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Investigation of Pretreatment and Melt Filtration in Mechanical Recycling of Polyethylene Film Waste Streams

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ABSTRACT

Efficient recycling of polyethylene (PE) waste depends on optimized pretreatment and processing strategies. This study presents a systematic evaluation of how washing conditions (temperature, time, and NaOH concentration) and melt filtration affect the quality of recyclates from three industrially relevant PE waste streams: food-grade stretch film (rPE LLD), post-consumer PE low-density (rPE-LD), and mixed PE films (rPE mix). Unlike previous studies, this work combines laboratory-scale washing and twin-screw extrusion, including single and double melt filtration, with comprehensive material characterization. Results reveal that washing significantly influences melt mass-flow rate (MFR), oxidation onset temperature (OOT), and tensile strength positively and negatively, while melt filtration primarily improves optical quality and reduces volatile organic compounds (VOCs). Puncture resistance was largely unaffected. The most substantial property differences stem from the heterogeneity of the input streams. These findings demonstrate that tailored washing, using cold water without NaOH for rPE-LLD, NaOH-assisted cold washing for rPE-LD and rPE mix, can enhance quality while minimizing degradation. A second melt filtration step is recommended when optical clarity is critical, though its thermal impact must be considered. This study provides guidance for industrial recyclers seeking to optimize process settings based on input stream characteristics and application-specific quality requirements.

1 | Introduction

Flexible packaging, encompassing films and bags, has gained popularity due to its numerous benefits. Its ability to reduce material usage makes it lightweight for convenient storage and transportation. Flexible packaging protects the contents from external factors without causing dents or visual defects. In addition, its adaptability to conform to product shapes and its cost-effectiveness compared to non-polymeric counterparts underscore its advantages [1–4]. However, if the flexibles are not handled properly, they can cause major environmental problems at the end of their life [5–7].

To address this challenge, the European Union (EU) has issued a directive [8] to achieve specific recycling targets for plastic packaging of 50% by the end of 2025 and a further target of 55% by the end of 2030. Currently, Europe is transitioning from a linear economy to a circular economy. The post-consumer plastic packaging waste recycling rate in Europe lies at around 38%, using the new calculation point where materials enter pelletizing, extrusion, and molding processes; therefore, after impurities and unsuitable substances have been removed from the sorted materials [9].

To achieve the above-mentioned recycling targets, the recycling rate must be increased tremendously. This rate is calculated using three different efficiencies: the collection efficiency, the sorting efficiency, and the recycling efficiency [9, 10]. To achieve not only higher recycling rates but also good quality, the recycling process needs to be investigated and further improved. One key step for improving the recycling process is pretreatment. Conventional pretreatment steps include sorting, washing, and shredding, not necessarily in this order [11, 12]. The main pretreatment step investigated in this paper is washing.

Temperature, time, and detergent are the main setting parameters that are varied in the pretreatment step for recycling. In general, literature reviews indicate that the influence of the water temperature is trivial compared to other washing parameters [13–15]. Others have reported that washing for a longer time greatly increases the washing efficiency [16, 17]. Longer washing time allows for more thorough cleaning and removal of contaminants, making it a highly influential variable that recyclers must carefully optimize. Additionally, other authors have found that the influence of the washing solvent also plays a significant role. For example, NaOH has been reported to have a poor influence on polyolefins, but is still used in industry since it is a good way to remove grease, VOCs, and odors from recycled materials [14, 18–20].

A major influence on the quality of recyclates comes from the composition of the input stream itself [21–23]. The adage "garbage in, garbage out" certainly applies, as the inherent quality and consistency of the collected plastics determine what can be achieved in the recycling process. Films and multilayer packaging are known to be a difficult stream to recycle due to their heterogeneous nature [24, 25]. Recyclers must therefore carefully manage the incoming waste stream to minimize contamination and optimize the final properties of the recycled material.

As shown, different authors have addressed the issue of flexible film recycling and its pretreatment. In the following, some publications are discussed in more detail. Soto et al. [14] conducted a study to evaluate the feasibility of a washing process to clean post-consumer polyethylene (PE) film from municipal solid waste. The authors evaluated several parameters, including washing temperatures and media (room temperature with water, 60°C with water, and 60°C with water and NaOH), prewash drying, and water treatment. Their results showed minimal differences between room temperature and 60°C washing. However, the inclusion of NaOH resulted in the most effective chemical cleaning, albeit with challenges in wastewater treatment. The results also showed that drying before washing significantly improved the decontamination of post-consumer PE film, reducing the consumption of fresh water and the need for disposal. However, the authors mainly investigated the influence of the washing parameters on the wash water. No information on the effect of the washing process on the recyclates produced from the post-consumer PE film is given.

Boz et al. [26] used flexible PE packaging waste from municipal solid waste and washed it on a laboratory scale at 25°C and 40°C with water, with water containing NaOH, and with water and another detergent added. The washed materials were then converted using a twin-screw extruder and injection molded. The results were compared with those for an unwashed sample and with a sample from a large-scale high-intensity washing with NaOH at 70°C–80°C. The mechanical properties were influenced by washing. However, the thermal properties were less affected. In conclusion, trade-offs between washing conditions and degradation need to be considered for optimized flexible packaging recycling. The authors used low washing temperatures and comparatively low NaOH amounts for their lab samples. Furthermore, the washing machine's washing process was relatively long, at 45 min, compared to industrial washing times. To investigate the mechanical behavior, ISO 527-2 type 5A specimens were injection molded, which indicates injection molding applications as the goal for the produced recyclates.

Furthermore, Boz et al. [27] investigated PE flexible packaging waste from mixed municipal solid waste from a recycling plant in Norway. The waste was analyzed at three different points of the industrial recycling process: i) directly after sorting, ii) after sorting and washing, and iii) after sorting, washing, and melt-compounding, including melt filtration. The samples in flake form (unwashed and industrially washed) were further processed on a laboratory scale. Industrial washing consisted of prewashing at room temperature, wet grinding, friction washing with NaOH and defoamer at 70°C–80°C, rinsing at room temperature, centrifuging, dewatering, and thermal drying. The washed flakes were then industrially compounded, including filtration with a 120 μm mesh screen, and pelletized. The unwashed and industrially washed samples were laboratory compounded and then injection-molded, whereas the industrially recycled sample was injection-molded as it was received. The melt-processing parameters had a small influence on the properties of molded samples, and the influence of the compounding temperature was greater than that of the screw configuration for both the unwashed and industrially washed samples. No differences in the degradation of the unwashed samples as a result of different melt-processing parameters were observed. Washing led to a significant degradation, leading to further degradation in the subsequent melt-compounding. The mechanical properties implied that the material would be useful for suitable applications. The authors focused on molded products, and no information concerning the use of the recyclates for film applications was given.

This research aimed to study the optimization of mechanical recycling processes. Since other studies have only looked at single waste streams, the washing performance concerning the water quality, or the end application being injection molding products, this study focuses on the effect of the washing process in combination with melt filtration on key performance indicators. For this purpose, three different post-consumer waste streams were investigated and processed into films, which were further investigated.

2 | Materials and Methods

2.1 | Sample Description

This study used three different waste streams. The first consisted of stretch film derived from commercial waste collected from a grocery store. These films were originally used to package pallets containing food products; the main polymer in this fraction was PE linear low-density (PE-LLD). Throughout the study, samples from this waste stream will be referred to as "rPE-LLD". The second waste stream originated from Upper Austrian collection centers operated by the O.Ö. Landes-Abfallverwertungsunternehmen GmbH (LAVU). These PE low-density (PE-LD) films have a surface area of more than 1 m^2 and consist mainly of packaging materials from furniture, construction, and agricultural products [28]. Samples from this waste stream are referred to as "rPE-LD". The third waste stream is the sorted film fraction stemming from the curbside collection "yellow bag", which collects the lightweight waste fractions in Austria, such as plastic packaging, metal cans, or composite materials. The sorted film fraction is specified by "Duales System Deutschland (DSD) GmbH" as DSD 310. This fraction mainly consists of used, residue-emptied PE plastic foil with an area larger than DIN A4, such as bags, shrink films, including closures, labels, etc. with a purity of at least 92% [29]. This fraction is further called "rPE mix".

2.2 | Pretreatment

To investigate the influence of the pretreatment on the recyclate properties, the following steps have been taken: First, the material was shredded to a flake size of approximately 30 mm using a Micromat 1500 shredder (Lindner-Recyclingtech GmbH, Spittal/Drau, Austria) and further washed using a Miele PW818 EL WEK washing machine (Miele GmbH, Wals, Austria) in 2 kg batches. Additionally, for some samples, sodium hydroxide (also known as caustic soda, NaOH) was added. The machine used 35 L of water, 2 kg of material, and 2.5 w% of NaOH (purity 99%, (Algin, Neustadt-Glewe, Germany)) was added, resulting in 875 g added directly into the washing machine. The washing parameters studied are shown in Table 1.

After washing, the material was dried in a Binder FED 56 heating oven (Binder GmbH, Tuttlingen, Germany) at 60°C for 4 h. Furthermore, to ensure a constant feed into the compounder, the material was compressed into pellet form using a PP200C pellet press (EverTec, Dieburg, Germany) with a matrix plate having a die hole diameter of 6 mm.

TABLE 1 | Investigated washing parameters for the three waste streams rPE-LLD, rPE-LD, and rPE mix.

Sample	Temperature in °C	Time in min	NaOH in %
A	25	5	0
B	25	5	2.5
C	25	15	2.5
D	80	5	0
E	80	5	2.5
F	80	15	2.5

2.3 | Melt Filtration in the Extrusion Process

To investigate the influence of further processing steps such as melt filtration, the material was processed through a laboratory scale twin-screw extruder ZSE 18MAXX (L/D = 50/1, Leistritz AG, Nuernberg, Germany). A very coarse mesh filter (mesh size 18 = 1 mm hole size) was used for the first pass to remove coarse contaminations, and a higher mesh size filter (mesh size 30 = 0.6 mm hole size) was used for the second pass. The compounder was operated at a screw speed of 300 rpm with a throughput of approximately 2.5 kg/h. The temperature of the compounder was set at 25°C in the feed zone and then steadily increased from 170°C to 210°C in the following twelve zones. Samples of the pellets were taken after each compounding step. It is noteworthy that the filtration processes under investigation are not directly comparable to commercially available industrial double filtration processes. In the latter case, material typically passes through two filter systems within a single heating and melting process. For this study, the material underwent two process steps involving heating, filtration with two different filter sizes (coarse and fine), and cooling.

2.4 | Characterization

2.4.1 | Melt Mass-Flow Rate (MFR)

MFR measurements were performed using the Aflow (ZwickRoell GmbH and Co. KG, Ulm, Germany) according to ISO 1133, with a test weight of 2.16 kg and 5.00 kg using a measurement temperature of 190°C. Approximately 3 g of material was loaded into the preheated cylinder of the Aflow, where it was compacted and heated for five minutes. The measurement process began automatically as the piston forced the molten material through the die. Six samples were taken from the center strand and weighed. Each of these six samples was weighed twice for accuracy.

2.4.2 | Oxidation Onset Temperature (OOT)

OOT measurements were performed using a DSC 4000 instrument (Perkin Elmer Inc., Waltham, MA, USA) according to ISO 11357-6. Samples weighing 5.0 ± 0.5 mg were heated from 30°C to 280°C at a rate of 10 K/min. The samples were heated continuously in an air atmosphere until a characteristic temperature was reached, indicating the onset of an exothermic event and oxidative degradation, as indicated by a baseline shift.

2.4.3 | Ash Content

Ash content analysis was performed according to ISO 3451-1 standard procedures. The ashing process is done using quartz fiber crucibles and a Phoenix microwave muffle furnace (CEM Corporation, Matthews, North Carolina, USA). Approximately 3 g of material was placed in each crucible and subjected to

TABLE 2 | Summary of the parameters of the automated thermal desorber (ATD) coupled to a gas chromatograph including a flame ionization detector (GC/FID) and mass spectrometer (MS).

ATD parameters		GC/FID-MS parameters	
Mode	**2 stage desorption**	**Column**	**HP Ultra 2**
Column flow [psi]	130	Column size	50 m x 0.32 mm; 0.52 µm
Desorption flow [mL/min]	40	FID temperature [°C]	300
Inlet split flow [mL/min]	44	H_2 [mL/min]	30
Outlet split flow [mL/min]	19	Synthetic air [mL/min]	450
Trap temperature [°C]	−30-280	Transfer line to MSD [°C]	280
Heating rate [K/s]	99	Ion source [°C]	250
Trap hold [min]	20	Temperature program:	
Valve temperature [°C]	280	Start temperature	60°C, 13 min
Transfer line temperature [°C]	290	Ramp 1	6 K/min to 215°C
		Ramp 2	25 K/min to 280°C, 12 min hold
		Scan mode	29–450 amu
		MS solvent delay [min]	4.5

direct calcination in the muffle furnace at 750°C for 15 min. After calcination, the crucibles were weighed, and the ash content was calculated using Equation (1):

$$A\% = \frac{m_1}{m_0}\, x\, 100 \tag{1}$$

Where $A\%$ is the resulting ash content, m_0 is the initial mass of the test sample, and m_1 is the measured mass of the obtained ash. Each of the six samples was tested three times.

2.4.4 | Optical Contamination Detection (OCD)

OCD was performed using an ME30 measuring single screw extruder (L/D = 26/1) and an FSA100 modular film analyzer (OCS Optical Control Systems GmbH, Witten, Germany). Extruder settings included a temperature of 30°C in the feed zone, gradually increasing from 180°C to 200°C in zones 1 to 6. The screw speed was maintained at 18 rpm, and a 150 mm x 1 mm sheet extrusion die was used. The chill rolls were set at 30°C at a speed of 5.6 rpm, while the film was held at a tension of 6 N and rewound at a force of 7 N. Contamination measurements were performed with a base grayscale value of 180, with the measurement system triggered upon detection of a 20% deviation (darker). To ensure optimal camera detection, all samples were blended with PE linear low density (PE-LLD) at a ratio of 90% virgin to 10% recycled material.

2.4.5 | Volatile Organic Compounds (VOCs)

The analysis of volatile organic compounds (VOCs) in the produced recyclates was conducted using a Clarus 690 SQ 8 gas chromatograph, including a flame ionization detector and coupled with a mass spectrometer (GC/FID-MS) from PerkinElmer Inc. (Waltham, MA, USA). Additionally, the instrument was equipped with a TurboMatrix 650 automated thermal desorption unit (ATD) from the same manufacturer, with helium gas serving as the carrier flow. Samples, sized 1.5 mm x 3 mm x 14 mm (T x D x L), were punched from previously hot-pressed small plates. These plates were produced on an Atlas 15T press (Specac Ltd., Orpington, UK) at approximately 210°C for 5 min. To extract VOCs from the solid polymer matrix, the samples underwent heating in the ATD at 90°C for a duration of 30 min. Subsequent separation of VOCs in the GC was enhanced by employing a specific temperature program and parameters outlined in Table 2. To obtain semi-quantitative results, a one-point calibration reference was executed before measurements. This calibration process involved injecting 4 µL of a 0.5 µg/µL toluene in methanol solution via a Control Solution Loading Rig (CSLR, Markes International) under a nitrogen atmosphere (100 mL/min) onto a tube containing the adsorbent TENAX TA. The tube was then left on the CSLR for 6 min to remove as much methanol solvent as possible. This calibration process generated a response factor (Rf), representing the ratio between the mass of toluene (m_{ref}) in micrograms (µg) and the corresponding peak area (A_{ref}) as described in Equation (2) [30].

$$Rf = \frac{m_{ref}}{A_{ref}} \times 10^6 \tag{2}$$

Moreover, the semi-quantitative VOCs in µg/g were determined by multiplying Rf by the ratio of the peak area of the sample (A_s) to the sample mass (m_s) in milligrams (mg) as expressed in Equation (3) [30]. For each sample, two measurements were performed.

$$VOCs = Rf \times \frac{A_s}{m_s} \times 10^{-3} \tag{3}$$

2.4.6 | Tensile Properties

During the OCD measurements, films were produced and subsequently used for tensile testing. Specimens of type 5 were punched using a universal toggle-lever press (Gechter GmbH, Obermichelbach, Germany), and ten specimens were tested in accordance with ISO 527-3 [31]. The stress–strain diagrams were recorded using a zwickiLine Z2.5 testing machine (ZwickRoell GmbH und Co. KG, Ulm, Germany) equipped with a load cell with a nominal force of 500 N [19]. A preload of 0.1 MPa was applied at the beginning of the test. All specimens were tested at a rate of 50 mm/min until failure by fracture occurred.

2.4.7 | Puncture Resistance

Puncture tests according to EN 14477 [32] were performed on the film samples produced by OCD. The testing machine used was the same zwickiLine Z2.5 (ZwickRoell GmbH and Co. KG, Ulm, Germany) with a load cell with a nominal force of 500 N. All specimens were tested ten times at a test speed of 50 mm/min until failure by breakage occurred.

3 | Results

3.1 | rPE-LLD

Figure 1 shows the results originating from the chosen characterization methods for the rPE-LLD film samples. Samples A, B, and C, which were washed at the lower temperature of 25°C, exhibit notably higher MFR values (around 1.4 g/10 min) compared to samples D, E, and F (around 1.0 g/10 min), which underwent washing at the higher temperature of 80°C. Particularly for the 25°C washed samples (A, B, and C), the MFR values remain rather constant regardless of differences in washing time and NaOH content. In contrast, the 80°C washed samples (D, E, and F) show a more pronounced drop in MFR, indicating that the higher temperature may be causing changes to the material. A significant drop to 0.9 g/10 min can be seen in sample E when NaOH is added. Importantly, the data reveal no major differences in MFR across samples A, B, and C processed and filtered once (black curve) and processed and filtered twice (red curve). Only slight deviations between the first and the second process can be noticed when the pretreatment is performed at higher temperatures (samples D, E, and F).

Samples A and B show that prolonging the washing time allows for an OOT increase (from 231.2°C to 233.4°C), while the comparison of samples B (233.4°C) and C (237.8°C) shows that an even higher OOT increase is possible when NaOH is added. On the other hand, samples D (232.8°C), E (231.9°C), and F (230.8°C) display rather constant OOT values over the different washing parameters. Furthermore, there are no distinct differences in OOT measurements between the samples processed and filtered once or twice.

The ash content analysis provides additional insights. For samples A, B, and C, the ash content is lower after the second melt

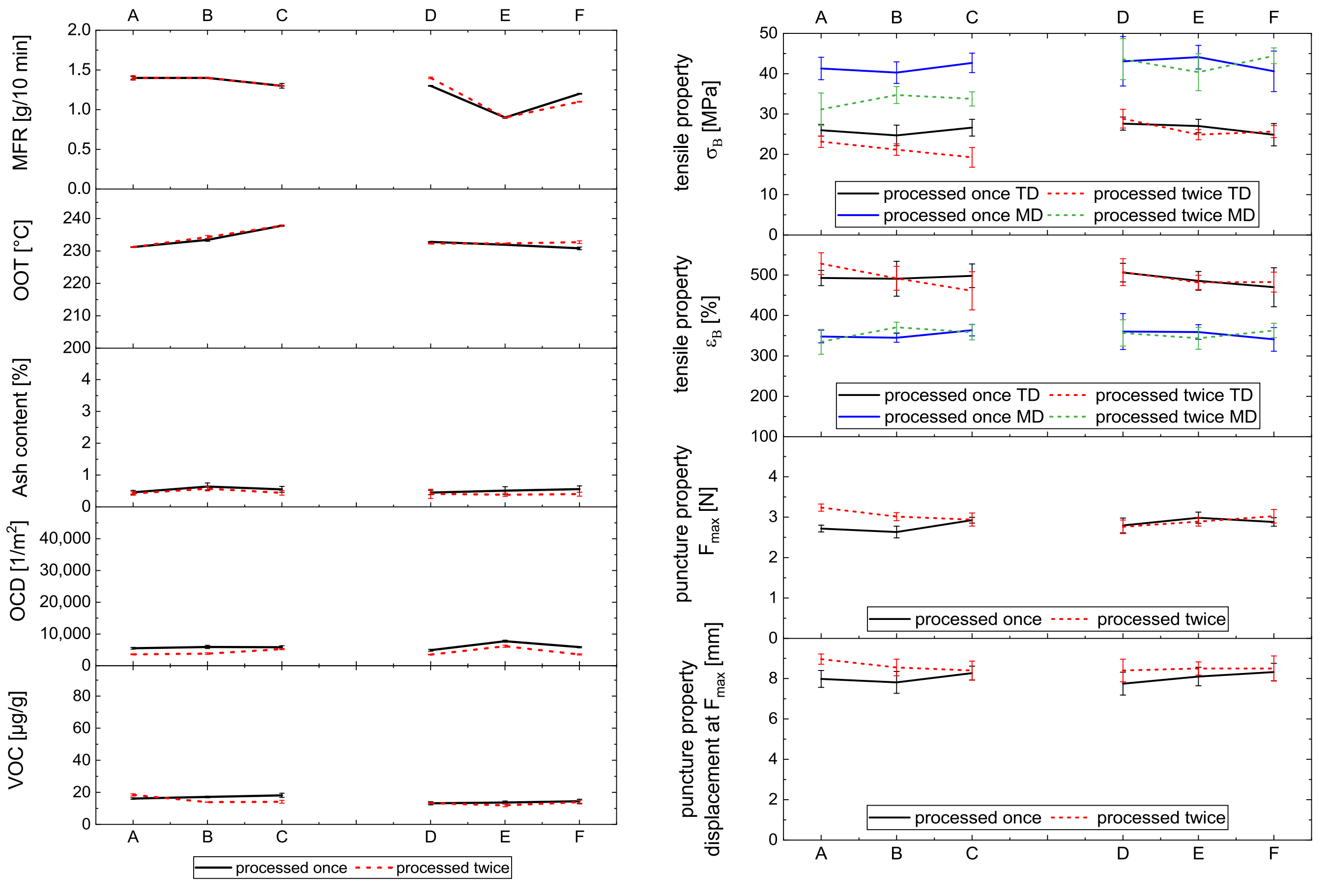

FIGURE 1 | Results originating from MFR, OOT, ash content, OCD, VOC, tensile (σ_B and ε_B), and puncture (F_{max} and displacement at F_{max}) tests performed on the rPE-LLD samples. [Color figure can be viewed at wileyonlinelibrary.com]

filtration process (red curve) compared to the first filtration process (black curve), although the influence of the washing parameters does not appear to be physically logical. These differences in ash content are more likely attributable to the inherent heterogeneity of the input material. Similarly, samples D, E, and F exhibit lower ash content values after the second filtration process, with the washing parameters again not exhibiting a clear logical influence. Notably, the overall ash content values are relatively low since the waste stream was rather clean already at the beginning, below 1%, but with significant standard deviations, indicating a certain degree of variability in the samples.

Samples A, B, and C of the OCD measurement show rather constant values of around 5500 $1/m^2$ for the samples processed and melt filtered first (black curve). As the temperature increases (samples D, E, F), the amount of impurities tends to increase. These results may be due to sample variability, similar to the ash content results, or, in this case, due to degradation processes (formation of gel points) from the whole recycling process, which is in correlation with the MFR values. Here a minimum value is obtained for E, whereas the OCD depicts a maximum value for this sample. All samples generally show lower contaminant levels after the second filtration process (red curves).

The VOC analysis reveals that at low temperatures (samples A, B, C), after the first melt filtration process (black curve), the samples exhibit a steady increase with increasing NaOH content and washing time (from16.2 to18.2 μg/g). When the washing temperature is higher (samples D, E, F), the same tendency as for the lower temperatures is observed, but the VOC values are lower (from 13.2 to 14.5 μg/g). After the second melt filtration process (red curve), the VOC values decrease when NaOH is added (from A (18.5 μg/g) to B (14.0 μg/g)) and remain constant when the washing time is increased (from B to C). At higher temperatures, the second filtration process has little effect except for sample E (12.0 μg/g).

Since the films were already produced during the OCD measurements, they were also used for the tensile measurements. Although the films were blends of the produced recyclate with virgin material, the tendencies of the different processing parameters are discernible. The film samples were tested in transverse direction (TD) and machine direction (MD).

The stress-at-break σ_B for the once-processed and melt-filtered samples in TD (black curves) shows rather constant values when washed at lower and higher temperatures. Nevertheless, σ_B is slightly higher when washed at higher temperatures. After processing and melt filtering twice (red curve), the TD samples washed at lower temperatures (samples A, B, C) show lower σ_B values and a decreasing trend with the addition of NaOH and longer washing times. At higher temperatures, samples D, E, and F show similar values for the first and the second melt filtration processes. All of the MD samples show similar trends as the TD samples, but at higher values, except the samples processed and melt filtered twice (green curve) washed at lower temperatures (samples A, B, C). Here, a small increase in σ_B is observed when NaOH is added, and the values remain constant even when washing is prolonged (sample C).

The strain-at-break ε_B values in TD for the samples processed and melt filtered once (black curve) remain relatively constant for samples A, B, and C, but show a decreasing trend for samples D, E, and F, which are washed at higher temperatures. However, when the samples are filtered and processed twice (red curve), ε_B shows a decreasing trend over all samples, but at the same strain levels. The MD samples display ε_B values that are generally lower than their TD counterparts but remain more consistent across samples A through F. There is no clear, discernible difference between the first melt filtration process (blue curve) and the second melt filtration process (green curve), indicating that the additional processing and filtration have little effect on ε_B in MD. Only small variations can be observed between the once- and twice-processed and melt-filtered MD samples.

Figure 1 shows the maximum force F_{max} that the film samples, produced by the OCD extruder, were able to withstand during a puncture test. Samples A, B, and C processed and melt filtered once (black curve) show an increase in F_{max}, while samples D, E, and F show a slight initial increase from D to E, and sample F shows a further decrease. The cold-washed samples processed and filtered once tend to show slightly lower F_{max}, values compared to their hot-washed counterparts. The cold-washed samples processed and filtered twice initially show higher values compared to the results of the samples processed and filtered once, but decrease with the addition of NaOH and further decrease with longer washing times. The hot-washed samples are in the same range as the samples processed and filtered once, but show a steeper increase with the addition of NaOH and longer washing times.

Looking at the displacement at F_{max}, the samples melt filtered and processed once (black curve), samples A, B, and C, as well as D, E, and F, show an increase in displacement when NaOH is added to the washing process and a further increase happens when the washing time is increased. The samples processed and melt filtered twice (red curve) A, B, and C show a decrease when the same washing parameters are changed, while the values for samples D, E, and F remain constant.

3.2 | rPE-LD

The MFR values shown in Figure 2 show clear trends for the rPE-LD stream. Processing and melt filtering once (black curve) after washing at lower temperatures (samples A, B, C), a decreasing trend from 1.2 to 1.1 g/10 min in MFR is observed with the addition of NaOH. More stable MFR values are measured when the samples are additionally washed for a longer time. At higher temperatures (samples D, E, F), the trend is reversed, with no difference in MFR when NaOH is added (1.1 g/10 min), but a steep increase to 1.3 g/10 min when washing is prolonged. Looking at processing and melt filtration twice after washing (red curve), the MFR is initially higher at 1.5 g/10 min for sample A, but a similar decreasing trend is visible in the figure for samples B (1 g/10 min) and C (1.1 g/10 min). For samples D, E, and F, processing and melt filtration twice shows a different trend compared to the samples processed and filtered once; further, an increase from sample D (1.1 g/10 min) to E (1.3 g/10 min with the addition of NaOH) and a decrease to the lowest value for sample F (1.0 g/10 min) is measured.

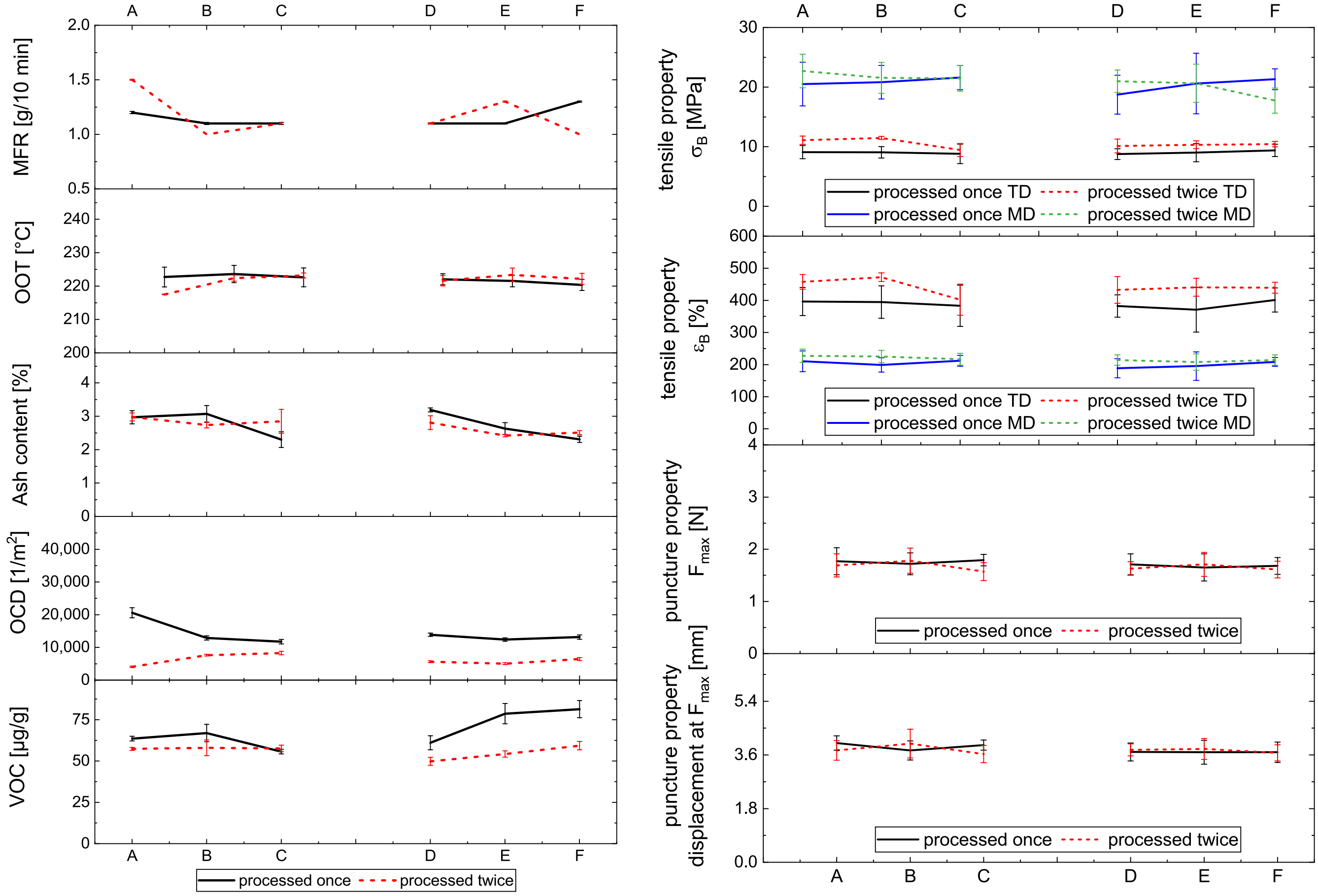

FIGURE 2 | Results originating from MFR, OOT, ash content, OCD, VOC, tensile (σ_B and ε_B), and puncture (F_{max} and displacement at F_{max}) tests performed on the rPE-LD samples. [Color figure can be viewed at wileyonlinelibrary.com]

For all samples processed and melt filtered once (black curve), the OOT values are quite stable. For processing and melt filtering twice (red curve), sample A shows the lowest value at 217.5°C, but a slight increase can be seen when NaOH is added to the washing process (sample B). When washing at higher temperatures, the OOT is slightly higher for the twice-processed compared to the once-processed samples.

The ash content for the samples processed and filtered once (black curve) is rather stable at around 3% for samples A and B, but shows a decrease to around 2% with NaOH and longer washing times (sample C). A further decreasing trend from sample D to E and E to F, from 3% to 2%, is also visible in Figure 2. The samples processed and melt filtered twice (red curve) show more stable values around 3% when washed at lower temperatures and a very slight decrease when washed at higher temperatures.

A clear decrease in OCD for samples A to C processed and filtered once (black curve) is noticeable; on the other hand, rather stable values are measured when washing at higher temperatures (samples D to F). The highest value of 20,600 1/m^2 was measured for sample A, and the lowest value of 4080 1/m^2 for sample A processed and melt filtered twice (red curve). The same trend for twice processed and filtered samples D to F can be found as for the once processed and melt filtered samples. In general, the twice-melt-filtered samples exhibit significantly lower contamination amounts compared to the once-melt-filtered ones.

For processing and melt filtering once (black curve), after cold washing, addition of NaOH, and washing at longer washing times (samples A, B, C), the samples show a decrease in VOCs. On the other hand, washing at higher temperatures shows a steady increase in VOCs. After processing and melt filtering twice (red curve), the VOC values are lower than after processing and melt filtering once.

For the stress-at-break values, the MD values are generally higher than the TD values, as expected. The TD values are generally rather constant, but the samples processed and filtered twice (red curve) show higher values than the ones processed and filtered once (black curve). Samples A, B, and C processed and melt filtered once from the MD experiments (blue curve) show rather constant values. The hot-washed samples show an increase when NaOH is added to the washing process and a further increase when washed for a longer time. For the samples in MD processed and filtered a second time (green curve), the results show a decrease at both washing temperatures with the addition of NaOH or with longer washing times.

The TD strain-at-break values tend to be higher than the MD values, which was expected. When processed once and measured in TD (black curve), the values of all six samples are quite stable. The samples processed and filtered twice, measured in TD (red curve), also show rather stable values, except for sample C, which is lower. In general, the values for the twice-processed samples are higher than those for the once-processed samples. In MD, the values for the once-and twice-processed samples are very constant and almost identical, except for slightly higher values for the twice-processed and melt-filtered samples.

When performing puncture tests, all samples show rather constant values of F_{max} (around 1.9 N) and the displacement at F_{max} (around 3.7 mm).

3.3 | rPE Mix

Figure 3 shows the results for the rPE mix. The highest MFR value is observed after a five-minute wash without detergent at 1.1 g/10 min (sample A). Introducing NaOH during the washing process results in a decrease in MFR to 1.0 g/10 min (sample B), with a further steady value observed when the washing time is extended to 15 min (sample C). At higher temperatures (80°C), MFR values for samples D, E, and F show no significant difference under varying conditions of around 1 g/10 min. After the second processing and melt filtration, the MFR values remain relatively constant around 1.0 g/10 min regardless of the washing method used.

The black curves, representing the first melt filtration process, indicate that a five-minute cold wash without detergent yields an OOT of approximately 222.8°C (sample A). Notably, the addition of sodium hydroxide (NaOH) in the washing process (sample B) results in a similar OOT, suggesting that the chemical additive does not significantly alter the material's structure at this stage. An increase in OOT is observed with an extended washing time (sample C) to around 226.5°C. Introducing a second processing and melt filtration step (red curve) slightly increases the OOT values, but the overall trends remain consistent with the single melt filtration process. For the samples washed at higher temperatures, similar OOT values as for low-temperature samples were obtained, independently of the number of melt filtrations used. However, the addition of NaOH and an increasing washing time led to a slight decrease in OOT.

The once processed and melt filtered samples, depicted by the black curve, indicate that the lowest washing temperature without detergent yields the highest ash content value of 3.6% (sample A). However, adding NaOH reduces the ash content to 3.4% (sample B), with similar results observed with extended washing (sample C). When analyzing the samples washed at higher temperatures (D, E, and F), a similar pattern is observed, but with a steeper decline. By examining the twice processed and melt filtration results (red curves), notable differences are evident. Samples A and C exhibit significantly lower ash contents of 3.1% and 3.0% compared to their once processed and melt filtered counterparts, whereas

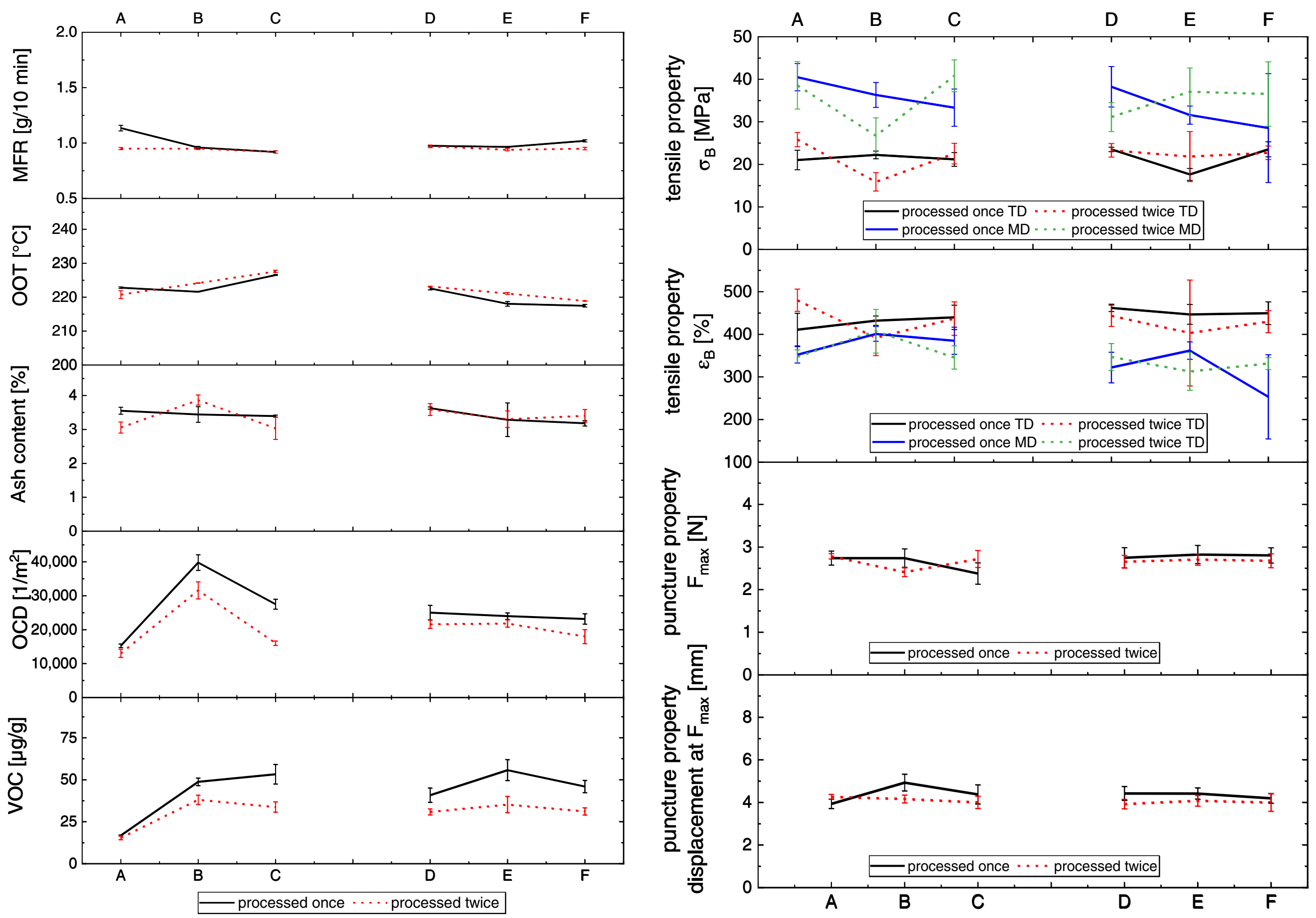

FIGURE 3 | Results originating from MFR, OOT, Ash content, OCD, VOC, tensile (σ_B and ε_B), and puncture (F_{max} and displacement at F_{max}) tests performed on the rPE mix samples. [Color figure can be viewed at wileyonlinelibrary.com]

sample B stands out with the highest recorded value of 3.9%. The samples D, E, and F processed and filtered twice follow a similar trend to the samples processed and filtered once, but with slightly higher values, particularly for sample F.

The first melt filtration process, depicted by the black curves, shows that sample A has the lowest number of impurities per m^2 of 15,270 $1/m^2$. The addition of NaOH in washing results in a sharp increase in impurities to around 39,800 $1/m^2$ (sample B), which then decreases with a longer washing time to 27,500 $1/m^2$ (sample C). This trend is mirrored in the second processing and filtration for samples A, B, and C (red curve). For samples D, E, and F, which were washed at higher temperatures, impurity levels remain almost constant across both filtration steps. The second melt filtration step for all six samples removes more contaminants, as indicated by the red curve being below the black curve.

For the VOCs values of the rPE mix, first and second processing and melt filtration show the same trends. Sample A shows the lowest value for both filtration steps (around 16.0 μg/g), the addition of NaOH increases the VOCs (sample B), and the additional prolongation of the washing time increases the VOC content again (sample C). At higher temperatures, sample D already shows a higher VOC value than sample A, with 40.8 μg/g. The addition of NaOH also increases the VOCs, but extending the washing time for these temperatures reduces the VOCs. Although both processing steps show the same trends, the twice melt filtered samples (red curve) show lower values than the once melt filtered samples (black curve).

For samples A, B, and C, which were washed at lower temperatures, the stress-at-break (σ_B) values remain fairly constant at around 21.5 MPa in the TD. However, increasing the temperature results in a significant decrease in stress-at-break for sample E to around 17.6 MPa, where NaOH was added to the washing process. This decrease is also observed in samples B and E after processing and filtering twice (red curve). In the MD, with processing and filtering once (blue curve), there is a decreasing trend in stress from samples A to C, similar to samples D to F. When the material is washed at 25°C and melt filtered twice in MD (green curve), the stress-at-break decreases again for sample B, where NaOH was used in the washing process. At higher temperatures, the addition of NaOH increases the stress-at-break, while extended washing times show no change.

The samples processed and melt filtered once in TD strain-at-break (ε_B) measurements (black curve) display a clear increasing trend for samples A, B, and C tested at 25°C, indicating that the first melt filtration process results in progressively higher TD strain values. Conversely, the TD strain values for samples D, E, and F tested at 80°C remain almost constant, showing little variation during the first melt filtration process at this elevated temperature. In contrast, the second melt filtration TD measurements (red curve) exhibit a decreasing trend, indicating that the additional filtration step reduces the TD strain values compared to those once melt filtered. The MD measurements processed and filtered once (blue curve) for samples A, B, and C also show an increasing trend, similar to the TD values for these samples. However, the MD samples processed and filtered twice (green curve) do not exhibit a clear influence from the washing process, maintaining relatively constant values within the standard deviations.

The force values shown in Figure 3 are stable at both low and high temperatures, with samples processed and filtered once (black curves) achieving higher maximum force levels of around 2.7 N compared to the samples processed and filtered twice (red curves) of around 2.6 N. This trend persists throughout the measurements, clearly distinguishing the force profiles between the once and twice melt-filtered methods. Similar trends are observed in the displacement data, where once melt-filtered samples exhibit greater displacement values than those subjected to melt filtration twice.

4 | Discussion

The MFR measurement is a simple tool for industrial quality assurance processes. Further analytical methods typically need specifically trained employees, which are most likely not affordable in industry. Even though the MFR measurement only indicates the zero-viscosity of a material, it does provide insights into the melt flow behavior. When comparing the MFR values of all three waste streams, it is noticeable that the rPE-LLD shows slightly higher values (around 1.4 g/10 min), especially for the samples A, B, and C washed at 25°C. The rPE-LD samples washed at 25°C show lower values around 1.1 g/10 min, and the rPE-mix samples washed at 25°C show even lower values around 1.0 g/10 min. For the samples washed at 80°C, the rPE-LLD and rPE-LD samples show very close values around 1.1 g/10 min. The rPE mix again shows the lowest values around 1.0 g/10 min. The most significant factor influencing the MFR of recycled polymers is the input, as different processes necessitate differing flow capabilities (e.g., cast film vs. blown film). The rPE-LLD is a relatively homogeneous stream, as the input was separately collected and could only be contaminated by the product packaged, the labels on the film, and their adhesives. One disadvantage of the rPE-LLD input stream is that the stretch films are typically stuck to one another due to their adhesive nature. This traps contaminants on the surface of the film, making them difficult to remove through washing. The rPE-LD was also collected separately, but this stream was significantly contaminated by the products it contained (e.g., dirt, concrete, etc.). Moreover, the stream is highly heterogeneous with regard to PE-LD types, as each producer utilizes a distinct material. The rPE mix is a markedly heterogeneous waste stream, given that it is not collected separately but rather in the yellow bag. Sorting facilities are highly efficient, yet the collected waste stream will inevitably be imperfect, resulting in contamination of the PE waste with other polymers and residuals of the packaged products.

Furthermore, concerning the melt filtration of the three waste streams, it is noticeable for all of them that the samples processed and melt filtered once (black curve) washed at lower temperatures (A, B, C) show a decreasing trend, while the samples washed at higher temperatures (D, E, F) show an increasing trend, except for rPE-LLD sample D. The samples processed and melt filtered twice of the three waste streams do not show any similarity. The decreasing and increasing trends can be explained by the degradation processes taking place inside the polymeric materials or the heterogeneity of the wastes. The typical degradation mechanisms for PE are chain branching and chain scission, both of which can occur in the material [33–35]. Literature shows that the influence of NaOH leads to degradation by chain scission [12]. A rather small influence on the MFR can also originate from contaminants inhibiting the flowability [36].

When comparing the OOT values of all three waste streams, it is noticeable that the rPE-LLD has values above 230°C for all washed and filtered samples. For the rPE-LD, the OOT is between 220°C and 230°C, and for the rPE-mix, some samples even reach values below 220°C. The variations may be due to the different input streams, as for the MFR, since each product discarded after its use contains different residual stabilizers [37]. Another reason may be the impurities. Organic material contains oxygen, which can cause premature oxidation when heated in an extrusion process. The more organic material that is present in the recyclate, the earlier the oxidation process can occur, thus lowering the oxidation temperature [21, 38, 39].

Furthermore, for all three waste streams, the OOT values of samples A, B, and C for the once and twice melt filtered trials predominantly show an increase in OOT. When washed at higher temperatures (samples D, E, and F), a decrease predominates for both first and second processing and melt filtration. This is in accordance with the literature, where it was found that a decrease in stability can be measured when polyolefin waste is washed in NaOH [12, 40].

Even though the ash content measurement is not the most accurate method to analyze the inorganic content, compared to other methods, it analyzes a rather large amount of sample, which yields more representative results for inhomogeneous material streams. In this study, the rPE-LLD films exhibited an increasing trend, albeit with generally very low values (between 0.4% and 0.7%). In contrast, rPE-LD demonstrated a decreasing trend with increasing washing parameters (from 3% to 2%), while rPE mix exhibited relatively constant ash content values after washing (more than 3%). These findings suggest that the rPE-LLD waste stream is relatively uncontaminated, which was anticipated based on the elevated OOT measurements. Moreover, the rPE-LLD waste stream was utilized for packaging food products. These contaminations were effectively eliminated in the muffle furnace, leaving no residual matter. This measurement method further affirms that the rPE-LD waste stream had a comparatively higher degree of contamination, which may have originated from the packaged products, such as dirt or sand, thus resulting in elevated ash content values. The rPE mix once again demonstrated the poorest results in comparison to the other two waste streams, which is also consistent with the OOT values measured. Due to the small number of samples used for the ash content measurement, even small variations can have a big impact. Additionally, residual inorganic salts from the washing process (e.g., NaOH or hardwater minerals) can further elevate the measured ash content [41]. In general, according to literature, the ash content of unwashed material is higher than that of washed material [40].

The use of OCD enables a quick measurement of representative sample sizes. However, due to the optical limitations inherent to the technique, not all inorganic contaminants can be detected. The OCD results of the three waste streams further back up the results of the OOT and ash content measurement, that the rPE-LLD is the cleanest, exhibiting values below 10,000 contaminants per m^2, rPE-LD is more contaminated, exhibiting contaminant amounts between 6000 and 20,000 contaminants per m^2 and rPE mix is the most contaminated of the three streams, exhibiting contaminant amounts of approximately 10,000 up to 40,000 contaminants per m^2. Furthermore, all samples processed and melt filtered twice of the three waste streams exhibited lower values compared to the samples processed and melt filtered once, which is also partly consistent with the ash content measurements. The rPE-LLD samples show the lowest levels of contaminants, while the rPE mix samples demonstrate the highest levels. It is important to note that processing and filtering twice may result in a reduction in contaminant size but an increase in number. Consequently, future studies should incorporate a size distribution evaluation to accurately assess the contaminants since it is possible that the number of particles may be higher due to their crumbling under heat and pressure [21].

The VOC measurements yield comparable outcomes to those observed in the OCD values. Furthermore, rPE-LLD exhibits the lowest VOC amounts of below 20 μg/g and therefore seems to be the cleanest material, in accordance with the measurement mentioned above. Opposed to the results of the measurement methods before, rPE-LD exhibits more VOC of approximately 50 to 80 μg/g than rPE mix, approximately 20 to 60 μg/g. In general, all VOC measurements demonstrate an increasing trend with NaOH addition and longer washing times. This increase is likely due to the formation of short-chain degradation products resulting from alkaline-induced degradation of the polymer matrix, as well as the breakdown of inks, adhesives, and other additives present in the waste stream [20].

The differences in MD and TD can be explained by the processing of the film. In MD, the chains of the polymer are oriented and prestretched; since, for this study, no clamping system or other devices were used to hold the film in shape, the polymer was able to contract more freely in the transverse direction, so the chains were not preoriented [42]. These different states of orientation lead to different failure mechanisms (strain hardening for MD and cold drawing for TD) [43].

The rPE-LLD samples exhibited the highest stress and strain values between 15 and 50 MPa and 300 to 550%. Furthermore, PE-LLD exhibits elevated stress and strain values relative to those observed in virgin materials, which are superior to those observed in PE-LD. Since this waste stream is collected separately, there is very little incorrect material in this stream, resulting in a fairly stable recyclate. However, this waste stream is often contaminated with labels and adhesives, affecting the contamination levels and sometimes leading to defects that reduce the mechanical performance [44]. With regard to the mechanical properties, the rPE-LD displays the lowest stress and strain values between 10 and 25 MPa and 200 to 500%. The rPE mix exhibits stress and strain values that are similar to those of the rPE-LLD. However, rPE-LD and rPE mix often display heterogeneity, which significantly impacts the tensile strength of the PE. The presence of contaminants often results in immiscibility within the recyclate, which can lead to defects in the final product. These defects can result in the product failing earlier than it would have done if it had been made from virgin material [45–48].

As observed in the tensile properties, the rPE-LLD and rPE mix demonstrate comparable puncture force values of approximately 2.0 to 3.5 N, with the rPE-LD exhibiting the lowest value of 1.0 to 2.0 N. This may be attributed to the heterogeneity of the waste stream. With regard to displacements, rPE-LLD attains higher values of 7.5 to 9.0 mm than rPE-LD and rPE mix, between 3.0 and 5.0 mm. This discrepancy may be attributable to the elevated PE-LLD content in the recyclate.

5 | Conclusion

This study investigated the pretreatment and melt filtration of mechanical recycling using three different polyethylene waste streams. For this purpose, washing parameters such as temperature, sodium hydroxide (NaOH) concentration, and time were varied. A laboratory scale co-rotating twin screw extruder with two different filter sizes was used to further investigate the effect of melt filtration. In this study, material characterization was performed using melt mass-flow rate (MFR), oxidation onset temperature (OOT), ash content, optical contamination detection (OCD), volatile organic compounds (VOC), tensile, and puncture tests. According to our results, all three investigated washing parameters (temperature, sodium hydroxide (NaOH), and time) seem to influence the MFR. On the other hand, processing and melt filtration once or twice do not seem to have a large impact on the MFR. Distinct influences of melt filtration once or twice can be detected by OCD and VOC measurements. The difference between processing and melt filtration once or twice cannot be detected by the maximum force measured by the puncture test. All other measurement methods show some differences in one or two waste streams. However, the largest effect is presumably originating from the heterogeneity of the selected waste streams.

Based on the study's results and a review of the literature, it can be recommended that the pretreatment steps be tailored to the specific input stream. Despite the potential for degradation when washing with hot water and NaOH, the optical properties can be enhanced, and the presence of fatty residues may be decreased. In instances where the desired optical properties are less demanding, washing with cold water may be an adequate alternative. The objective is to achieve the highest quality possible, which necessitates the removal of as much contamination as possible. Therefore, for the rPE-LLD stream, it is recommended to perform washing with cold temperatures, without adding NaOH, since the results do not indicate a significant improvement when the detergent is added. For the rPE-LD and rPE mix, a positive influence on the washing efficiency is noticeable when the waste stream is treated with cold water and NaOH. Concerning the waste streams chosen for this study, washing with hot water does not show a significant improvement.

How often a material should be melt filtered is again dependent on the input stream and the end application. A second processing and filtration step reduces the amount of large contaminants, thereby enhancing the optical quality of the end product, as shown in this study. Nevertheless, pressure occurring in front of the filters does lead to degradation of the polymeric material as well. Consequently, it is essential to assess whether the optical properties, despite the presence of contaminants, outweigh the adverse effects associated with the degraded material. In light of these considerations, a second melt filtration step is advised to ensure the optimal optical quality of the end product.

Author Contributions

J. Langwieser: conceptualization (equal), data curation (lead), formal analysis (lead), investigation (lead), methodology (equal), validation (lead), visualization (lead), writing – original draft (lead). **S. Czaker:** data curation (supporting), formal analysis (supporting). **J. Fischer:** conceptualization (equal), funding acquisition (lead), project administration (lead), supervision (lead), writing – review and editing (lead).

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Conflicts of Interest

The authors declare no conflicts of interest.

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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