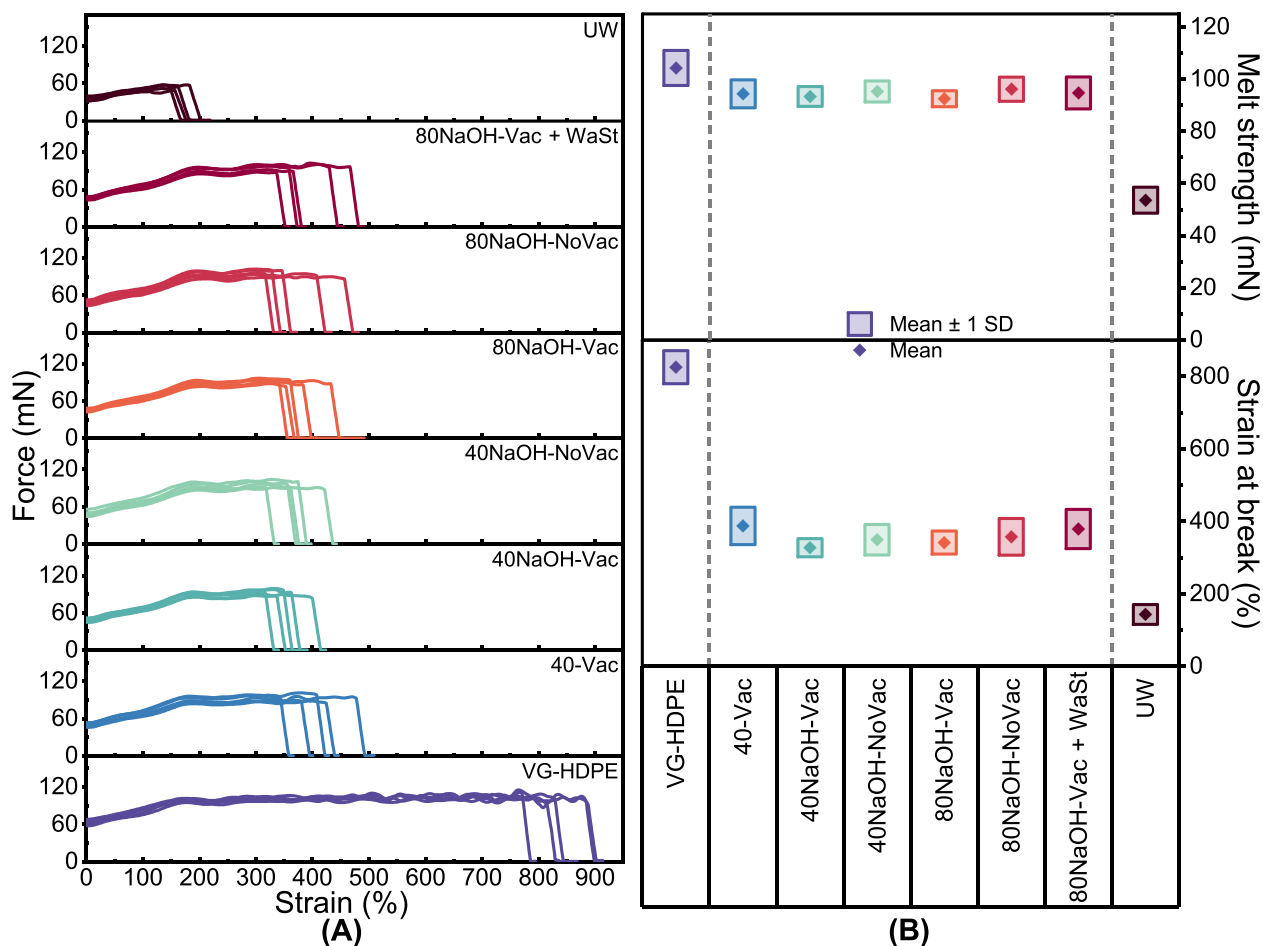


FIGURE 7 (A) Force-strain curves for some of the melts at 200°C. (B) Melt strength and strain-at-break of the melts, where a diamond indicates the mean and the height of the box the standard deviation. HDPE, high-density polyethylene.

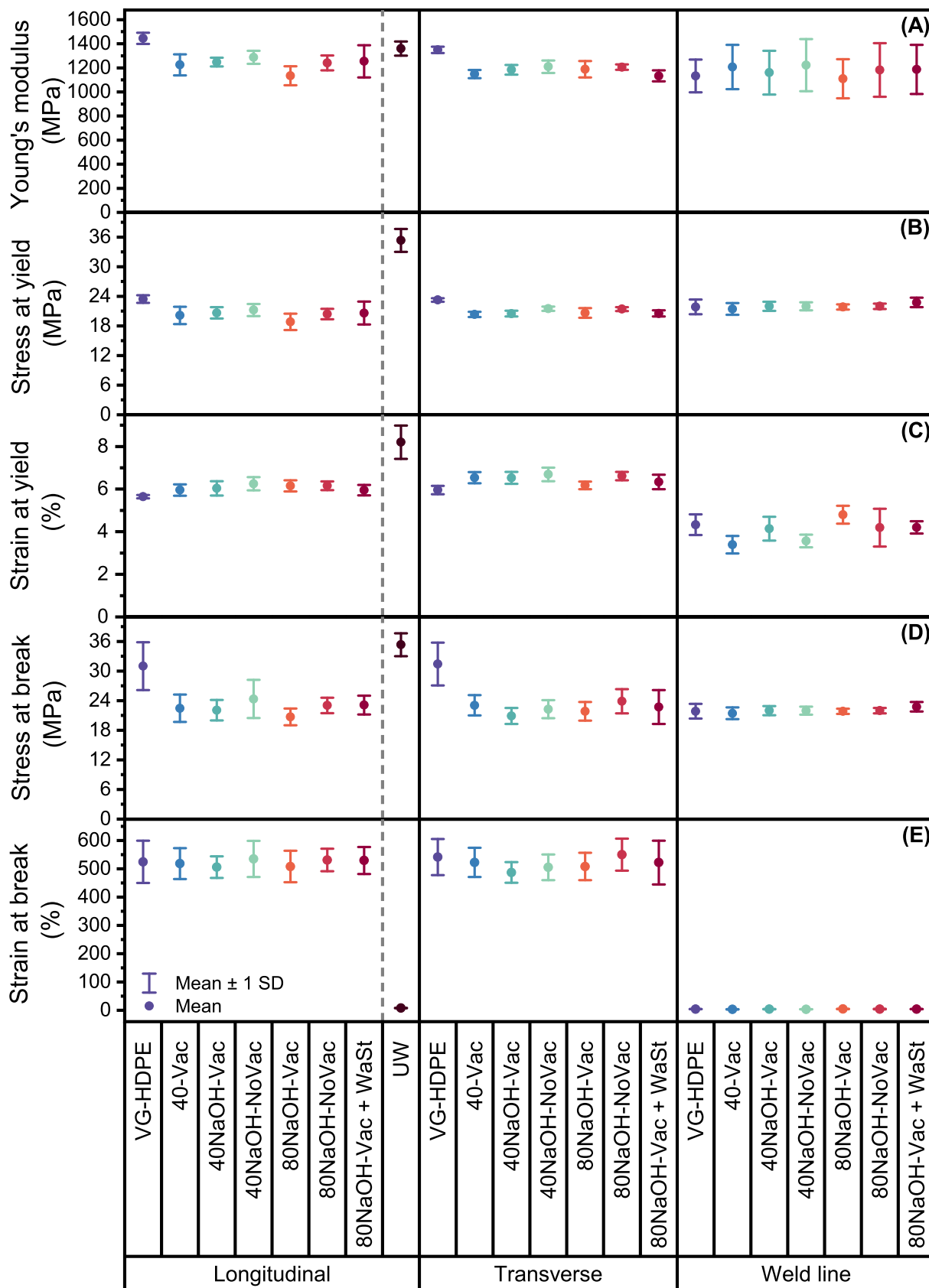


FIGURE 8 Mechanical properties of the samples taken from the 4 L-containers in the longitudinal and transverse directions and in the weld line regions: (A) Young's modulus, (B) stress-at-yield, (C) strain-at-yield, (D) stress-at-break, and (E) strain-at-break. *Injection-molded tensile bar of UW material considered as being in the longitudinal direction. HDPE, high-density polyethylene.

No major differences in mechanical properties between VG-HDPE and recycled PE-rigids were observed. In both the longitudinal and transverse directions, the VG-HDPE samples had a higher Young's modulus, 1400 MPa, than the recycled PE-rigids which varied between 1100 and 1300 MPa. Within the latter materials, the ones washed at 40°C were slightly stiffer in the longitudinal direction than those washed at 80°C but the values were similar in the transverse direction. The average stress-at-*yield* was 23 MPa for VG-HDPE and varied between 19 and 21 MPa for the recycled PE-rigids. The average strain-at-*yield* in both test directions was between 5.6% and 6.7% for all the materials. The VG-HDPE samples had a stress-at-*break* of 31 MPa which was higher than the PE-rigids which had values between 21 and 24 MPa in both directions. These slightly higher values of stiffness and strength for VG-HDPE correspond to the slightly higher crystallinity of the VG-HDPE.^{6,64} In contrast to the difference in molten-state strain-at-*break* between VG-HDPE and recycled PE-rigids, the strain-at-*break* in the solid-state was similar for all the samples in the range 480%–560%.

To investigate the weld line region formed in the blow-molding process, specimens were cut from the bottom of the 4 L-containers. The Young's modulus was between 1000 and 1200 MPa for all the materials. The yielding generally started at the weak weld line and the elongation took place in this region. The stress- and strain-at-*yield* were therefore considered to be equal to the stress- and strain-at-*break* for those specimens. The average weld line strength was 21–22 MPa, which was as high as the strengths exhibited by samples in the longitudinal and transverse directions. The ultimate strains in the weld line region varied between 3.5% and 5%, which is a low value.

Since the UW material was not blow-molded, no direct comparison with the containers is possible. In the injection-molded bars there was no yield but a direct ductile fracture. The average Young's modulus was 1360 MPa and the average with average stress- and strain-at-*break* were 35 MPa and 8%, respectively.

4 | CONCLUSIONS

The washing temperature and the water stripping during compounding had no significant influence. The washing medium influenced the thermo-oxidative stability, and the vacuum application during compounding probably led to a different level of chain branching. Also, the color of the resulting pellets differed due to different washing mediums, either because of differences in cleaning efficiency by better removal of contaminants or because of

different levels of degradation caused by washing and subsequent compounding. These observations implied that different processing conditions change the extent of the degradation which in turn results in structural changes. Therefore, the processing conditions should be considered carefully for the long-term durability of recycled materials. The recycled post-consumer rigid PE packaging waste showed, in general, a slightly lower crystallinity, a lower thermo-oxidative stability, a higher ash content, a lower viscosity and entrance pressure losses, and a lower melt strength and drawability than the virgin grade HDPE used as a reference blow molding grade.

In connection with the observed differences in properties of recycled PE-rigids and virgin grade HDPE, the blow molding process window was shifted to lower pressure levels with the same die-opening during the parison shaping of the recycled materials than those used with the virgin grade. Four liters containers were successfully blow molded with 100% recycled content, but the sample washed without NaOH at 40°C showed a fisheye formation. The slight differences in mechanical properties between 4 L-containers made of recycled materials and those made of virgin-grade PE were considered to be unimportant for their function. An important observation was that the weld line strength at the bottom of the container was almost the same for recycled the PE-rigids as for the virgin grade HDPE. The useful properties of recycled PE-rigids and the successful production of containers supported the recyclability and applicability of post-consumer rigid PE.

AUTHOR CONTRIBUTIONS

Conceptualization, methodology, and writing—review and editing: E.C.B.N. and A.B. Validation, formal analysis, investigation, data curation, writing—original draft preparation, and visualization: E.C.B.N. Resources, supervision, project administration, and funding acquisition: A.B. All authors have read and agreed to the published version of the manuscript.

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DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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