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A comparison between laboratory-scale and large-scale high-intensity washing of flexible polyethylene packaging waste

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Abstract

Sorted flexible polyethylene packaging waste was washed on a laboratory scale at both 25 and 40°C with water alone, with water and added NaOH, and with water and added detergent. The washed materials were then compounded using a twin-screw extruder and injection molded. The results were compared with those for an unwashed and a large-scale high-intensity sample washed with NaOH at 70–80°C. An intensive washing combined with high temperature gave a lower extensibility both in the molten and solid states presumably because of a greater degradation of the recycled material. Washing with NaOH resulted in the lowest strain-at-break in both the melt and solid states, whereas washing with water alone or with a detergent gave similar strain-at-break levels. The melt strength, the stiffness and tensile strengths at room temperature, and the thermal properties were less affected by the washing procedure. Although the final properties seem to be useful in general there were still some trade-offs between the washing condition and the degradation. While these results were expected, the influence of washing parameters on final product, for example, injection molded sample, has to our knowledge not been shown before especially regarding the mechanical properties.

Highlights

- Post-consumer flexible polyethylene packaging waste was studied with a focus on washing conditions.
- The influence of washing conditions on thermal, rheological, and mechanical properties of recycled samples was investigated.
- High-intensity washing at high temperature caused significant degradation of the material, this was to our understanding shown for the first time.

KEYWORDS

degradation, flexible packaging waste, mechanical properties, melt strength, plastic recycling, washing

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1 | INTRODUCTION

Packaging for food, beverages, personal hygiene products, and household goods has for a long time been the largest application sector for plastics, representing about 40 wt.% of the plastics production both in EU and globally,¹ where products such as bottles, trays, bags, films, disposable cutlery, food packaging, and wrapping foil mostly have a short lifetime, typically less than 1 year.^{2,3} In EU 27 + 3 in 2020, 61 wt.% of the total collected post-consumer plastic waste was packaging waste.¹ The same report stated that the distribution of waste management in post-consumer packaging plastics was 46% recycling, 37% energy recovery, and 17% landfill.

Flexible materials account for the fastest-growing segment in plastic packaging. In 2017, more than 50% of the household waste in Norway and Sweden was polyethylene (PE) film,⁴ and a greater recycling rate, especially in the case of flexible plastic packaging, would be important. Flexible packaging waste is however a challenging waste stream for recycling and the recycling rates are still quite low.⁵ The main difficulty is the heterogeneous composition of the waste, which contains multi-layer materials, non-plastic components, a broad range of additives, and a high level of contamination by food residues and other impurities.^{6–10} In addition, the molecular degradation occurring during the service-life and in the recycling process may lead to significant changes in the material properties.^{3,11–15}

Mechanical recycling, including waste collection, sorting, shredding, washing, extrusion-melt-mixing (compounding), and granulation of the extruded material, is a well-known technology for transforming polymeric waste into “new” raw material.^{4,15–18} After sorting, washing is an important step to remove surface contaminants, especially if the composition of the waste is very heterogeneous. The focus of many ongoing studies seems to be on the sorting step, but although sorting is important, the effect of washing on the properties is of great interest.

The main techniques commonly used to remove contaminants employ water, a solvent or friction.¹⁹ Wet-friction washing usually involves intensive mechanical agitation at a temperature of 70–90°C, with added detergent, caustic soda (NaOH) or a surfactant.^{20–22} One study concerning hard packaging plastic waste (high-density PE, HDPE) reported that the detergent acted as a plasticizer, reducing the tensile strength, increasing the elongation-at-break, and accelerating the thermal degradation.²³ Santana and Gondim²⁴ compared the effect of detergent, NaOH, and a combination of both in the washing of recycled post-consumer HDPE. The different washing media resulted in differences in crystallinity and melt viscosity, and the degradation increased

when NaOH was used. Another study investigated the effect of washing temperature, room temperature, and 60°C, on a post-consumer PE film fraction. Hot water washing facilitated the removal of greases and oils, but the overall difference in cleaning efficiency between room temperature and 60°C washing was insignificant.¹⁹ In a study of artificially contaminated HDPE bottles, Devlieghere et al.²⁵ investigated the influence of NaOH concentration and temperature on the removal of contaminants. Washing HDPE, low-density PE (LDPE)/LLDPE, and polypropylene (PP) wastes using only NaOH as an active substance has been reported to result in a lower cleaning efficiency than washing in combination with sulfonic acid and sodium lauryl ether sulfate.²⁶ The influence of the washing medium on the deodorization of several post-consumer plastic waste fractions was studied by Roosen et al.,²⁷ who found that the odor-removal efficiency of different washing media varied with different plastic waste fractions. For PE terephthalate bottles, washing with water and NaOH was the most efficient, but for polyolefin (PE, PP) samples the opposite was observed, since both a detergent and an organic solvent gave a better deodorization. The influence of washing conditions appears so far to have been studied primarily with hard packaging plastics. Studies on flexible packaging waste are rather scarce, and to our knowledge, the effects of washing conditions on the mechanical properties of flexible packaging waste have not been reported. This gap is addressed in the present work.

2 | MATERIALS AND METHODS

2.1 | Materials and chemicals

A batch of ca. 700 kg flexible PE packaging waste sorted from mixed residual waste was received in March 2021 from a large-scale waste sorting and recycling facility in Norway. Here, the mixed plastic stream was separated from metals and papers with a magnetic separator, an eddy current separator a set of near infrared sensors. The separated mixed plastic stream then went through ballistic separators and another sets of near infrared sensors to sort out flexible PE.²⁸ The purity of the flexible PE fraction was stated to be 96 wt.%.²⁸

Sodium hydroxide (NaOH) supplied by Sigma Aldrich (Merck) and a detergent (D) “Via professional liquid colour, perfume free” (Unilever Professional) were used as washing agents. The detergent contained 5%–15% anionic surfactants, <5% non-ionic surfactants and soap and <1% enzymes, phenoxyethanol, and methylisothiazolinone.

2.2 | Shredding and washing

The sorted plastics waste was shredded using a Rapid Granulator 300-45KU with a sieve size of 17 mm. The shredded flakes were then soaked, washed and dried prior to extrusion compounding.

A 1 kg sample of the shredded plastic waste was placed in a metal tub and soaked in 60 L of tap-water at room temperature, at a solid-to-liquid ratio of 1/60. The plastic flakes were manually agitated to improve wetting and then allowed to soak for 7 min. The floating fractions were collected. Two batches of the floating fraction were used in each washing cycle performed in a Vortex M6, SDL Atlas machine using 72 L of water. Each washing cycle lasted 45 min and included 15 min washing with agitation at 100 rpm, draining, 15 min rinsing with fresh water, draining and 4 min spinning at 600 rpm. The soaking, washing, and rinsing times were chosen based on earlier reported studies.^{24,26} The washing types were washing in water without any added agent, washing with 0.5% of NaOH in 72 L of water and washing with 0.4% of detergent having 0.05% active substances in 72 L of water. For each type of washing, the temperatures were 25°C (cold washing) and 40°C (moderate temperature washing). The rinsing temperature was 25°C for all the cycles. Three to four batches of the machine-washed flakes of each type were dried in a Moretto SX201 dryer at 60°C for at least 18–20 h until 4% RH was recorded by an EL-USB-2-LCD Lascar Electronics hygrometer inside the dryer.

2.3 | Extrusion compounding and injection molding

A Werner & Pfleiderer ZSK 30 M9/2 co-rotating intermeshing twin-screw extruder was used for compounding with an L/D of 32, the screw diameter being 30 mm. The screw configuration used, from the feeding to the metering section, was 7xC42/42, 2Xc28/28, 4xC20/20, 2xK45/5/28, C42/21, C42/42, C28/14, 2xC20/20, 2xK45/5/28, 4xC42/42, C28/28, 2xC20/10 and 4xC20/20.²⁹ The extruder cylinder had five heating zones and a die section, and the temperature profile was set to 100, 150, 200, 240, 240°C in the five zones and 250°C in the die. This temperature profile was the same for each type of washed sample and is referred to as high-temperature (HT) compounding. The sample washed with NaOH at 40°C was also extruded using a temperature profile of 100, 150, 200, 200, 200°C through the barrel and 210°C at the die, referred to as low-temperature (LT) compounding. The plastic flakes

were fed into the extruder by hand at a rate of 1.8 ± 0.3 kg/h with a screw rotation rate of 80 rpm. The compounded strands were granulated into pellets using a Dreher pelletizer type SG10Ni. About 2.4 ± 1.2 kg of pellets were produced for each type of sample.

An Arburg Allrounder 221M-250-5 machine was used for the injection molding to produce frame-shapes samples, providing three sample regions with different molecular orientations.²⁹ The injection molding was conducted with a temperature profile of 120, 170, 200, and 220°C along the barrel and 220°C at the nozzle. The injection and holding pressures were 500 and 700 bar, respectively. The injection volume was adjusted to ensure at least 80% meeting of the weld line width before the holding pressure was applied. The notations of the specimens produced are given in Table 1 with their processing details. The properties of the laboratory-scale washed materials produced were compared with those of the unwashed (UW) material and large-scale high-intensity washed (IW) materials from the same sorted fractions reported in our previous paper.²⁹

2.4 | Characterization

Differential scanning calorimetry (DSC) with a Mettler-Toledo DSC 2 was used to determine the thermal transitions and the oxidation induction temperatures (T_{ox}), according to ISO11357-1 and ISO 11357-6, respectively. Both compounded pellets and injection-molded samples were characterized. Air was used for purging when determining the T_{ox} and nitrogen when assessing the thermal transitions, both at a heating rate of 10°C/min. Duplicate measurements were made in all cases. For the melting enthalpy (ΔH) the baseline was taken from 60 to 134°C. The results are reported for the first heating cycles.

The ash content of powdered samples was determined at 550°C by thermogravimetric analysis using a thermogravimetric analysis/DSC 3+ Star system from Mettler Toledo. A 3.1 ± 0.04 mg sample was heated from 25 to 650°C at a rate of 10°C/min in air at a flow rate of 50 mL/min. Duplicate measurements were made for each type of material.

The molar masses of selected samples were assessed by HT gel permeation chromatography (HT-GPC) after dissolution at a concentration of 3 mg/mL in 1,2,4 trichlorobenzene with 200 ppm butylated hydroxytoluene as antioxidant. The masses were determined at 160°C, using a Polymer Laboratories GPC220 instrument with PlOlexis and PlOlexis guard columns with lengths of 3×30 cm, an injection volume of 200 μ L, and a flow rate of 0.8 mL/min. Data were recorded and analyzed using Polymer Laboratories Cirrus software. The weight

average molecular mass (M_w) and polydispersity index (PDI) were calculated from two independent measurements except in the case of sample NaOH40 which had one measurement.

A Ceast Modular Melt Flow instrument was used to determine the melt mass-flow rate (MFR) of the pellets, using a standard weight of 2.16 kg at 190°C in accordance with ISO 1133-1:2011. The rheological behavior of the melts from pellets obtained after compounding was evaluated using a High-pressure Rheograph 20 capillary rheometer (Göttfert) at 220°C using a constant piston speed at each shear rate between 10^3 and 10^1 s^{-1} . Three dies were used, having diameters of 2 mm and aspect ratios (L/D) of 5, 10, and 15 for the application of the Bagley correction according to ISO 11443:2021. The Weissenberg-Rabinowitsch shear-rate

correction was also applied. The entrance pressure losses were evaluated using Bagley plots.

The elongational properties of the melts were determined using the same capillary rheometer equipped with a haul-off unit consisting of a strand wheel connected to a force transducer and a take-off wheel. ISO16790:2021-02 was used as a guideline for the measurements made at a melt temperature of 220°C in an ambient environment of $23 \pm 1^\circ\text{C}$ and $26 \pm 4\%$ relative humidity, using a capillary with a diameter of 2 mm and a length of 20 mm. The piston speed was kept constant at 0.2 mm/s, which corresponds to an initial velocity of the extruded strand at the exit of the capillary (v_0) of 11.25 mm/s, and the starting tangential speed of the take-off wheel (v_1) was 17.5 mm/s for the UW and laboratory-scale washed samples, but 25 mm/s for the large-scale high-IW samples. The speed of the take-off wheel was increased at a rate of 1.2 mm/s^2 . The force required to extend the melt was measured with a transducer and recorded together with the time and velocity of the take-off wheel until melt fracture. The strain (ϵ) values were taken as $\epsilon = (v_1 - v_0)/v_0$.³⁰ The melt strength was measured on the laboratory-scale washed samples and also on the UW and large-scale high-IW samples, described in our previous study.²⁹ The mean values of the melt strength and of the strain-at-break were based on at least six measurements.

The tensile properties of the injection-molded samples were measured with a Zwick/Z2.5 instrument equipped with a 2 kN load cell. Test bars were cut from the three different regions of the molded frames, as shown in Figure 1, using an Elastocon EP 04 ISO 37-2 cutting die, corresponding to specimen type 5A of ISO 527-2:2012 and conditioned according to ISO 527-1:2012. The Young's modulus, the tensile strength, and the elongation-at-break were evaluated at a strain rate of 1 min^{-1} in an ambient environment of $22 \pm 1^\circ\text{C}$ and $35 \pm 7\%$ relative humidity. The averages and standard deviations were based on five independent measurements.

TABLE 1 The processing conditions and sample notations.

Sample notation	Washing temperature (°C)	Washing medium	Compounding temperature profile
H ₂ O25	25	Water	HT
H ₂ O40	40	Water	HT
NaOH25	25	Water, NaOH	HT
NaOH40 (200)	40	Water, NaOH	LT
NaOH40	40	Water, NaOH	HT
D25	25	Water, D	HT
D40	40	Water, D	HT
UW (200) ^a	–	–	LT
UW ^a	–	–	HT
IW (200) ^a	70–80	Water, NaOH, D, and other additives	LT
IW ^a	70–80	Water, NaOH, D, and other additives	HT

Abbreviations: HT, high-temperature; IW, intensity washed; LT, low-temperature; UW, unwashed.

^aPrepared in our previous study.²⁹

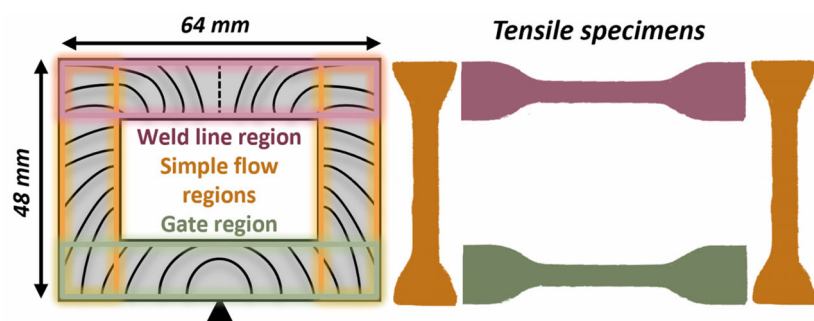


FIGURE 1 Schematic representation of the injection molded frame with a flow pattern and the tensile specimens cut from different regions, thickness is 2 mm.

3 | RESULTS AND DISCUSSION

The properties of UW and large-scale high-IW materials produced in our previous work were also involved in Table 2 and Figures 3–5 for comparison with the laboratory-scale washed materials produced in this study.

The first heating endotherms of the pellets (P) and of the injection-molded samples (IM) are shown in Figure 2, and the T_{ox} , ash contents, MFR , M_w , and PDI are given in Table 2.

All the samples exhibited a main endotherm peak at 125°C, with a shoulder at 111°C and a small peak at 161°C. The main peak with a shoulder was related to the different grades of PE, T_{p1} to LDPE and T_{p2} to medium-density PE and HDPE, and the small peak T_{p3} was attributed to PP.^{31–33} There were no significant differences in thermal properties of compounded pellets and IM samples as shown in Figure 2. Neither the washing medium nor the washing temperature had a significant effect on the melting characteristics, but the ΔH was slightly lower after NaOH washing. Santana and Gondim²⁴ reported that NaOH washing leaves alkaline residues which cause some degradation, and NaOH washing may also leave oxidative moieties which hinder the crystallization.³⁴

In the case of the laboratory-scale washed samples, the oxidation temperature (T_{ox}) was apparently unaffected by the washing medium and all the samples had a T_{ox} between 225 and 233°C which was much higher than expected of unstabilized virgin PE ($180 \pm 5^\circ\text{C}$) and implied that the washed material still contained a significant residual amount of active stabilizer.³⁵ The laboratory-scale washed samples exhibited T_{ox} values

similar to those of the UW samples (231°C), but the large-scale high-IW samples showed much lower T_{ox} values (184–195°C), which indicated a significant loss or inactivation of stabilizers.

Neither the washing medium nor the washing temperature affected the ash content. The measured values were intermediate between those of the UW (11%) and large-scale high-IW (5%) samples reported in the previous study.²⁹ After laboratory-scale washing, the ash content was reduced, as expected.^{33,36,37}

Neither the M_w nor the PDI were affected by the washing conditions on a laboratory-scale, being between 92,000 and 112,000 g/mol and between 3.3 and 5.8,

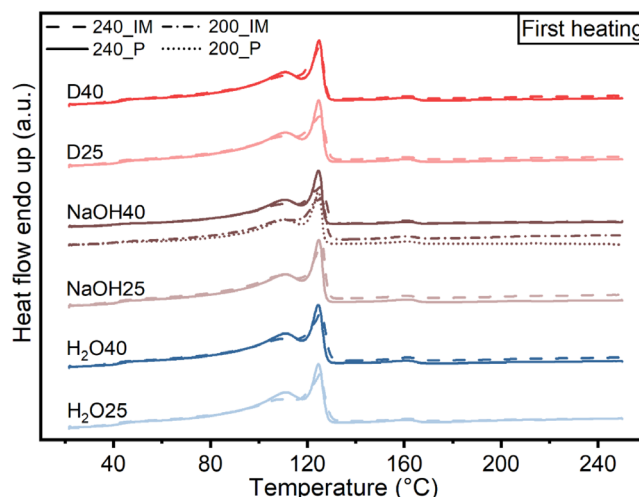


FIGURE 2 The first heating endotherms of the samples studied. IM, injection-molded; P, pellets.

TABLE 2 Thermal, structural, and rheological properties of the compounded pellets.

Sample	T_{p1} (°C)	T_{p2} (°C)	T_{p3} (°C)	ΔH (J/g)	T_{ox} (°C)	Ash content (%)	M_w (g/mol)	PDI	MFR (g/10 min)
UW ^a	111	125	161	69	231	11	122,000	5.0	0.6
H ₂ O25	111	125	161	105	227	7	101,000	3.6	0.8
H ₂ O40	111	125	161	102	225	7	96,000	4.6	0.8
NaOH25	111	125	162	99	229	8	95,000	3.3	0.6
NaOH40	111	125	162	85	231	7	112,000	5.8	0.7
D25	111	125	161	103	230	7	92,000	4.3	0.8
D40	111	125	162	98	227	7	99,000	4.5	0.8
IW ^a	113	123	160	76	184	5	71,500	4.4	3.9
UW (200) ^a	111	125	162	68	232	11	124,500	5.2	0.5
NaOH40 (200)	111	125	162	83	232	7	95,000	4.8	0.6
IW (200) ^a	113	126	161	76	195	5	87,000	4.1	2.4

Abbreviations: IW, intensity washed; MFR , melt mass-flow rates; M_w , weight average molecular mass; PDI , polydispersity index; T_{ox} , oxidation induction temperatures; UW, unwashed; ΔH , melting enthalpy.

^aData taken from Boz Noyan et al.²⁹

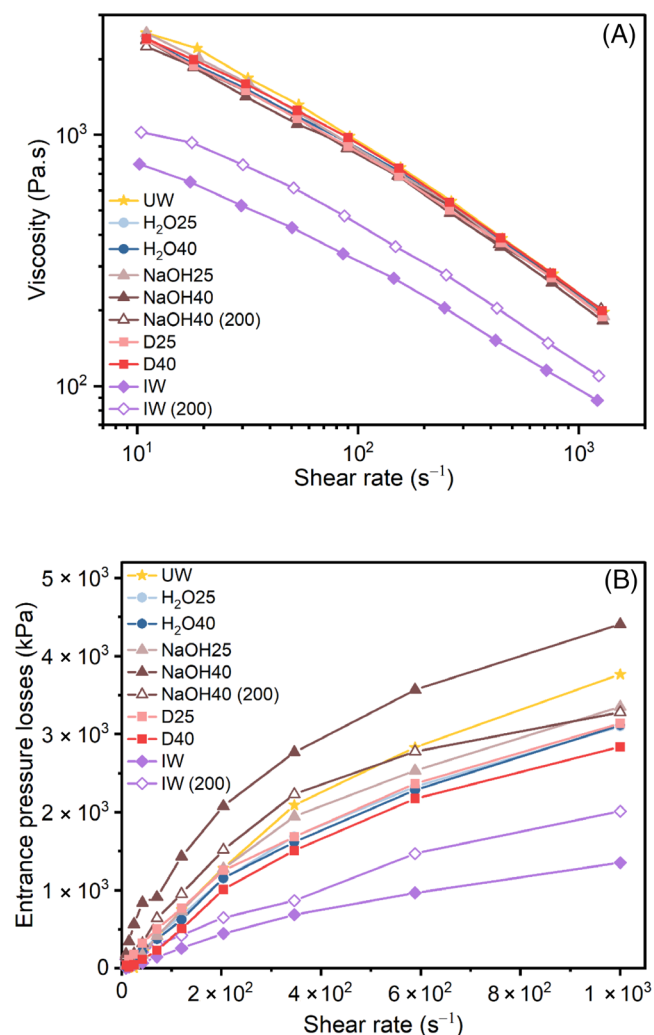


FIGURE 3 (A) Viscosity versus shear rate at 220°C, (B) Entrance pressure loss versus shear rate at 220°C. IW, intensity washed; UW, unwashed.

respectively. The laboratory-scale washed samples exhibited M_w values intermediate between those of the UW samples and the large-scale high-IW samples. The lower values of both M_w and PDI after laboratory-scale washing suggest that chain scission was the predominant degradation mechanism.^{34,38,39} The M_w was much lower after the large-scale high-intensity washing.

The MFR were similar for all the laboratory-scale washed and the UW samples, between 0.5 and 0.8, but the rates for the large-scale high-IW samples were much higher which corresponded well with the lower M_w values.

Figure 3A shows the viscosity as a function of the shear rate and Figure 3B shows the entrance pressure loss versus the shear rate.

The laboratory-scale washed samples and the UW sample had similar viscosities over this shear rate region, supported by the measured MFR and M_w values given in

Table 2. The large-scale high-IW samples had much lower viscosities.

A higher entrance pressure loss (Figure 3B) indicates a more elastic melt.⁴⁰ The elasticity affects the melt behavior during processing,^{41–43} and a more elastic melt results in a higher die swelling ratio during extrusion.^{44,45} Iannuzzi and Rigdahl⁴⁶ reported that the greater melt elasticity of PP-compounds may be one of the factors reducing the appearance of flow marks during injection molding and providing a more stable melt flow in draw-down experiments. Montes⁴⁷ noted that haze formation increases during film blowing when the extrudate swell increases.

In the present study, the NaOH₄₀ sample exhibited the greatest and the IW sample had the lowest entrance pressure loss, although all the NaOH-washed samples exhibited rather high entrance pressure losses.

The force-strain plots measured for some of the melts are shown in Figure 4A, and the melt strengths and strains-at-break are given in Figure 4B. The curves in Figure 4A illustrate the drawability when the material leaves the extrusion die. In the case of the samples extruded with the HT profile, the UW sample had the lowest strain-at-break with an average of about 380%, whereas the large-scale high-IW sample had the highest value with an average of 2190%. The laboratory-scale washed samples had intermediate values, where the NaOH-washed samples showed the lowest strain-at-break. The melt strength values were in the same range for the laboratory-scale washed samples but the UW and IW samples had lower melt strengths.

The samples showed the same trend when compounded to an LT profile. The IW sample showed the highest melt extension-to-break and the lowest melt strength, while the UW sample had the lowest extension-to-break and a low melt strength. The NaOH₄₀ sample exhibited a rather low extension-to-break and the highest melt strength.

The UW samples had a low ultimate strength and a low extension-to-break. In general, washing improved the extensional properties of the melts. Washing solely with water or with a detergent had similar effects, which may be related to a lower level of contamination. The strain-at-break of the NaOH-washed specimens was much lower than that of the samples washed with water alone or with a detergent, despite similar ash contents. This may indicate a change in polymer properties and structure due to the NaOH treatment, probably associated with a degradation, but this requires a more detailed study. In the case of the IW samples, a change in polymer properties due to the washing may explain the low melt strength and enhanced extensibility. The NaOH-washed samples exhibited the greatest and the IW samples the

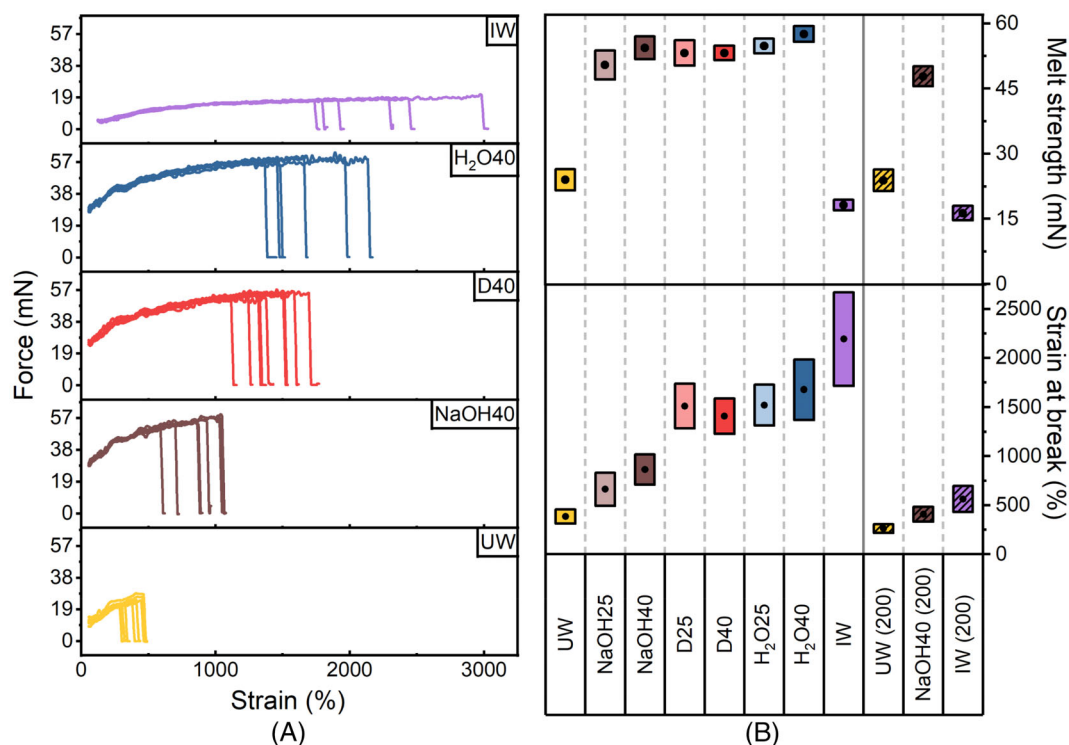
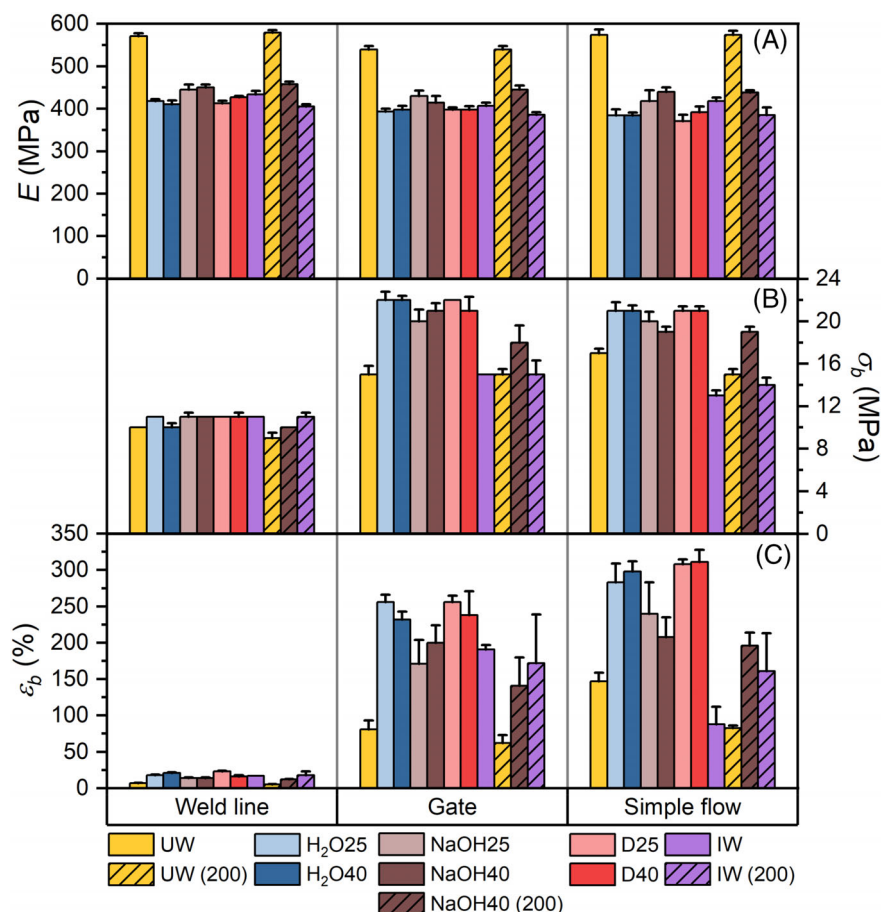


FIGURE 4 (A) Force-strain curves measured for some of the melts at 220°C, (B) Melt strength and strain-at-break of the melts; where a dot in a box indicates the average value and the standard deviation is indicated by the length of the box. IW, intensity washed; UW, unwashed.

FIGURE 5 Mechanical properties of the samples in the different regions of the IM frame: (A) Young's modulus, E , (B) tensile strength, σ_b , and (C) elongation at break, ϵ_b . IW, intensity washed; UW, unwashed.



lowest entrance pressure losses (cf. Figure 3). The washing temperature (25 or 40°C) had a negligible effect on the extensional behavior of the melts.

Figure 5 shows that the mechanical properties (Young's modulus, E , tensile strength at break, σ_b , and elongation at break, ε_b) of the molded samples in the different regions of the IM frame varied substantially after washing.

There were only small differences in Young's modulus between the different regions of the IM samples, but both laboratory-scale and large-scale high-intensity washing reduced Young's modulus, see Figure 5A. The samples washed in the laboratory with NaOH showed slightly higher Young's modulus values than those washed with water alone or with a detergent, perhaps because NaOH washing led to a slightly lower crystallinity.⁴⁸ In general, neither the washing temperature nor the compounding temperatures had any great impact on the modulus of elasticity.

The strength-at-break values, Figure 5B, differed between different regions of the IM samples. As expected, the weld line regions had the lowest strength, at 10–11 MPa, but the values were nevertheless close to the reported value (13 MPa) for virgin linear low-density PE.⁴⁹ The laboratory-scale washing increased the tensile strength in both the gate and simple flow regions, but the large-scale high-intensity washing had no influence on the gate region and resulted in a lower strength in the simple flow region. In the laboratory-scale washed samples, NaOH gave a slightly lower tensile strength than water alone or water with added detergent. The laboratory-scale washing temperature had no great influence on the tensile strength, but the compounding temperature had some influence. In the simple flow region, the UW sample had a lower strength whereas the large-scale high-IW sample had a slightly higher strength when LT compounding was used.

The elongation-at-break, Figure 5C, was very different in the different regions of the molded frames, being lowest in the weld line region. Washing increased the elongation-at-break. The UW samples had the lowest elongations, probably due to the presence of impurities that created more stress concentrations.⁵⁰ The large-scale high-IW samples had a lower elongation-at-break than the laboratory-scale washed samples, probably due to the greater molecular degradation indicated by the T_{ox} , M_w , and viscosity data. In the case of laboratory-scale washing, similar changes in elongation-at-break were observed in all the regions, the NaOH washing resulting in the lowest average elongation-at-break and washing with water alone or with a detergent giving similar elongation-at-break levels. The washing medium affected the ductility but the washing temperature had no major impact. The compounding temperature had some influence. LT compounding resulted in lower elongation-at-break values except in the case of the large-scale high-IW sample in the simple flow region.

In general, the slightly different tensile properties with NaOH washing implied that the samples were differently degraded. The lower extensibility of the NaOH-washed melts, Figure 4B, was also reflected in the lower elongation-at-break in the case of the molded frames.

4 | CONCLUSIONS

The laboratory-scale washing led to specimens with structural and rheological properties intermediate between those of the UW and the large-scale high-IW samples. The mechanical properties of the samples were improved by washing, but there were indications of possible degradation when more severe conditions, for example, a higher washing temperature, were applied with large-scale high-intensity washing, especially in combination with high temperature compounding. In the case of the laboratory-scale washed samples, the washing medium had a significant influence on most of the properties. Washing with water alone or with a detergent gave similar mechanical properties but washing with NaOH resulted in a higher melt elasticity, a slightly higher stiffness, and a slightly lower strength in the molded samples and a much lower extensibility, both in the molten and solid states, indicating some degradation of the polymer. The washing temperature of 25 or 40°C was not, however, significant.

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DATA AVAILABILITY STATEMENT

Research data are not shared.

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