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ORIGINAL ARTICLE

Impacts of washing and deodorization treatment on packaging-sourced post-consumer polypropylene

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Abstract

Emerging legal requirements will likely considerably heighten demand for high-quality recycled raw materials for e.g., packaging and automotive applications; key EU legislation mandates recycling as the future end-of-life option for municipal solid plastic waste. Yet recycled plastic use remains low due to safety concerns, undesirable aesthetic, olfactory, and mechanical properties, mainly attributable to contaminants present in recyclates. Advanced treatment options for recovered polypropylene (PP) packaging and the impact of such treatments on the polymer are currently poorly documented. We investigated the effectiveness of hot/cold washing and hot air devolatilization treatments in removing volatile substances from residential post-consumer PP plastic waste to improve its scope of application and value and to assess possible side effects on mechanical and processing parameters. Cold- and hot-washed recyclates exhibited similar contaminant levels and most substances were removed within 7 h. The recycling procedure had no adverse effects on mechanical or processing parameters although reprocessing caused polymer degradation, indicated by decreasing viscosity, elongation at break, and tensile strength. Washing and hot air devolatilization treatment of plastic wastes improve their scope of application and value by enhancing mechanical properties and considerably reducing the amounts of odorous substances, but is often not suited to high-quality applications, such as packaging. The dominance of packaging waste and strict legislation on food-grade recyclate applications will make widespread recyclate use challenging since it represents the primary use of plastic. Recyclate must consequently be extensively utilized in non-food contact applications until advances in waste sorting, washing, and devolatilization yield less contaminated recyclates with improved properties.

Keywords Volatile organic removal · Polypropylene · Recycling · Circular economy

Introduction

Plastic is now omnipresent and has reached a level that surpasses waste management system capacities [1]. The European Union's end-of-life vision for polyolefin (PO) resins focuses on mechanical recycling. Upcoming legislation mandates that at least 50 wt.% of EU plastic packaging be recycled by 2025. Additionally, the incorporation of a minimum percentage of post-consumer recyclates (PCR) in newly produced plastic packaging is discussed. This vision aims to establish a net zero plastic waste industry by 2050 [2, 3]. Such legislative steps indicate that recycling represents the future of waste management in most of Europe. Mechanical recycling has been shown to be economically attractive for second life plastic products [4]. It also typically saves more energy than incineration would generate [5], conserves crude oil reserves [6], and reduces EU dependence on fossil fuel imports [7].

Despite such a certain role in the future of Europe, in practice, recycled material is currently used in just 8.5% of new products [8]. Reprocessed high-density polyethylene (PE-HD) and polypropylene (PP) only made up 8% and 3%, respectively, of European converter demand in 2018 [9]. Recycled PO also has a narrow scope of application, such

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as pipes and ‘low-risk’ commodity applications, such as containers, e.g., buckets, flower (nursery) pots, crates and pallets [9–12]. The limited scope of application of PO regranulates (compared to their respective virgin resins) is attributable to their poorer mechanical properties, less appealing aesthetic and olfactory properties, which are in turn caused by contamination and to a lesser extent polymer degradation [13].

Odours from food rests, cosmetics and detergents complicate mechanical recycling [14] and malodor is a common reason for the lack of use of recycled plastics in packaging, automotive and indoor applications [15]. These factors pose a considerable challenge when attempting to obtain high-quality PO recyclates and could explain why authority-approved processes (e.g., those associated with PE-HD milk bottles) are not more widely implemented [9]. It should be noted that low use of recycled PO in the packaging, construction and automotive sectors constitutes a big problem as these sectors have the largest market share in plastics by application (31%, 17% and 12%, respectively) [16]. Washing and deodorization of recycled resins improve regranulate aesthetic, olfactory properties, and contamination levels and increase regranulate scope of application and value, making them ever more timely and important research areas that are still very much underexplored [17].

Plastics can contain substances of very high concern, such as flame retardants, stabilizers and plasticizers, which are used to optimize properties in virgin polymers [18]. Brominated flame retardants, for example, can be found in household waste plastics [19]. PCR can also contain fragrances, such as limonene [20], and malodors resulting from thermal degradation, processing, additive degradation, microbial metabolism, or contact with chemicals arising from spoiled foodstuffs [21]. Due to the permeability of PO, these contaminants are challenging to remove [22].

Various impurities can be removed or reduced by common washing methods [15], such as stirred tank reactors [23, 24] or continuous friction-based systems inspired by helical-conveyor centrifuges for dewatering suspensions [25], with water, caustic soda and a selection of washing agents e.g., detergents [26]. When combined to form a complete washing line, these machines usually consume between 2 and 3 m^3 of water per ton of processed plastic material [27]. The resulting contaminated wastewater is usually treated using flocculants to precipitate impurities, which are then filtered off before the water is fed back into the washing apparatus. That said, after several washing cycles, the water becomes too contaminated for further reuse and must be treated and discharged. This step is frequently tasked to municipal wastewater treatment plants [28].

Hot washing has been shown to increase removal of volatile organic compounds [22, 29] but requires more energy than cold washing and is reported to be up to 1.9–3.6 times more expensive although more recent research indicates lower values [30, 31]. Multiple tank hot wash has so far only been realized for polyethylene terephthalate (PET) [15]. Generally accepted parameters for hot washing of polyolefins are also yet to be established. Ethyl acetate has been shown to be the most effective washing medium for odour removal [26], but being a flammable liquid, the evolution of explosive vapor–air mixtures poses a safety risk. Since substances tend to adhere more strongly to polymers with decreasing molecule polarity, i.e., are not washable using only (polar) water, most currently employed washing processes are also unable to remove apolar compounds from recycled material [21]. Current industrial washing practice and ordinary regranulation are largely ineffective in odour removal [32, 33].

Other deodorization, such as degassing of polymers over several hours using air, is an established conditioning process (for smell sensitive applications of virgin materials) and could be relevant to plastic recycling [34]. Hot air stripping is also a cost efficient and common practice for conditioning PE-HD PCR [17], and alternative methods, such as steam stripping and polyethylene glycol (PEG) extraction for odour removal, have been investigated at pilot scale [17]. However, assessments of (hot and cold) washing and hot air devolatilization processes on reducing contaminants and the effect of this conditioning on the mechanical properties and processing of common regranulates are lacking.

In this work, we investigated industrially recycled PP regranulates and flakes from multi-coloured, cold-, and hot-washed input streams obtained from the “source separated” Austrian yellow bag collection system. The effectiveness of hot air devolatilization in improving recyclate scope of application and value through the removal of volatile organic compounds was assessed. Possible side effects of the conditioning on mechanical and processing parameters were also studied. The polymer composition and the inorganic content of the recyclates were initially characterized prior to chemical, mechanical, and rheological analysis based on conditioning time and reprocessing cycles.

Materials and methods

Materials

Cold- and hot-washed PP flakes (from lightweight PP packaging containers with volumes < 10 L, such as cups, trays, bottles, and blister packaging) were made available by Steinbeis PolyVert GmbH (Völkermarkt, Austria). Regranulates, with and without hot air devolatilization treatment, were kindly provided by Engineering, Recycling, Maschinen und Anlagen (EREMA) Ges.m.b.H (Ansfelden, Austria). RB707CF PP (random co-polymer) was kindly provided by Borealis AG (Schwechat, Austria) as a virgin PP reference material. Cold- and hot-washed flakes were visibly distinguishable, making variations in composition or origin batch heterogeneity highly likely.

Washing procedures

Sorted PP flakes (grain size: 1 cm) were cold-washed (20–40 °C) in tap water using a designated sink-float apparatus with a throughput capacity of 2–3 metric tons per hour. A batch (<400 kg) of sorted PP flakes was independently hot-washed (50–85 °C) for 10–15 min in aqueous NaOH solution (1–3 wt.%) using a friction washer. The washed flakes were mechanically dried using a friction washer, centrifuge and thermal air dryer, respectively, reducing the moisture content to ≤ 3 wt.%. Hot washing of post-consumer PET is well documented [35, 36] and similar procedures are usually adopted for polyolefins with only minor variations. Cold- and hot-washed flake samples were collected for further analysis. Figure 1 depicts the main processing steps, sampling points for tests, and analyses performed.

Conditioning of (cold and hot)-washed flakes and regranulates

Washed flakes of common PP fractions were homogenized, warmed, and dried before being re-melted and melt filtered using industrial extrusion machinery. Solid particles were filtered off prior to degassing. Submerged pelletizing was applied to shape the degassed melt. Warm pellets were then transferred by conveyor to a silo-shaped, pilot scale (experimental) devolatilization unit, and purged with hot air (120 °C) for 0, 3, 5, 7, and 22 h. Pellet samples prepared from cold- and hot-washed flakes were collected prior to the devolatilization process and at the noted processing time intervals.

Structural and thermal analysis of the flakes and regranulates

Plastic flakes were sorted by hand using a mIRoGun 4.0 handheld near-infrared (NIR) gun (IoSysand GUT GmbH, Walheim, Germany). Flakes of the same polymer were further classified based on appearance—transparent, white, coloured, white/coloured (“co-extruded”), or in-mould labelled. IR spectra of three separate, compression-moulded disks (⌀ 25 mm) produced from regranulate (three locations per disk) were also recorded using a Bruker TENSOR 27 FT-IR instrument (Billerica, MA, USA) with a silicon carbide (SiC) infrared source (mid-IR) and DLaTGS (deuterated L-alanine-doped triglycine sulfate) detector. Measurements were performed using an attenuated total reflectance (ATR) diamond (DuraSampl IR II) with single reflection. 16 scans were conducted in the mid-IR spectrum between 600 and 4000 cm^{-1} with a resolution of 2 cm^{-1}.

Thermal properties of post-consumer granulate were analyzed prior to processing (extrusion) using a TA Instruments Q2000 DSC (New Castle, DE, USA). A 6 ± 0.5 mg sample was placed in an aluminum testing pan and encapsulated. All measurements were conducted at a heating/cooling rate of 10 $\text{K}\bullet\text{min}^{-1}$. Samples were heated to 220 °C, cooled to 30 °C, and then reheated to 220 °C. A nitrogen atmosphere was maintained at all times using a flow rate of 50.0 $\text{mL}\bullet\text{min}^{-1}$. The melting temperature T_m of the second heating run was analyzed using TA Universal Analysis software (v. 4.5A b. 4.5.0.5). Analysis was based on three replicate specimens for each sample type.

The inorganic content of industrially treated samples was assessed using a TA Instruments Q500 (New Castle, DE, USA) thermogravimetric analyzer (TGA). Regranulate samples of ~ 10 mg were transferred to an alumina crucible and heated from 30 to 800 °C at a heating rate of 10 $\text{K}\bullet\text{min}^{-1}$. Samples were tested in air and nitrogen atmospheres (both 90 $\text{mL}\bullet\text{min}^{-1}$).

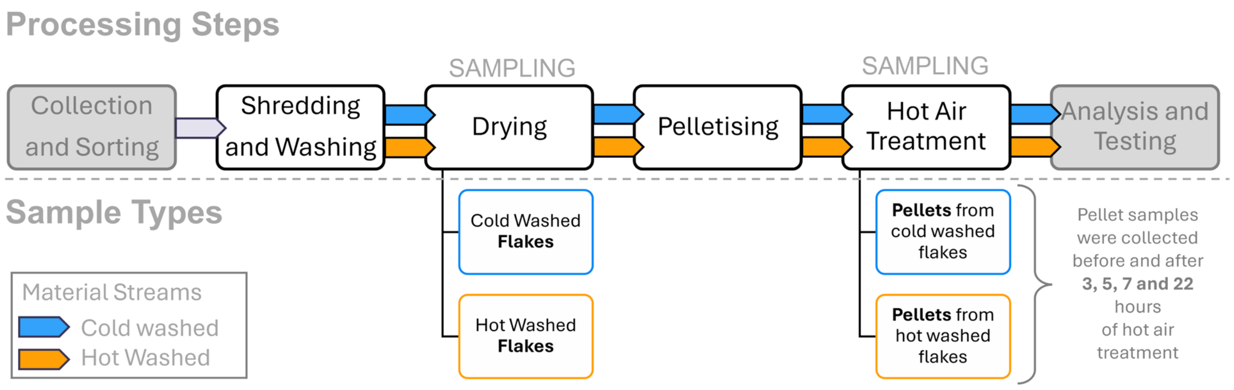


Fig. 1 Generalized flow chart of material processing steps and sampling points

Chemical analysis of the regranulates

Regranulates were measured using gas chromatography–mass spectrometry (GC–MS). 5 g of sample was immersed in 100 mL of ethanol (95%) and migrated at 60 °C for 10 days. 100 µL of ethanol migrate, 5 ml ultrapure water, and 10 µl internal standard (dodecan-d26) were applied. A pre-conditioned solid-phase micro-extraction (SPME) sampler comprising 2 cm, 50/30 µm divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS) (Stableflex Fibre, Supelco) was used at 80 °C for 20 min to extract the headspace of the samples. Separation and identification were performed using an Agilent 6890 gas chromatograph with mass-selection detector HP 5975 (Agilent, USA), a quartz capillary (HP 5MS) and helium as a carrier gas. Substance identification was performed through comparison with a database (Wiley, National Institute of Standards and Technology) with acceptance criteria of > 80% match probability. Peak areas were calculated and compared to the internal standard for a semi-quantification as decan-d22-equivalent. Peaks below the analytical threshold (based on external calibration of decan-d22) were excluded from the evaluation. Identified substances were assessed using the ToxTree in silico decision tree tool and grouped into Cramer Classes I–III based on structural properties.

Extrusion of recycled flakes and regranulates

Regranulates were reprocessed multiple times to simulate the recycling cycle. Flakes and industrially prepared regranulates were extruded using a single screw extruder (EX-18–26-1.5, Extron Engineering, Oy, Finland) with a screw diameter of 18 mm, length-to-diameter ratio of 25:1 at a maximum temperature of 240 °C and 70 rpm screw speed. The extruded material was then ground into particles using a cutting mill (Fritsch Pulverisette 19, FRITSCH GmbH, Idar-Oberstein, Germany), and a sieved fraction of ≤ 4 mm collected. The reprocessing and grinding process was repeated over a total of five cycles. Samples were collected after the first, third, and fifth processing step.

Injection and compression moulding of regranulates and flakes and preparation of the mechanical test specimens

Flake and regranulate materials were injection-moulded to prepare tensile strength specimens using a Haake Mini Lab II twin screw extruder coupled with a Haake Mini Jet II injection-moulding unit (Thermo Fisher Scientific, Waltham, MA, USA). Extrusion was completed at 230 °C with a screw speed of 100 rpm. Injection-moulding was completed at a mould temperature of 40 °C and a pressure of 350 bar with injection and post-injection times both of 10 s. At least eight dog-bone tensile test specimens (thickness 2 ± 0.2 mm) were moulded in accordance with ISO 527-2-5A [37]. The described materials were also compression-moulded at 210 °C and cooled to room temperature at a rate of 15 °C•min^{-1} using a Collin P 200 P hydraulic press (COLLIN Lab & Pilot Solutions GmbH, Germany) to form 25 mm diameter disks for dynamic shear rheology measurements. The compression die setup comprised an aluminum frame with corresponding cut-outs, which was covered with Teflon® film and sandwiched between two cover plates.

Tensile and rheological testing of the flake material and regranulates

A universal testing system comprising a Zwick Z050 frame (Zwick Roell, Germany) with 2.5 kN load cell (Xforce P, sensitivity 2 mV•V^{-1}) and extensometer (makroXtens) was used to perform tensile tests on the prepared specimens at a constant velocity of 20 mm•min^{-1}. Tensile strength σ_M and elongation at break ε_B were calculated using the ZwickRoell testXpert II software (v. 3.6) across at least eight replicate tests. The elastic modulus E_t was calculated based on the region 0.05 to 0.25% elongation using regression.

Dynamic shear rheology tests (frequency sweeps) were performed on an MCR 302 rheometer (Anton Paar, Graz, Austria) equipped with a 25 mm diameter parallel-plate system (1 mm gap size) and temperature-controlled heating hood (H-ETD400) purged with nitrogen. Temperature was held constant at 230 °C during which deformation was raised logarithmically from 1 to 2% at angular frequencies ranging from 600 to 0.1 rad•s^{-1}.

Results and discussion

Characterisation of cold- and hot-washed and hot air decontaminated flakes and regranulates

Cold- and hot-washed flakes both exhibited noticeable unpleasant odours, which were stronger for cold-washed samples compared to hot-washed samples. This was expected as hot washing with aqueous caustic soda solution (80 °C, NaOH (aq.)) has been shown to considerably reduce odour in post-consumer PP flakes [38]. Both cold- and hot-washed flakes had a similar polymer composition, comprising ~ 90 wt.% PP and ~ 6 wt.% PE with 3–4 wt.% unidentifiable material (Fig. 2a). This polymer composition is typical of such regranulates, including those investigated in recent Dutch studies [39, 40], which are typically 96% PP with minor fractions of PE and PET. Further improvements in recyclate quality could be obtained through manual post-sorting. [39]. The PP component of our samples comprised primarily white (39–44 wt.%) flakes but also coloured (18 wt.%), co-extruded/in-mould

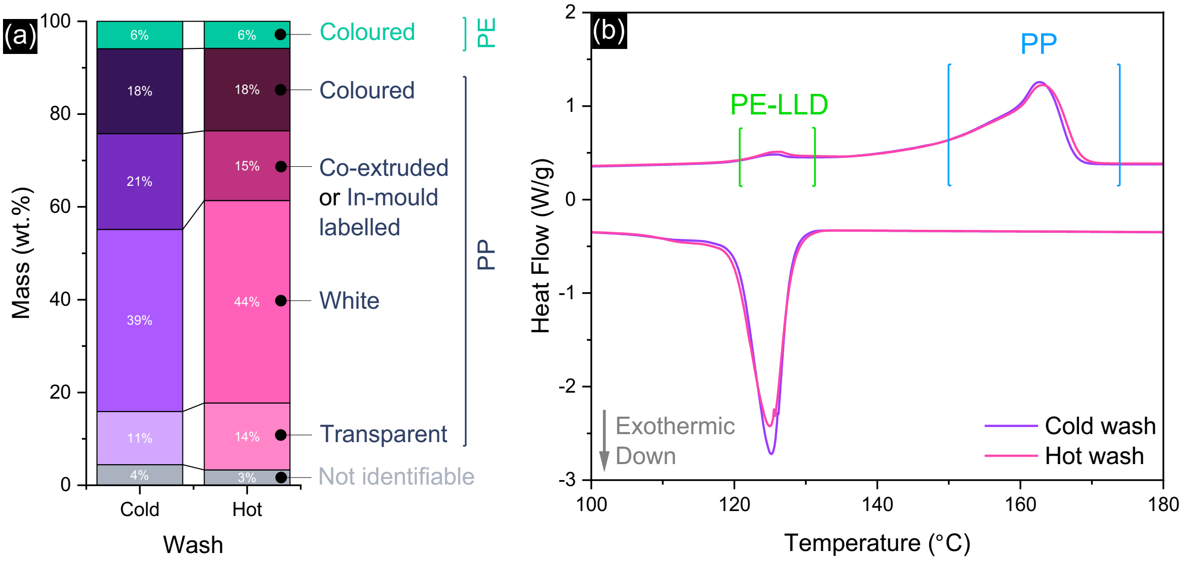


Fig. 2 Polymer composition of cold- and hot-washed recyclates: **a** Polypropylene (PP) and polyethylene (PE) content and colour as characterized by near-infrared (NIR) spectroscopy and appearance; “co-extruded” referring to unprinted flakes with one white and one coloured side and “in-mould” referring to flakes with corresponding decoration, **b** identification of linear low-density PE (PE-LLD) based on differential scanning calorimetry melting peaks (colour figure online)

labelled (15–21 wt.%) and transparent (11–14 wt.%) flakes. The presence of such printed labels (in-mould/ coextrusion fraction) was indicative of imperfect sorting and packaging design and consequently yielded material insufficient for high-quality applications. DSC melting peaks for both cold- and hot-washed flakes and regranulates at ~ 162 °C (for PP [41, 42]) and ~ 125 °C (for linear low-density polyethylene, PE-LLD [43, 44]) suggested that all PP contents were homo- or ethylene block co-polymer rather than random ethylene co-polymer [42] mixed with residual labels made of PE-LLD (Fig. 2b).

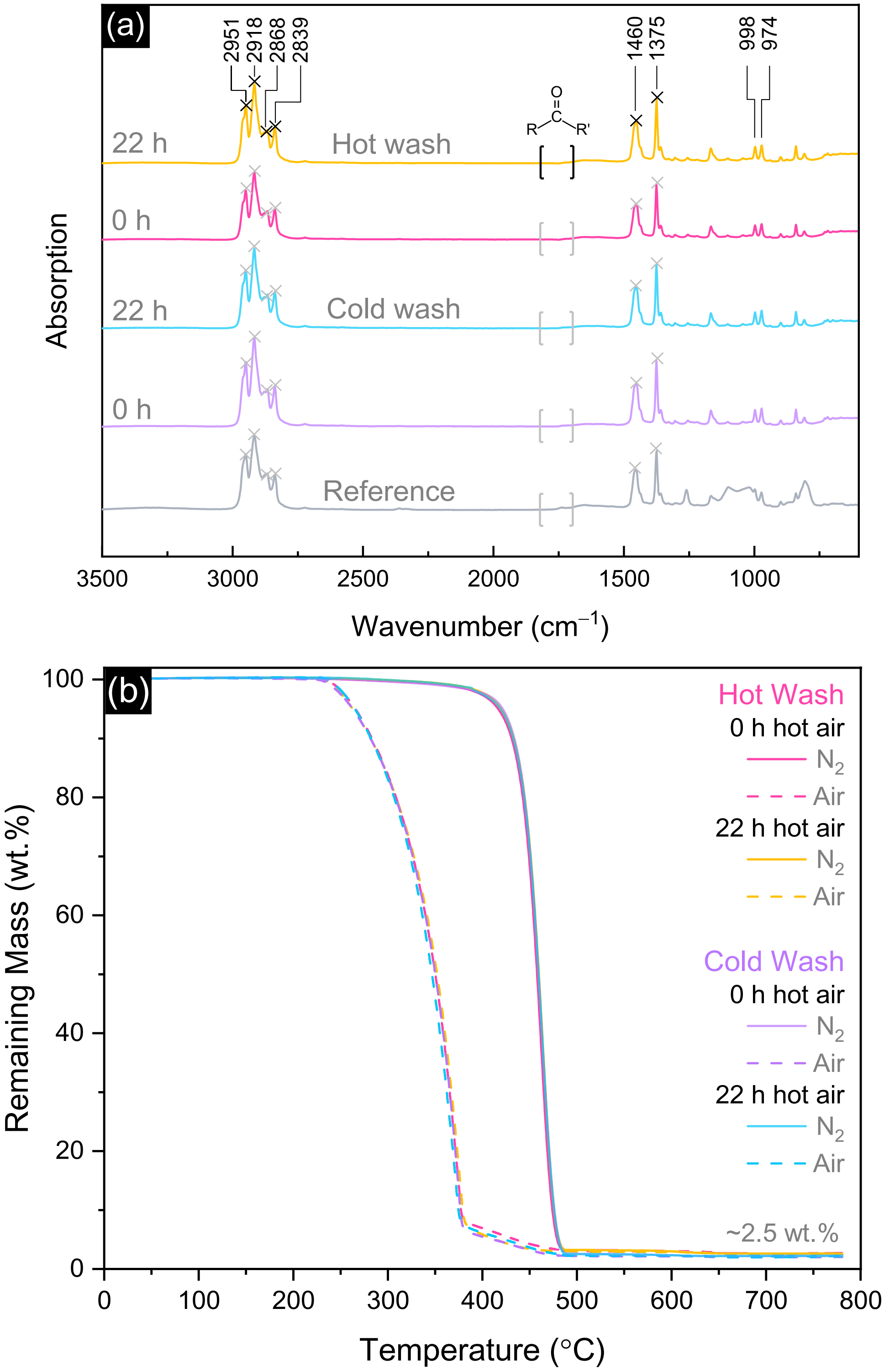
FT-IR bands characteristic of PP appeared at 974 cm^{-1} (CH_3 rocking + C–C chain stretch), 998 cm^{-1} (CH_3 rocking + CH_2 wagging + CH bending), 1375 cm^{-1} (CH_2 bending δ), 1460 cm^{-1} (δ CH_2), 2839 cm^{-1} (CH_2 stretching vibration ν_{st}), 2868 cm^{-1} (ν_{st} CH_3), 2918 cm^{-1} (ν_{st} CH_2) and 2951 cm^{-1} (ν_{st} CH_3) [45–48] (Fig. 3a). No band at 1700–1800 cm^{-1} [49, 50] associated with ν_{st} C = O was observed, which indicated that neither cold- nor hot-washed flakes and regranulates were oxidised [51]. The 3200–3600 cm^{-1} band (ν_{st} O–H) [49, 50, 52] was also absent, which suggested that neither alcohols, carbonic acids, esters, nor hydro-peroxides were present [53]. All samples had a low inorganic content of ~ 2.5 wt.% as identified by TGA in an air atmosphere (Fig. 3b).

GC/MS screening of cold- and hot-washed and hot air decontaminated regranulates

Overall, 85 substances were found in a semi-quantitative GC/MS screening of the migrated granulates, of which 47 could be identified (Table S1). Industrially cold- and hot-washed regranulates not subjected to hot air devolatilization (0 h) exhibited similar concentrations (acknowledging batch heterogeneity common in plastic packaging waste [54]) of most substances, with no discernible improvement associated with hot washing (Figure S1, Table S1). The potential of industrial scale cold- and hot washing to remove recycling contaminants is not well documented; however, the average deodorization efficiency of water at 25 °C is 54%, while washing with water, cetyltrimethylammonium bromide (CTAB) and NaOH is 65–75% [55]. Higher washing temperatures and decreasing particle sizes are also associated with higher volatile organic compound removal efficiency in PE films and bottles [22].

D-Limonene (CAS Nr. 5989-27-5), which was detected in all samples not subjected to hot air devolatilization (0 h), is also a common contaminant in post-consumer bottle PET regranulates [56, 57], but contaminants were otherwise quite unique to the applications of the polymer. This makes sense. Many contaminants relate to the original product’s application. For example, in PET, one would expect application-specific contaminants, such as methyl salicylate, (+)-carvone, 1,8-cineole (mouthwash), and γ-terpinene (soft drink) [58, 59]. However, since these applications are not typically associated with PP, other application-specific contaminants would be expected for PP. Several flavor substances, as well as degradation products of antioxidants, were identified: phenoxybenzene (also known as diphenyl ether), which is used as a flavoring agent [60], 2,6-Di-tert-butyl-P-benzoquinone, which is a degradation product of

Fig. 3 Infrared spectra and inorganic content of cold- and hot-washed recyclates: **a** lack of oxidation in PP observed using Fourier-transform infrared (FT-IR) spectroscopy and **b** inorganic residue remaining following thermogravimetric analysis in nitrogen and air atmosphere



sterically hindered phenolic antioxidants [61, 62], (2-tert-) and (4-tert-butylcyclohexyl) acetate often used in scents [63, 64], and Undecan-2-one, possibly a degradation product of fatty acids (e.g., fish oil) [65] or fragrance ingredients [66], were identified in all samples (Table S1). Evolution of odorants, such as terpenes (e.g., limonene) and hydrocarbons (aliphatic, olefinic and aromatic), more specifically flavor and fragrance components and halogenated compounds is also not uncommon and has been found in post-industrial PP and recycled PE and PP [29, 32]. Although not classified as posing immediate health risks to humans, prolonged exposure to these substances e.g., during manual sorting or as volatiles upon re-extrusion, must be included in any assessment of potential health risks for sorting plant workers [67]. That said, dust, noise, and washing media are often more imminent risk factors [68].

The effect of hot air devolatilization was assessed using cold-washed regranulates. This was due to the prevalence of cold washing in industry—cold washing procedures represent current industrial practice for cleaning during PO recycling since optimized temperature, time, and chemical parameters for hot washing of PO prior to mechanical recycling still require further investigation—and the similarity between substance concentrations present in cold- and hot-washed regranulates. Both initial peak count and signal intensity associated with concentration of volatiles reduced with increasing hot air devolatilization time (Fig. 4). Substances associated with the numbered peaks are listed in Table 1. Diphenyl ether levels were reduced to undetectable levels within 5 h. Most other substances were reduced to undetectable levels within 5–7 h although several required 22 h. Notably, ethyl dodecanoate, ethyl hexadecanoate, ethyl octadecanoate, and 2,2-dimethylhexane could not be fully removed within 22 h of hot air treatment. These substances could either originate from virgin polymers through melt-flow-improving processing aids [69] or product residues like emollients, flavoring [70, 71], fragrances and adjuvants [60, 72–74], or fatty foodstuffs (e.g., olive oil, milk) [75, 76]. The difficulty in removing these substances with hot air devolatilization is likely attributable to them being potentially diffused into the PP prior to washing. These substances also possess boiling points between 190 °C and 270 °C, except 2,2-dimethylhexane (boiling point: 107 °C [77]), which slows evaporation. Additional chromatographic data for the complete dataset (0, 3, and 5 h) is available in Figure S1.

It should be acknowledged that diffusion speed and vapor pressure of contaminants (e.g., n-alkanes) in solvent-free desorption-based (hot air treatment) devolatilization processes are indirectly proportional to molecule size [78–80] and therefore influence devolatilization efficiency. However, optimizing the preceding washing step(s) to remove non-volatile contaminants (e.g., employing non-aqueous basic solutions) has the potential to reduce hot air treatment times for equivalent or increased devolatilization success [26].

Mechanical and processing properties of cold- and hot-washed and hot air decontaminated flakes and regranulates

Elastic modulus E_t, which almost exclusively fell between 1000 and 1300 MPa (Fig. 5a), and tensile strength σ_M, which fell between 25 and 35 MPa (Fig. 5b) did not vary considerably between industrially cold- and hot-washed samples. With the exception of unextruded, cold-washed flake samples, these values were lower than those associated with virgin PP homopolymer: E_t ~1600 MPa [42, 43] and σ_M ~31 MPa [43, 81]. Estimations of likely regranulate composition based on E_t values were made using the equation proposed by Liang and Ma [82], which assumes minor polymer fractions to be shaped as spherical particles. For this estimation, it was further assumed that only PP homo- and co-polymer and PE-LLD were present in the regranulate. E_t values used in the equation were obtained from literature: 1600 MPa for PP homopolymer [42, 43], 1050 MPa for block-co-polymer [42, 83] and 500 MPa for PE-LLD [83]. Estimated E_t results for the samples suggested the vast majority (~ 90 wt.%) of the PP fraction to be block co-polymer. Regranulates and flakes exhibited approximately the same E_t. Multiple extrusions did not considerably affect E_t or σ_M, only leading to minor deterioration in these properties.

Industrially cold-washed regranulate samples exhibited considerably higher elongation at break ε_B than hot-washed samples, with differences of 20–200% in regranulates and 10–450% in flakes (Fig. 5c). Cold- and hot-washed regranulates had higher average ε_B than their flake counterparts in all cases. ε_B was also sensitive to deterioration, likely as chain scission [6], as a result of multiple extrusions [84, 85]. This phenomenon was especially apparent for samples prepared from cold-washed flakes. ε_B drops sharply for PP as molecular weights fall below 200 kg·mol^{-1} [86] (with our samples initially estimated to have an average molecular weight maximum of 200–300 kg·mol^{-1}, based on viscosity comparison with virgin PP at an angular frequency of 0.1 rad·s^{-1} using a power law model fit [87–89] (Figure S2, Table S2). This can be observed as a decrease in complex viscosity $|\eta^*|$ [42], which did reduce as a result of multiple extrusions in both cold- and hot-washed samples (Fig. 6). This indicated that extrusion and not washing affected $|\eta^*|$. Cold-washed regranulates extruded only once exhibited ε_B parity with PP virgin material but rapidly deteriorated after additional extrusions, making it suitable for only low-grade applications. Selected stress–strain curves for each sample type are shown in Figure S3.

It should also be acknowledged that hot-washed flakes did exhibit a lower initial $|\eta^*|$ than cold-washed flakes. Consequently, differences in mechanical properties not necessarily attributable to the washing process could be expected between cold- and hot-washed flakes. These differences are more likely the result of oxidative damage-based inhomogeneity of feed material during previous applications and processing steps [90].

Economic and sustainability aspects of hot air devolatilization treatment

Recent forecasts estimate a tripling in plastic production and a doubling of global plastic recycling rates (to 12%) by 2060 [91]. Recovery of post-consumer plastics for safe

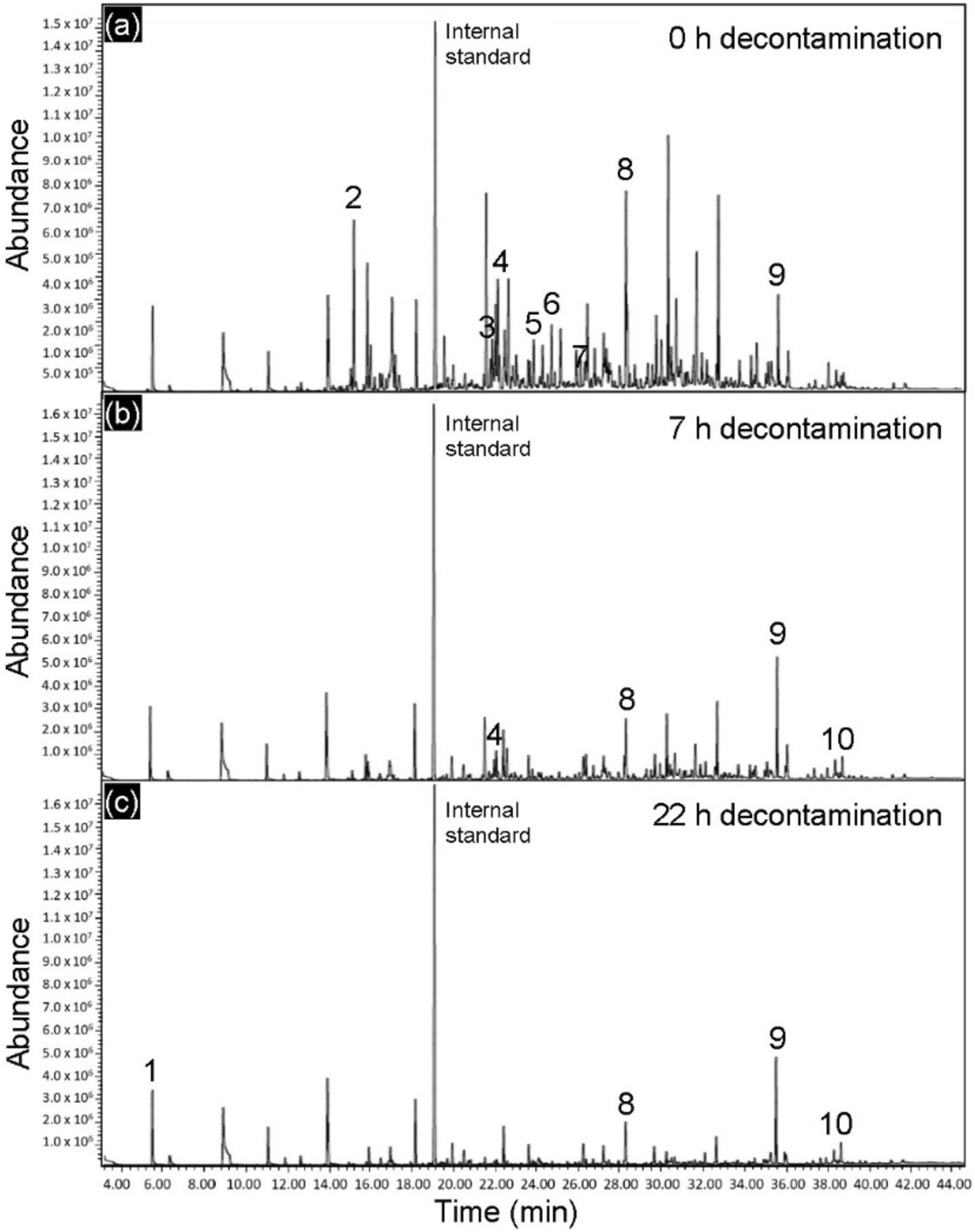


Fig. 4 GC/MS-screening chromatograms of cold-washed samples **a** before (0 h) and after **b** 7 and **c** 22 h of hot air treatment. Peaks are identified in Table 1

Table 1 Peak identification for substances detected in GC/MS-screening chromatograms of cold-washed samples before (0 h) and after 7 and 22 h of hot air treatment

Number	Retention time (min)	CAS-number	Substance
1	5.3	590-73-8	2,2-dimethylhexane
2	15.2	5989-27-5	D-Limonene
3	21.8	112-12-0	Undecan-2-one
4	22.1	88-41-5	(2-tert-butylcyclohexyl) acetate
5	23.8	32,210-23-4	(4-tert-butylcyclohexyl) acetate
6	24.7	101-84-8	Diphenyl ether
7	26.1	719-22-2	2,6-Di-tert-butyl-P-benzoquinone
8	28.3	106-33-2	Ethyl-dodecanoate
9	35.5	628-97-7	Ethyl-hexadecanoate
10	38.7	111-61-5	Ethyl-octadecanoate

and odour-neutral applications will be a critical element of this recycling reform. Washing procedures utilizing aqueous media can remove surface impurities, grease and odours from recovered plastics but produce considerable amounts of wastewater and sludges—1 kg of total suspended solids per m^3 of wastewater is not uncommon [27]. Treatment, disposal, and transport costs associated with sludges amount to 0.47 ± 0.28 € per kg of total solids content [92], but the cost of canal discharge is rarely documented. Devolatilization removes degradation products, volatile substances, and often objectionable malodor from mechanically recycled regranulates through application of heat (e.g., ≤ 120 °C for 7 h) at atmospheric pressure. The energy requirements of the process, (0.13 $kWh\bullet kg^{-1}$ or 0.47 $MJ\bullet kg^{-1}$) [14], which was also employed in this study, constitute approximately 12% of those associated with mechanical recycling (3.83 $MJ\bullet kg^{-1}$ for sorting and regranulation [93]). A relatively low energy consumption is achieved utilizing residual heat from freshly extruded pellets to reduce heating costs coupled with a cooling silo for finished pellets that transfers thermal energy back into the main devolatilization apparatus. This makes it a comparatively quite energy-efficient and likely economical inclusion in mechanical recycling processes that may extend recyclate scope of application [17] to dominant plastic applications with hygiene requirements e.g., packaging and consumer products like household items which constitute 41% of all plastic use [16]. Another promising option would be reuse in automotive parts [94] which is also in line with current European draft bills focused on a recycled plastic use rate of 25% in vehicles by 2030 [95]. That said, mechanically recycled rigid PP—still being largely unable to meet such requirements—can currently service just 3% of the annual demand for rigid PP in the EU (industrial pre-consumer recyclate excluded) [9]. Mechanical recycling costs approximately 0.10 $€\bullet kg^{-1}$ (as of 2021), and therefore can provide resins at 20–50% of the cost associated with virgin material [93]. There is consequently sufficient margin for a small premium to facilitate devolatilization, which will in turn improve recyclate scope of application, avoid market saturation with low quality regranulate and increase adoption rates i.e. promote circularity [96].

Conclusion

Extrusion of post-consumer polypropylene, including melt degassing and melt filtration, represent essential preliminary steps in mechanical recycling to produce high-quality regranulates. Regranulates prepared from cold- and hot-washed flakes exhibited similar concentrations of contaminating substances. However, until a generally accepted, optimized hot washing standard for polyolefins is established, direct comparisons regarding washing efficiencies may lack validity. Hot air devolatilization reduced the concentration of most identified volatile substances to undetectable levels within 7 h. Such a treatment time could prove a sustainable option for recycling companies, based on energy consumption and economic feasibility. Moreover, the devolatilization process effectively removed several volatile substances, resulting in a noticeable reduction in odour intensity. Washing, (subsequent industrial extrusion and melt filtration), and hot air devolatilization themselves did not alter the tensile or rheological properties of the recyclates. However, repeated extrusions did result in polymer degradation (as expected and likely as chain scission), indicated by a drop in complex viscosity, considerable reductions in elongation at break and smaller decreases in tensile strength.

Washing and hot air devolatilization processes can improve the aesthetic, olfactory properties, and safety of regranulates and consequently improve their scope of application and value. This in turn increases the viability of more comprehensive sorting, which improves regranulate properties, and promotes wider utilization in society. However, strict legislation on and requirements for food-grade recyclate applications, coupled with the dominance of packaging as the primary use of plastics, may prove an insurmountable hurdle impeding the widespread adoption of recycled material. Recycled plastic must consequently be widely adopted for all other (non-food contact) applications to offset the lack of use in food applications until collection, sorting, washing, and devolatilization technologies can (hopefully) deliver recyclates with sufficient properties (and safety) for such applications.

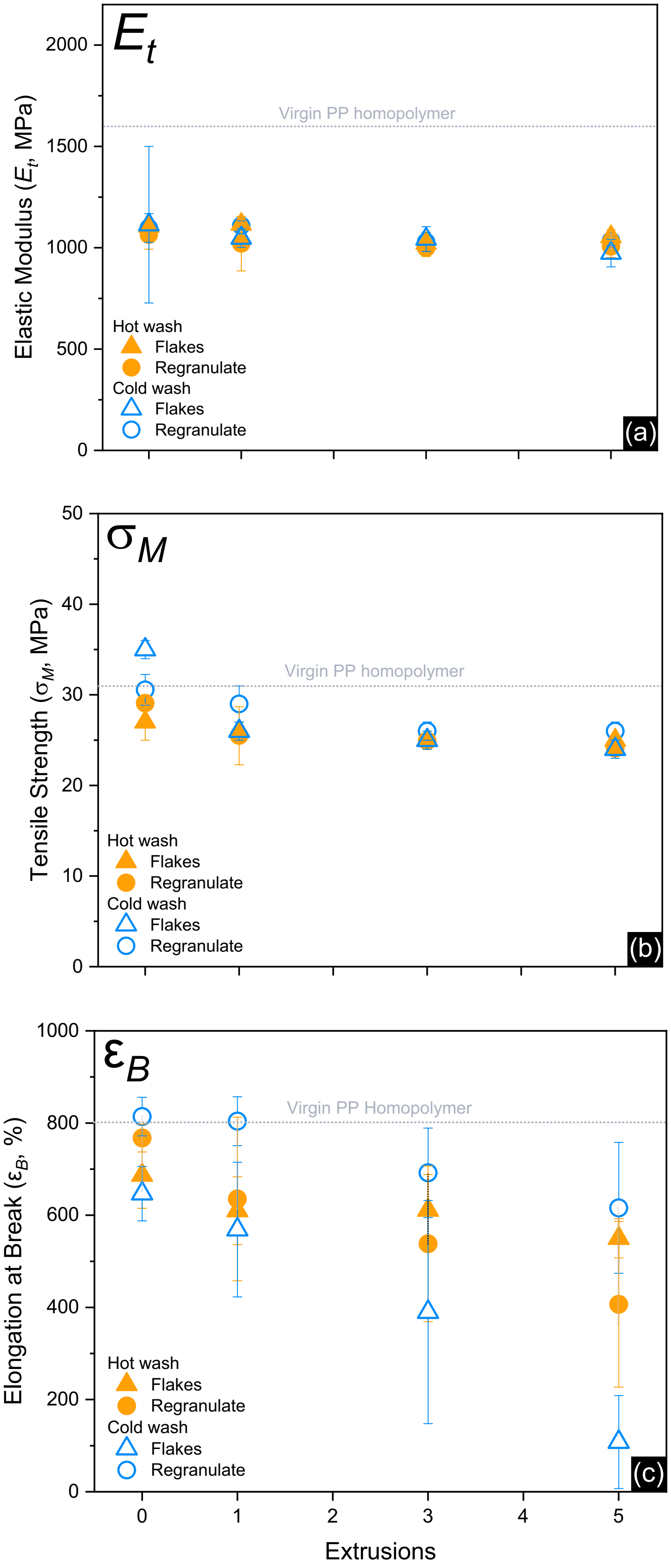


Fig. 5 **a** Elastic modulus E_t, **b** tensile strength σ_M and **c** elongation at break ε_B of cold (blue hollow markers) and hot (orange solid markers) washed flakes (triangles) and regranulates (circles) extruded 0, 1, 3, and 5 × compared to literature values for virgin PP homopolymer (grey broken lines) (colour figure online)

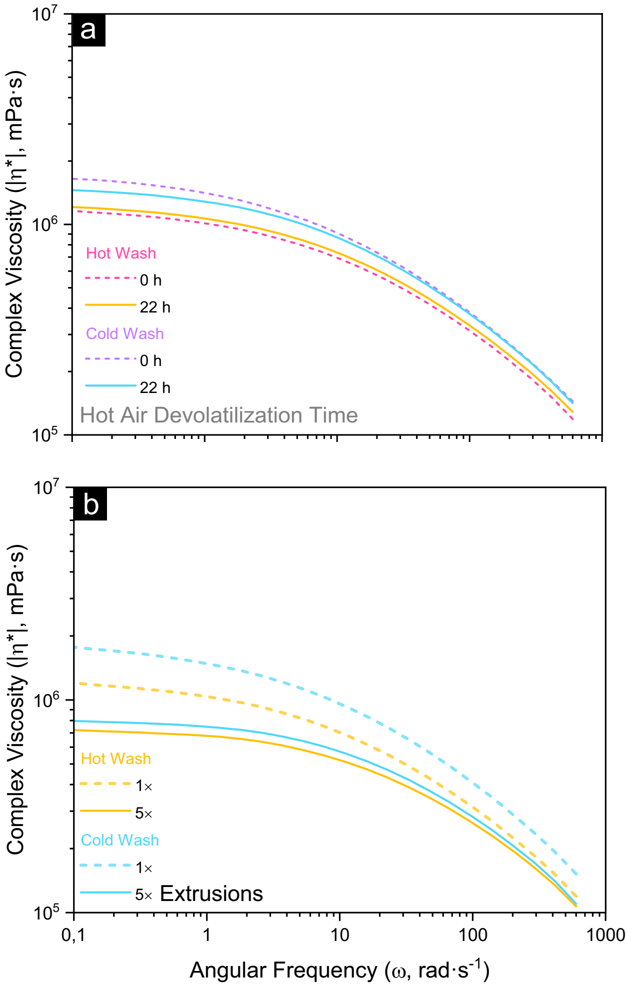


Fig. 6 Frequency sweeps for cold (blue) and hot (red) washed recyclates: **a** effect of hot air devolatilization (0 and 22 h) and **b** repeated extrusion (1 and 5×) (colour figure online)

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