



Deodorization of post-consumer plastic waste fractions: A comparison of different washing media



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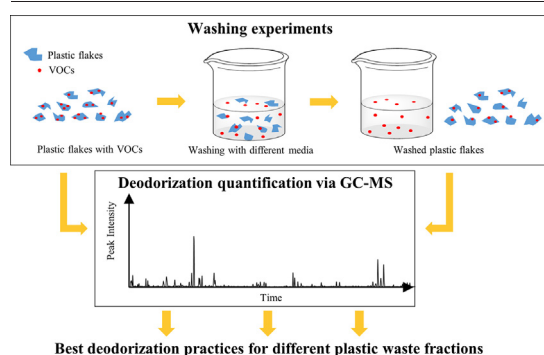
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HIGHLIGHTS

- Odorous compounds impede high-end recycling of post-consumer plastic waste.
- Various washing media are tested for different waste fractions.
- Odor removal is quantified via GC–MS analysis.
- Different washing media result in different deodorization efficiencies.
- Washing PET and polyolefin packaging types is fundamentally different.

GRAPHICAL ABSTRACT



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ABSTRACT

An important impediment to the acceptance of recyclates into a broader market is their unwanted odor after reprocessing. Different types of washing procedures are already in place, but fundamental insights into the deodorization efficiencies of different washing media are still relatively scarce. Therefore, in this study, the deodorization efficiencies of different types of plastics after washing with different media were determined via gas chromatography and mass spectrometry analysis. A total of 169 compounds subdivided into various chemical classes, such as alkanes, terpenes, and oxygenated compounds, were detected across all packaging types. Around 60 compounds were detected on plastic bottles, and around 40 were detected on trays and films. Owing to the differences in physicochemical properties of odor compounds, different deodorization efficiencies were obtained with different washing media. Water and caustic soda were significantly more efficient for poly(ethylene terephthalate) bottles with deodorization efficiencies up to 80%, whereas for polyethylene (PE) and polypropylene bottles, the washing media were relatively inefficient (around 30–40%). Adding a detergent or an organic solvent could increase deodorization efficiencies by up to 70–90% for these packaging types. A similar trend was observed for PE films having deodorization efficiencies in the range of 40–50% when washing with water or caustic soda and around 70–80% when a detergent was added.

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Abbreviations

CMC	Critical micelle concentration
CTAB	Hexadecyltrimethylammonium bromide
CS ₂	Carbon disulfide
EtOAc	Ethyl acetate
GC-MS	Gas chromatography—mass spectrometry
HDPE	High-density polyethylene
HS	Headspace
IS _w	Peak area of the internal standard of a washed sample
IS _u	Peak area of the internal standard of an unwashed sample
log P _{ow}	Octanol-water partition coefficient
NaOH	Sodium hydroxide/Caustic soda
PA _w	Peak area of a certain VOC detected on a washed sample
PA _u	Peak area of a certain VOC detected on an unwashed sample
PDMS	Polydimethylsiloxane
PE	Polyethylene
PET	Poly(ethylene terephthalate)
PP	Polypropylene
PS	Polystyrene
SIM	Selective ion mode
SPME	Solid-phase micro-extraction
VOC	Volatile organic compound
γ	Surface tension
η	Deodorization efficiency

1. Introduction

Plastic packaging is abundantly used today and has proven its utility for many applications. Yet, plastic recycling faces many challenges, and much progress is still needed in terms of packaging design, sorting, pre-treatment, and recycling technologies to achieve closed-loop recycling (Hahladakis and Iacovidou, 2018). Progress is urgent, as the European recycling targets for plastic packaging are ambitious, aiming for recycling rates of 50% by 2025 and 55% by 2030 (European Commission, 2015). One of the main barriers to the high-end recycling of plastic packaging is the typical undesired odor that is present after disposal (Cecon et al., 2021). During their use phase, odorous compounds can migrate into the packaging material. This continues after disposal when microbial activity creates odorous components. As in most European countries, plastics are curbside collected, and cross-contamination and degradation of different polymers occur during waste collection, creating a highly complex mixture of odorous compounds (Saeed et al., 2021, 2018). Next, plastic waste generally enters a material recovery facility where the constituents are sorted into different waste fractions and are typically pressed into bales at the end of the sorting process for transportation purposes (Kleinhans et al., 2021). Subsequently, the bales are sent to a recycling plant where the recycling process takes place. At the plant, the plastic waste is consecutively washed, ground, and compounded and/or pelletized. Pellets are easier to use in converters for effective remanufacture of plastic products (Ragaert et al., 2017).

At such a recycling plant, an objective is to remove odor. However, it is common knowledge that odorous substances remain on the plastic materials after reprocessing. This is one of the reasons why large volumes of plastic waste are currently only suitable for downcycled applications, such as compost bins and street or garden furniture (Abnisa and Alaba, 2021). Hence, more effective deodorization strategies could potentially increase

the market value of recycled plastics. Therefore, the deodorization of post-consumer waste is gaining the interest of industry and research (Horodytska et al., 2020).

Various studies have identified many of the odorous constituents present on plastic packaging materials (e.g., Cabanes et al., 2020a; Demets et al., 2020; Denk et al., 2019; Fuller et al., 2020; Strangl et al., 2017, 2018, 2020), resulting in the identification of more than 400 different volatile organic compounds (VOCs; Cabanes et al., 2020b). Generally, studies have indicated that the smell of plastics is not caused by one specific odorant or chemical substance; it is instead caused by a complex mixture of substances.

Apart from odor characterization, the efficiencies of currently applied industrial washing processes have been investigated in various studies for different types of household plastic packaging waste. Water, detergents, and caustic soda (NaOH) are commonly applied washing media for plastic recycling. Solvents, such as ethyl acetate, are also gaining interest because of their potential deep-cleaning capabilities with polymers (Ügdüler et al., 2020; Zhao et al., 2018). An exemplar industrial process that applies ethyl acetate to purify plastic waste is the Nordenia Extraction and Cleaning process (Velzen and Jansen, 2011).

Demets et al. (2020) investigated the deodorization of plastic films using an industrial water-based washing process. Results showed that highly polar compounds were efficiently removed; however, apolar compounds were abundantly present after industrial washing. The poor deodorization efficiency achieved by hot water for polyethylene (PE) films was reported by Cabanes et al. (2020a). It was suggested that deodorization might be improved by the addition of surfactants or by applying fewer polar solvents. Such washing media were tested for a mixed plastic film fraction at the lab scale by Roosen et al. (2020a). Results showed that water and caustic soda were relatively inefficient, achieving average deodorization efficiencies between 50 and 65%. At least a detergent or organic solvent was needed, increasing the deodorization efficiencies to 70–90%.

Compared with plastic films, industrial washing procedures for poly(ethylene terephthalate; PET) bottles are quite mature. The PET bottle recycling industry generally applies a water medium containing 2–3% NaOH and certain detergents, such as alcohol ethoxylates and ethoxysulfates (Welle, 2011). Towards closed-loop recycling, PET bottles are, in most cases, also subjected to an additional post-treatment. For instance, during solid-state post-condensation, PET bottle flakes are heated to a temperature above 200 °C (below their melting point) and maintained for several hours (Molnar and Ronkay, 2019). Hence, the polymer chains can be restored in length, and the remaining VOCs and other contaminants can be further removed (Dutra et al., 2011).

Strangl et al. (2021) showed that washing polypropylene (PP) packaging with water at 80 °C significantly deodorized the material as the intensity declined from 7.4 to 4.0 on a scale of 0 to 10 based on the sensory evaluation of an odor panel. Still, the odor intensity was still not comparable to that of virgin plastics (intensity rating of 2.1). Hence, the authors suggested that additional approaches for comprehensive odor reduction were essential to ensuring high-quality post-consumer PP recyclates.

Strangl et al. (2018) also demonstrated that a conventional mechanical recycling process only resulted in a slight reduction of odor-causing substances on high-density polyethylene (HDPE) bottles. With a more advanced decontamination technique using a hot-air stream to flush out the odors, deodorization efficiencies up to 75% were reported (Strangl et al., 2019). An example related to the use of hot air to remove odorous substances is the ReFresher technology commercialized by the Erema company (EREMA, 2016). This process leverages a decontamination unit where extruded pellets are flushed with hot air for typically 4–7 h

(Kol et al., 2021). In another study by Cabanes and Fullana (2021), steam stripping and poly(ethylene glycol) extraction were applied as deodorization steps for HDPE bottles. Both methods showed a decrease of more than 70% in VOC content compared with conventional extrusion comprising a degassing system to vacuum the VOCs released through the extrusion line. Yet, the economic feasibility of these processes has yet to be demonstrated.

Various studies exist in the field of plastic waste deodorization. However, a systematic comparison of deodorization efficiencies achieved with different washing media is still lacking. Therefore, in this study, we compare the deodorization efficiency of different plastic packaging types by applying the following approach:

- (1) Identifying odor compounds that are present on different post-consumer plastic packaging waste fractions as sorted by a Belgian waste management company (i.e., PE bottles, PE films, PP bottles, PP trays, PET bottles, PET trays, and polystyrene (PS) trays);
- (2) Determining the efficiency of different washing media, including water, detergents, caustic soda, and ethyl acetate, for each waste fraction;
- (3) Investigating the influence of polarity and chemical classes of odor compounds on their respective removal efficiencies.

2. Materials and methods

2.1. Chemicals and reagents

Carbon disulfide (CS_2 ; >99.0% purity), sodium hydroxide (NaOH ; >99.0% purity), ethyl acetate (EtOAc ; >99.5% purity), and hexadecyltrimethylammonium bromide (CTAB ; >98.0% purity) were supplied by Sigma Aldrich (Merck). An industrial detergent mixture containing a mixture of nonionic surfactants and oxidizing agents was supplied by a plastic recycling company from The Netherlands, and the dishwasher tablets, consisting of 15–30% peroxide-based bleach solution, <5% polycarboxylates, nonionic surfactants, phosphates, and enzymes, were supplied by a Belgian retailer. All chemical compounds were used without any preceding purification. Acenaphthene-D10 (99 atom % purity, Sigma Aldrich) was used as the internal standard for gas chromatography–mass spectrometry (GC–MS) analysis to enable the use of relative peak areas. Prior to use, all glasswork was cleaned with water and acetone and dried at 105 °C for at least 2 h to avoid contamination.

2.2. Sample description

To cover a relevant fraction of the most commonly sorted plastic waste fractions in Europe, 5 kg of the following packaging types were sampled at the outlet of the Belgian material recovery facility: (1) PET bottles, (2) PE bottles, (3) PP bottles, (4) PET trays, (5) PP trays, (6) PS trays, and (7) PE films (Kleinhans et al., 2021). The samples were collected at the end of a typical sorting line unwashed and unshredded.

Subsequently, the samples were reduced in size using a Piovon type RSP15/30 shredder with a sieve diameter of 8.0 mm to obtain more homogeneous samples. An extra sieving step using a sieve shaker (Endecotts LTD) was applied to obtain particles of a narrower size distribution between 2.0 and 5.0 mm. For this fraction, the largest amount was obtained. The samples were stored in sealed plastic sample bags with a minimum amount of headspace in a dark environment under a controlled temperature between 3 and 5 °C.

2.3. Washing experiments

A sample of 5.00 ± 0.10 g of the shredded and sieved plastic material was weighed and transferred in an Erlenmeyer having a volume of 250 mL. Next, 100 mL of a washing medium preheated to 25 or 65 °C was added. Mixing of the plastics and the washing medium was performed using a multi-flask rotary shaker. The rotation speed was 200 rpm, as optimized in our previous work (Roosen et al., 2020a). Because the rotary shaker was provided with a heating element, the temperature of the washing medium was maintained at the desired temperature of 25 or 65 °C for 10 min. The overall methodology of the washing procedure is schematically illustrated in Fig. 1a.

The following washing media were tested in this study: (1) tap water of drinking quality; (2) CTAB in a water solution (9.2 mM corresponding to 10 times the critical micelle concentration (CMC)); (3) NaOH in water solution (2% w/w); (4) CTAB in NaOH solution (9.2 mM CTAB in 2% w/w NaOH solution); (5) EtOAc (concentrated); (6) dishwasher tablets in water (one capsule of 18 g per 100-mL H_2O) and; (7) an industrial detergent (0.5 v/v% in 2% w/w NaOH solution as described by the company applying this detergent). After the 10-min washing step, the plastics and washing medium were separated with a filtration step. Subsequently, the plastics were rinsed with 100-mL distilled water at 25 °C to remove the remaining chemicals. After rinsing, the plastics were dried in a desiccator at room temperature for 24 h.

2.4. Analytical method

2.4.1. Identification of VOCs

As a first step, VOCs were investigated via headspace solid-phase micro-extraction, followed by headspace (HS) solid-phase micro-extraction (SPME) GC–MS. Two SPME fibers were applied to identify a wide range of compounds, namely a 100- μm polydimethylsiloxane (PDMS) and a 75 μm carboxen/PDMS fiber (Supelco, Sigma-Aldrich N.V., Bornem, Belgium). Prior to sampling, the fibers were thermally conditioned at 250 and 300 °C, respectively. 5.00 ± 0.10 g of plastic material was transferred in a 40 mL vial and sealed with a Mininert valve. Next, the vial was preheated to 65 °C in a thermal bath for 10 min, followed by exposing the described fibers to the headspace above the sample. After 15 min, the fibers were retracted from the vial and transferred to an Agilent 6890 gas chromatograph coupled to a Hewlett Packard mass-selective detector 5973

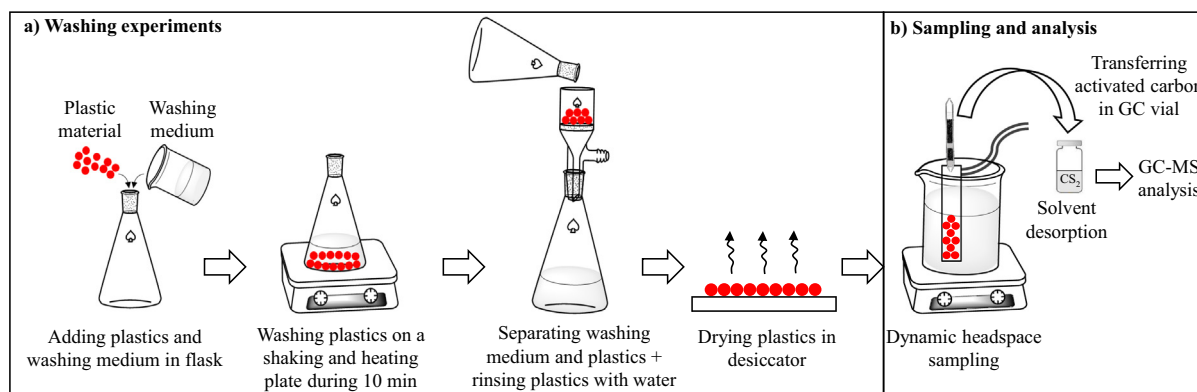


Fig. 1. Schematic representation of the methodology used, comprising (a) the washing experiments and (b) the sampling and analysis procedure.

equipped with a cross-linked (5%-phenyl)-methylpolysiloxane (HP-5 ms, Agilent) column (30 m × 0.25 mm, 0.25 μm). The initial temperature of the injection port was set to 50 °C. Helium was used as a carrier gas with a linear flow rate of 1 mL/min. The oven program was set to 50 °C for 1 min, then increased to 175 °C at 5 °C/min, maintained for 1 min, followed by an increase to 325 °C at 25 °C/min. Detected compounds were tentatively identified via the National Institute of Standards and Technology mass spectral library based on mass spectra and retention indices available in the literature.

2.4.2. Quantification of deodorization efficiencies

A dynamic headspace sampling method followed by solvent desorption and GC-MS analysis was applied to quantify deodorization efficiencies. The components analyzed with this method were selected based on three (or more) preliminary SPME analyses. The measurements were done in selective ion mode (SIM). Only compounds having a sufficient high mass spectral matching (at least 70%) were included in the SIM method. Like the methodology applied to tentatively identify VOCs, 5.00 ± 0.10 g of plastic material was transferred into a 40 mL vial and preheated in a thermal bath of 65 °C for 10 min (see Fig. 1b). An activated Dräger sampling tube in accordance with the procedures recommended by the National Institute for Occupational Safety and Health was attached to the plastic cap of the vial. The vial was flushed with pressurized air at a constant flow rate of 100 mL/min for 60 min.

Subsequently, the activated charcoal was transferred into a 1.5 mL microvial with 1.0 mL of CS₂ spiked with 50 ppm of the internal standard acenaphthene-d₁₀. The vials were sealed with polytetrafluoroethylene caps. Until injection into the GC-MS, the samples were stored in a dark environment with a controlled temperature between 3 and 5 °C. 1 μL of the solvent phase was splitless injected via a Supelco split/splitless-type wool-packed liner with a single taper design. Data analysis was performed via Agilent mass-selective detection Chemstation software.

2.5. Data processing

Based on the ratio of the relative peak area before and after washing, the removal efficiency of each individual odor compound was calculated, as shown in Eq. (1).

$$\eta = \frac{PA_w}{IS_w} \times \frac{IS_u}{PA_u} \times 100\% \quad (1)$$

where η is the deodorization efficiency (%), PA_w is the peak area of a certain VOC detected on the washed sample, PA_u is the peak area of the same VOC detected on the unwashed sample, IS_w is the peak area of the internal standard (50 ppm) of the washed sample, and IS_u is the peak area of the internal standard (50 ppm) of the unwashed sample.

The mean deodorization efficiency is calculated by averaging the deodorization efficiency of all analyzed odor compounds, giving equal weights to all odor compounds because odor is caused by a complex mixture of VOCs. This method was applied in our previous work (Roosen et al., 2020a). The experimental data were analyzed using SPSS® Statistics v27 software. Results were expressed as the mean of the analyzed odor compounds, accompanied by a box-plot to indicate the variation of the deodorization of the analyzed VOCs. The data were assessed for normality and conformance with a normal distribution. One-way analysis of variance was performed to compare the homogeneity of the assessed differences among groups and independent samples, and a *t*-test was used for between-group comparisons. A *p*-value <0.05 is considered statistically significant.

To investigate the influence of the polarity of the detected VOCs on the deodorization efficiencies, the octanol-water partition coefficient (log P_{ow}) of each detected VOC was calculated with ChemDraw Professional v19.1 software (PerkinElmer Informatics). The log P_{ow} describes the equilibrium distribution of a solute in a system composed of water and 1-octanol and is reported as the logarithm of the ratio of the concentrations of solute in the

two solvents (Kundi and Ho, 2019). The log P_{ow} is often used as a measure of the polarity of a certain solute.

3. Results

3.1. Identification of VOCs

In this study, we first tentatively identified VOCs from different packaging types via HS-SPME-GC-MS. In total, 169 VOCs were detected across all packaging types. These VOCs were divided into various chemical classes, as shown in Fig. 2a. A detailed overview of the detected VOCs per packaging type can be found in the Supplementary material, Table S1.

Fig. 2a indicates that the analyzed packaging types have different VOC profiles in terms of chemical groups and detected compounds. Generally, more VOCs were detected on plastic bottles (between 48 and 64 VOCs) than on plastic trays and films (between 29 and 46 VOCs). This was mainly caused by the significantly higher number of terpenes (*p*-value <0.001) and aromatic VOCs (*p*-value = 0.032) that were present in the bottle fractions. Trays and films, on the other hand, contained a significantly higher diversity of alkanes. For example, 16 different alkanes were detected in the headspace above the PE films, whereas, in the headspace above the PE bottles, only six different alkanes were detected.

The difference in terms of VOC chemical structure can be explained by the different applications and chemical structures of the packaging materials. PET bottles, for instance, are often used for the preservation of soft drinks, which frequently contain certain flavor substances. From a chemical perspective, such flavor substances often belong to the terpenes group (e.g., limonene and pinene; Franz et al., 2004). Household plastic trays or pots, on the other hand, are mainly used for the preservation of meat or dairy-based products, such as cheese or yogurt (Roosen et al., 2020b). This explains the presence of oxygenated compounds, such as aldehydes and ketones, which are known to be secondary oxidation products formed during the lifetime of meat and dairy products and are responsible for their rancidity (Song et al., 2020). Alkanes, alkenes, aldehydes, ketones, alcohols, esters, and acids are also known to be odor-causing thermal oxidation products of PE and PP (Demets et al., 2020). The findings in this work agree with those of Strangl et al. (2018), who already indicated that the possible sources, formation pathways, and routes of introduced odoriferous constituents are a combination of oxidation products and migration processes of fragrances and packaged food and the degradation/spoilage thereof.

Whereas PET and PE bottles contain a relatively high number of VOCs, their distributions in terms of polarity are narrower than the VOCs detected on trays and films, as indicated in Fig. 2b. For instance, the difference between the lowest and the highest octanol-water partition coefficient (log P_{ow}) values detected on PET bottles was 8.0, whereas for the PE films, this difference was 10.4. Moreover, the highest detected log P_{ow} values of odor compounds present on bottles (i.e., hexadecane with a log P_{ow} of 7.18) was lower than those on trays and films (i.e., pentacosane with a log P_{ow} of 10.93).

Another factor that can influence the number of odor compounds is polymer glassiness or rubberiness. Hence, polymers can be classified into rubbery or glassy polymers. Glassy polymers have a more compacted structure with closed internal nanoscale pores, whereas rubbery plastics have more volume between the molecules (Robeson et al., 2015). Therefore, when compared at equal permeabilities, rubbery polymers, such as PE and PP, have higher diffusion coefficients than do glassy polymers, including PSs. Thus, they may be able to adsorb more odor compounds than glassy polymers, which corresponds with our results. However, this hypothesis should ideally be confirmed by further research.

3.2. Deodorization efficiencies

The deodorization efficiencies per packaging type and medium are shown in Fig. 3 in the form of a box-plot with mean, median, maximum, minimum, 1%, and 99% values. A detailed overview of the deodorization efficiencies per analyzed odor compound and packaging type for each

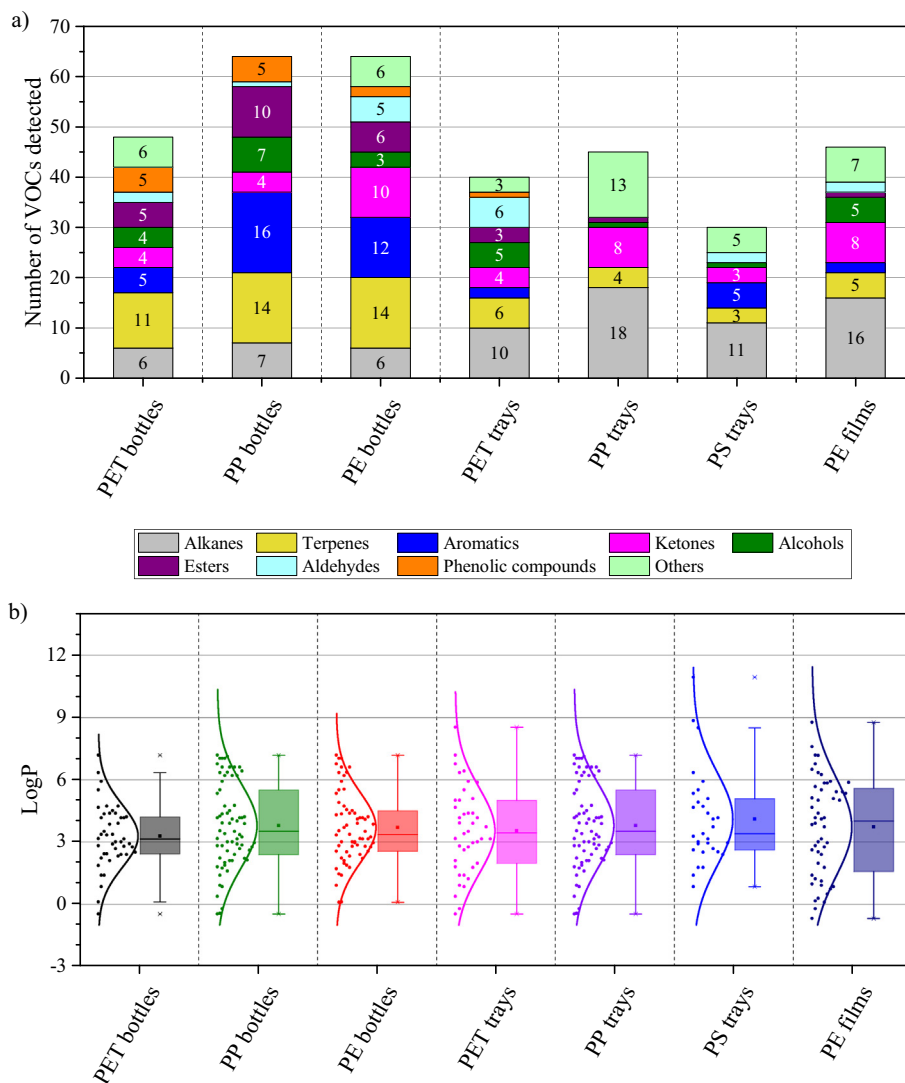


Fig. 2. Tentatively identified VOCs on different plastic packaging types: (a) categorized into various chemical classes and their respective number of detected compounds; (b) box-plot and data point distribution in function of their octanol-water partition coefficient ($\log P_{ow}$).

tested washing medium can be found in the Supplementary materials, Table S2. Note that for PS trays, no deodorization efficiencies were reported after treatment with ethyl acetate, as this solvent causes the dissolution of PS.

It can be observed that different washing media resulted in different deodorization efficiencies. Most washing media applied to PET bottles resulted in higher deodorization efficiencies compared with the same washing medium applied to other packaging types, such as PE and PP bottles. For instance, washing PET bottles with water at 25 °C resulted in an average removal efficiency of 63%, whereas the same medium only achieved 43 and 46% deodorization for PE and PP bottles, respectively, which is significantly lower (p -value <0.01). An even lower p -value <0.001 was observed for a NaOH solution with an average removal efficiency of 74% for PET bottles and 36 and 45% for PE and PP bottles, respectively. This can be explained by the fact that caustic soda can hydrolyze certain polymers, such as PETs, which might activate a polymer's surface. However, this also implies that the applied temperatures and caustic soda concentrations must be monitored to avoid complete alkaline hydrolysis of the polymer (Caparanga et al., 2009). The use of surfactants in an alkali environment is commonplace for the recycling of PET bottles. It has been stated that the role of surfactants is to lower the surface tension, γ , of the washing solution; hence, the penetration capacity is enhanced (Fukuzaki et al., 2006). This indicates that the wetting of a polymer's surface and

the displacement of contaminating substances by adsorption of detergent compounds alongside improved stabilization of the desorbed compounds in the washing solution are important deodorization mechanisms of detergent-containing solutions.

Alternatively, adding a detergent to polyolefin-based packaging types increases the deodorization efficiency by 20–30% compared with washing with a NaOH solution. For PET bottles, the effect of detergents was far less pronounced, with deodorization efficiencies in the same order of magnitude as achieved by a NaOH solution. This implies that for PET bottles, adding NaOH is key, whereas for polyolefin bottles, adding a detergent is essential to improving deodorization efficiencies. This is clearly an important conclusion of this study, as recyclers of PE and PP bottles typically mimic the washing chain applied to the PET bottle recycling industry, where a combination of caustic soda and detergents is used (Pinke et al., 2019). However, Fig. 3 indicates that, in terms of deodorization, a washing step with caustic soda and detergent is not the best deodorization strategy for PE and PP bottles, and only applying a detergent without NaOH is recommended. Moreover, in terms of cost, safety, and downstream cleaning of the washing medium (e.g., wastewater treatment), only the addition of a detergent without NaOH is an option. However, it should be noted that adding caustic soda might be required for other functionalities, such as label removal, and it is known to dissolve some glues (Jabłońska et al., 2019). However, for rigid PEs and PPs, a first wash with a caustic soda in

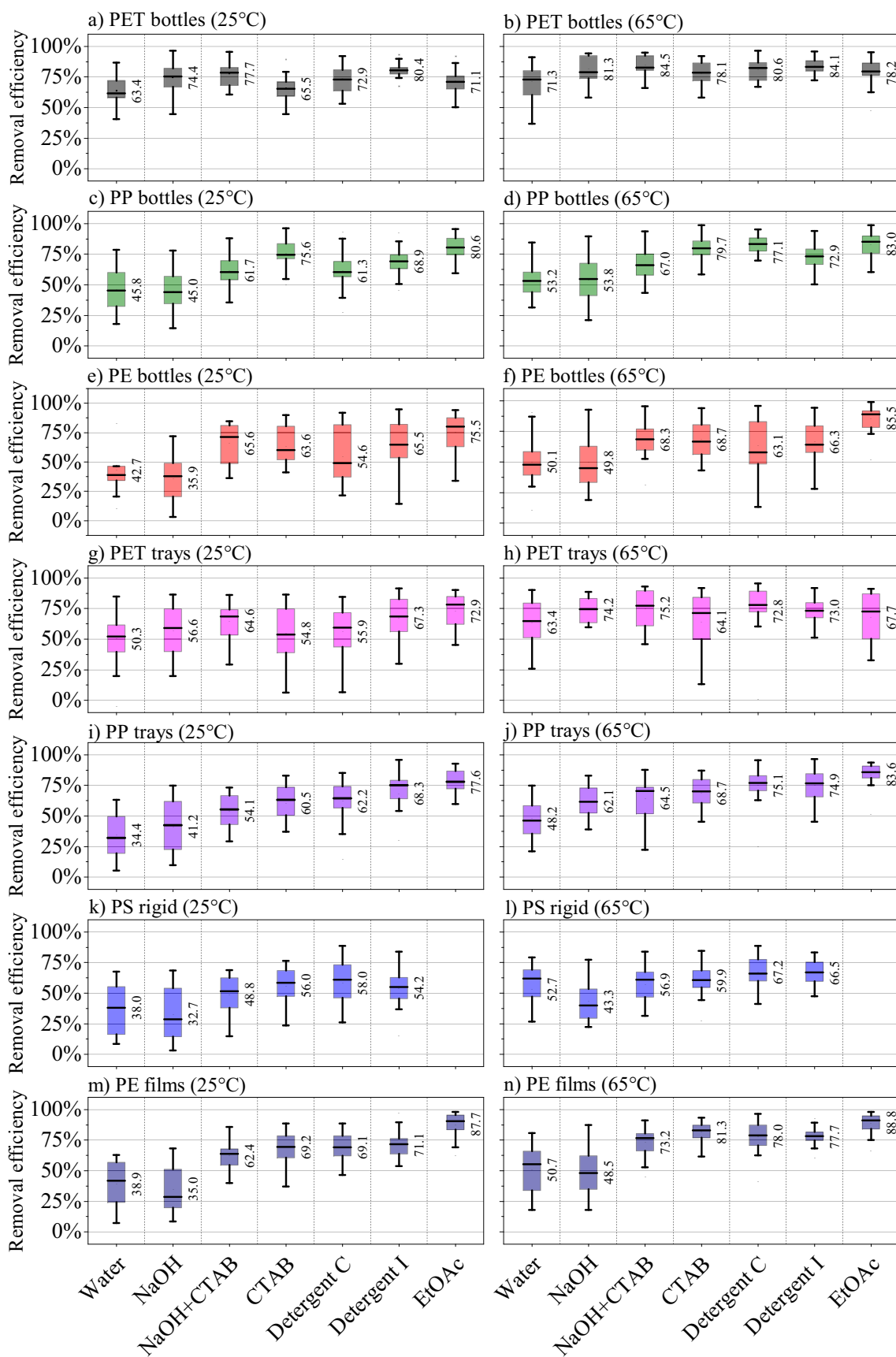


Fig. 3. Box-plot of the removal efficiencies of selected odor compounds for (a) PET bottles at 25 °C, (b) PET bottles at 65 °C, (c) PE bottles at 25 °C, (d) PE bottles at 65 °C, (e) PS trays at 25 °C, (f) PS trays at 65 °C, (g) PET trays at 25 °C, (h) PET trays at 65 °C, (i) PP bottles at 25 °C, (j) PP bottles at 65 °C, (k) PE films at 25 °C, (l) PE films at 65 °C, (m) PP trays at 25 °C, and (n) PP trays at 65 °C.

a (semi-) closed system to remove labels and organic residue followed by a second wash containing a detergent and with sufficient recirculation of the washing medium to deodorize the plastics might be a more preferred washing configuration.

Detergents clearly improve the deodorization efficiencies of plastic packaging, as reported by Roosen et al. (2020a). This can be explained by the chemical structure of detergents, which enables the stabilization of dissolved substances with redeposition prevention. Three different surfactant-containing solutions were compared in this study: (1) 9.2 mM CTAB, a cationic surfactant in water; (2) a commercially available dishwasher capsule containing non-ionic surfactants; and (3) an industrially applied detergent mixture also containing non-ionic surfactants and oxidizing agents. CTAB was selected to evaluate the deodorization efficiency because cationic surfactants are most effective for the removal of printing inks (Chotipong et al., 2006; Gecol et al., 2004, 2003, 2002, 2001; Songsiri et al., 2002), which, alongside odor, is an important prerequisite to enabling a circular economy for plastic packaging. Commercial dishwasher capsules were selected because of their potentially interesting composition since they contain surfactants, peroxide-based bleach compounds and enzymes. An industrial detergent mixture was selected as the benchmark. Fig. 3 shows that only slight differences in deodorization efficiencies were measured when using the different surfactants. For instance, deodorization efficiencies at 25 °C for a rigid PS varied between 54 and 58% for the industrial detergent and the commercial detergent, respectively, which is not statistically significant (p -value = 0.49). Similarly, for PE films, efficiencies between 69 and 71% were achieved with the different surfactants at 25 °C (p -value = 0.56). In summary, adding a detergent seems to be more important for improving deodorization efficiency than the category of surfactant added. Hence, additional detergent mixtures were not tested in the present work. However, as there is an enormous diversity in terms of detergents and mixtures thereof, other detergents, such as sodium dodecyl sulfate, might be effective in industrial application and testing in further research to investigate the exact mechanisms of detergents in the removal of odorous compounds and other substances.

For polyolefin-based packaging waste, surfactants typically achieve deodorization efficiencies between 55 and 70%, which is 10–20% better than water. Thus, for these waste fractions, the deodorization efficiency can be improved by changing the polarity and/or decreasing the surface tension of the washing medium. This can be confirmed by applying an organic solvent, such as ethyl acetate, which is apolar and has a lower surface tension (23.75 mN/m) than water (72 mN/m), resulting in higher removal efficiencies of up to 81% for PP bottles and 88% for PE films. This is 10–15% better than washing with a water-based detergent medium, which has surface tensions (at concentrations above the CMC) around 40 mN/m (Chotipong et al., 2006). However, further research is needed to gain more fundamental insights into the roles of surface tension in deodorization.

Related to PET trays, when considering just the base PET, it is expected that a caustic solution is more efficient than an organic solvent, such as with PET bottles. Yet, Fig. 3 shows the opposite results, with an average deodorization efficiency of 65% after washing with a caustic solution and 73% after washing with ethyl acetate. This might be explained by understanding the specific composition of multilayer PET trays. For these packages, PET is mainly used on the outside of the tray and lid for its water and gas-barrier properties and its mechanical strength. PE is mainly used on the inside for its attractive sealing properties (Roosen et al., 2020b). Thus, odorous constituents migrate/sorb from the packaging content first to the PE layer. Consequently, it can be expected that, in those cases, the PE layer is more contaminated with odorous substances than the PET layer. Hence, washing media that are more efficient for polyolefin-based packaging types, such as ethyl acetate, is also more efficient for PET trays. Likewise, Fig. 3 shows that for PE films, the highest deodorization efficiency was obtained with ethyl acetate: 89% at 65 °C, which is significantly higher than, for instance, the 78% achieved with the industrial detergent mixture (p -value <0.001). Hence, this study confirms the great potential of ethyl acetate as a deodorization medium (Roosen et al., 2020a; Velzen and Jansen, 2011). However, this is the first time it has been systematically tested for various packaging

types. A promising technology for the deodorization of plastics is the application of a (hot) aeration process. Strangl et al. (2019) investigated an aeration-based purification process for recycled HDPE pellets in a decontamination reactor after an optimized extrusion process that included homogenization, melt filtration, and degassing. In that reactor, the pellets were kept at a high temperature, and a hot air stream was applied to flush out the odors. It was stated that the residence time in the reactor is critical for odor reduction; it is also a decisive economic factor. Based on the data presented by Strangl et al. (2019), an average deodorization efficiency of 86% was achieved after a residence time of 7 h. When comparing this to the data obtained in this study from washing HDPE bottles with ethyl acetate at 65 °C, the same deodorization efficiency of 86% was achieved with a residence time of 10 min. Hence, both deodorization strategies are competitive in terms of efficiency. Nevertheless, solvent-based washing seems to be a considerably faster process. As a downside, applying a solvent in a recycling chain might induce higher costs than if water-based media or aeration were used. However, a solvent might also induce the removal of less volatile compounds, such as certain additives and inks, which could improve the cost–benefit ratio of such a process. Applying solvents or less polar washing media and applying aeration could be considered future research, including the design of appropriate reactors and equipment and the downstream processing of the applied medium. Ideally, this would be followed by a life cycle assessment and techno-economic assessment to compare the best available techniques in terms of their economic and environmental performance.

Another important aspect that should be included in future research is the potential remnants of the solvent used on the plastic material, especially regarding food contact approval. By using a semi-polar solvent, such as ethyl acetate, a water-based post-wash could remove a significant amount of the remaining solvent. Of course, this would create a water-solvent byproduct. Apart from this, mechanical and/or thermal drying followed by a degassing system during extrusion should eventually be applied to remove the applied solvent (Kol et al., 2021). However, the solvent that penetrated the plastic after washing and its potential release into the product still require investigation. Moreover, other technologies, such as sterilization and the use of certain probiotic bacteria, have potential in treating certain contaminated plastic waste streams and should remain within the scope of further research (Khoo et al., 2021; Mazari et al., 2019).

Fig. 3 also indicates that increasing temperature from 25 to 65 °C enhances the average removal efficiencies between 5 and 15%, resulting in a statistical relevance difference (p -value = 0.004). However, in some cases, the influence of temperature is less significant. For instance, washing PE films with ethyl acetate at 25 °C achieved a removal efficiency of 88%, whereas washing at 65 °C achieved a removal efficiency of 89%. Hence, it can be concluded that temperature has an impact, but it seems not to be the major factor in optimizing plastic deodorization. Reasonable efficiencies can be achieved at room temperature if a suitable medium is selected, and the washing medium is not saturated when it is brought into contact with the plastics. However, this hypothesis should ideally be confirmed by further research focusing on optimal temperatures.

3.3. Influence of chemical classes and polarity of odor compounds

The structural diversity of odor compounds makes them challenging to remove. As indicated in Fig. 2, the VOCs that were detected on different plastic packaging types contained a wide range of functional groups. In Fig. 4, the average deodorization efficiencies (from 0 to 100% deodorization with 100% being most preferred) of five chemical groups are plotted in a spider chart after washing with three different media: water, caustic soda with CTAB, and ethyl acetate per analyzed plastic packaging type. The chemical groups included carboxylic acids, aldehydes, terpenes, aromatics, and alkanes. The average $\log P_{ow}$ value of the VOCs belonging to the corresponding chemical group is indicated next to the name of the corresponding chemical group in Fig. 4.

It can be observed that, depending on the packaging, polymer type, and washing medium, the analyzed VOCs per chemical group behave

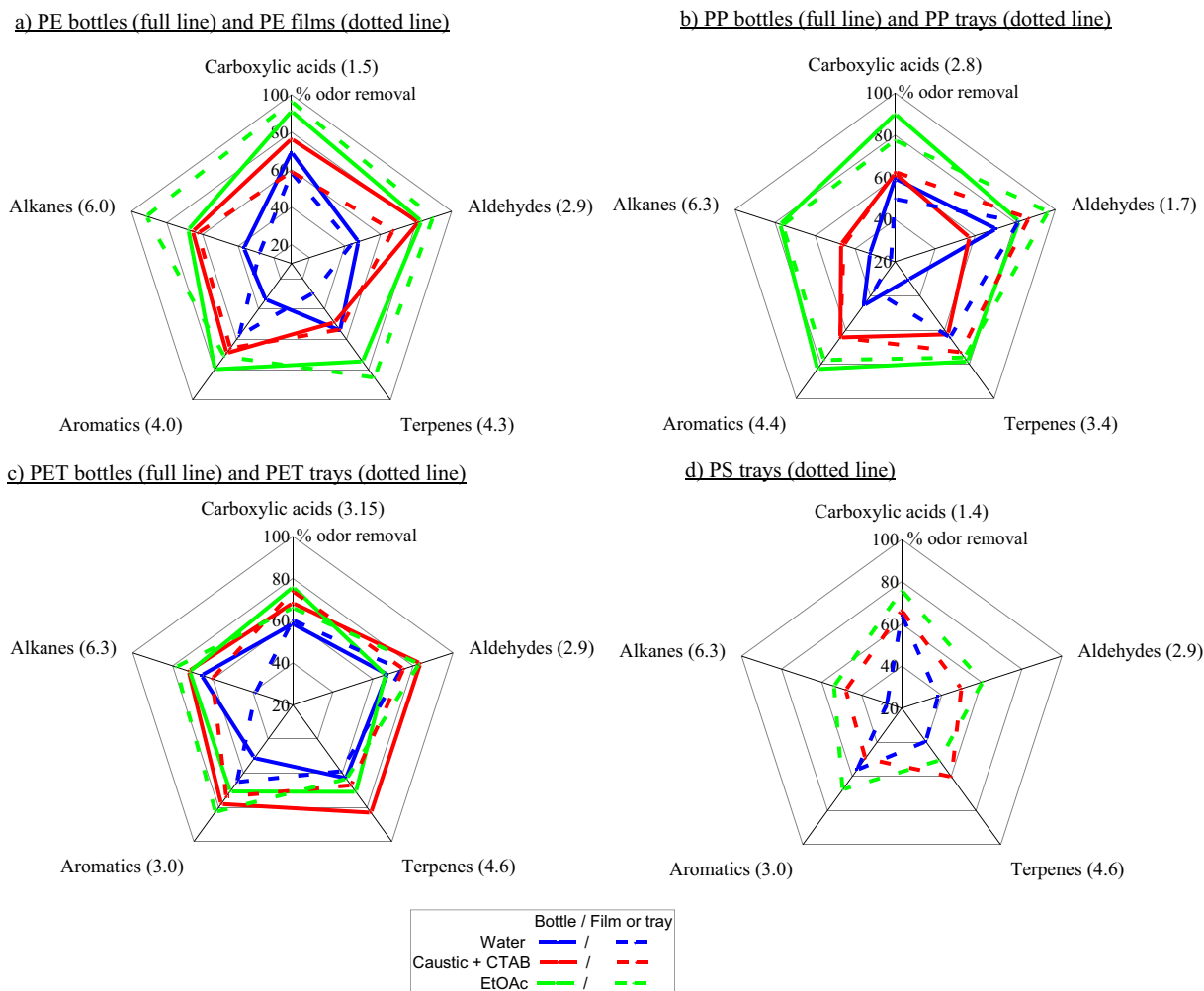


Fig. 4. Spider diagram of the average deodorization efficiencies on a scale from 0 to 100% for five different chemical classes (i.e., carboxylic acids, aldehydes, terpenes, aromatics, and alkanes) and the average octanol-water partition coefficient ($\log P_{ow}$) of the VOCs that belong to these chemical classes: (a) for PE bottles and PE films; (b) for PP bottles and PP trays; (c) for PET bottles and PET trays; and (d) for PS trays.

differently in terms of deodorization. For instance, alkanes were generally less efficiently removed after a washing step with water, and usually less than 40% removal was achieved. This is a logical result, as alkanes are the most apolar group among the analyzed chemical groups. Examples of alkanes that were detected on the analyzed packaging types are undecane ($\log P_{ow}$ of 5.09), dodecane ($\log P_{ow}$ of 5.51), tetradecane ($\log P_{ow}$ of 6.34), and eicosane ($\log P_{ow}$ of 8.84). On average, the alkanes detected in this study have a $\log P_{ow}$ value between 6.0 and 6.3. This is considerably higher than the other chemical compounds depicted in Fig. 4. When adding a detergent or solvent, higher removal efficiencies for alkanes can be achieved: between 60 and 80%.

A similar trend was observed for aromatic compounds and terpenes. They can still be considered apolar, with average $\log P_{ow}$ values between 3.0 and 4.4 and 3.4 and 4.6, respectively. In the case of aromatic compounds, the average deodorization efficiency with water was between 30 and 50%. This is slightly higher than the average deodorization efficiency of the more apolar alkanes. The most detected aromatic compounds were xylene, benzene, styrene, and derivatives thereof. Aromatic compounds can be formed based on the thermooxidative degradation of polymers (Demets et al., 2020) or, in the case of PS, due to the emission of, among other things, residual monomers, side products of the polymerization reaction, and degradation products containing a benzene ring (Cabanes et al., 2020b).

Terpenes are known to be present in post-consumer plastic packaging due to their use as flavor and fragrance substances in both food (Triaux

et al., 2021) and non-food (Buettner et al., 2018) applications. For instance, limonene and alpha- and beta-pinene are widely reported as odorous constituents present due to the migration of external substances during a packaging's lifetime or through the plastic waste collection chain (Cabanes et al., 2020b). For terpenes, the detergent-containing and solvent-based washing media were significantly more efficient than washing with only water. For instance, terpenes were removed on average around 30% from PP bottles when washed with water, whereas a 60 to 80% removal efficiency was achieved when washed with detergent and ethyl acetate, respectively.

Aldehydes having an average $\log P_{ow}$ between 1.4 and 3.2 and carboxylic acids with an average $\log P_{ow}$ between 1.7 and 2.9 are more polar compounds than terpenes, aromatics, and alkanes. This affects the removal efficiencies of these compounds after washing. Substances belonging to the groups of aldehydes and carboxylic acids include benzaldehyde, decanal, and acetic acids. Fig. 4 shows that washing with water is relatively efficient when removing such aldehydes and carboxylic acids (e.g., between 60 and 75% for PET bottles and trays). The improved efficiency of water for more polar odorous compounds was described by Demets et al. (2020), where a threshold of a $\log P_{ow}$ of two was indicated for efficient deodorization with an industrial washing process. Still, it can still be observed that, even for these more polar compounds, adding a detergent has a positive influence on the deodorization efficiencies. For instance, for PE bottles and films, carboxylic acids were removed at a rate of 90% with a detergent, whereas between 60 and 70% was achieved with only water.

The importance of polarity is more systematically illustrated in Fig. 5, showing the deodorization efficiencies of the analyzed odor compounds as a function of their $\log P_{ow}$ value for the different packaging types. It can be observed that the influence of polarity is most pronounced when packages are washed with water, as the slope of the linear regression line fitted to the experimental data points (indicated in blue for water) clearly decreases towards the more apolar compounds (on the x-axis). For instance, washing PE bottles with water results in a removal efficiency of around 60% for more polar compounds with a $\log P_{ow}$ of zero, whereas the fit indicates that when the $\log P_{ow}$ is higher than nine, the removal efficiency will decrease to less than 20%. Similar data were also obtained for the other analyzed packaging types. Only PET bottles were an exception to this, as with water, removal efficiencies between 50 and 60% for compounds having a $\log P_{ow}$ of nine were achieved. A potential explanation for this is found in the higher polarity of PET compared with PE, PP, and PS (McKeen, 2012). It is indeed stated that the adsorbate and adsorbent polarity have a strong impact on the adsorption equilibrium; as the polarity of an adsorbent increases, the affinity for apolar molecules decreases (Denayer et al., 1998). Thus, apolar odor compounds, such as alkanes, have a lower affinity

for more polar polymer surfaces, such as PET, than for more apolar surfaces, such as PE and PP.

The slopes of the linear fits can be decreased by adding a surfactant (indicated in red in Fig. 5). Generally, the decrease in removal efficiencies of the odorous compounds having a $\log P_{ow}$ of zero and for the odorous compounds with a $\log P_{ow}$ of 10 is only around 20%, whereas with water, this will be around 50%. Fig. 5 also shows that by using an organic solvent, such as ethyl acetate, the effect of the polarity of the odorous constituents can almost be neglected, as the slope of the linear fit is almost zero (indicated in green in Fig. 5). This was already reported for plastic films (Roosen et al., 2020a), but is now, for the first time, confirmed for other packaging types as well.

4. Conclusions

Depending on the polymer type and the main application field of a packaging type, odor compounds vary in number and physicochemical properties. Plastic bottles, for instance, contain more terpenes, whereas PE and PP trays and films contain more oxygenated compounds, such as aldehydes

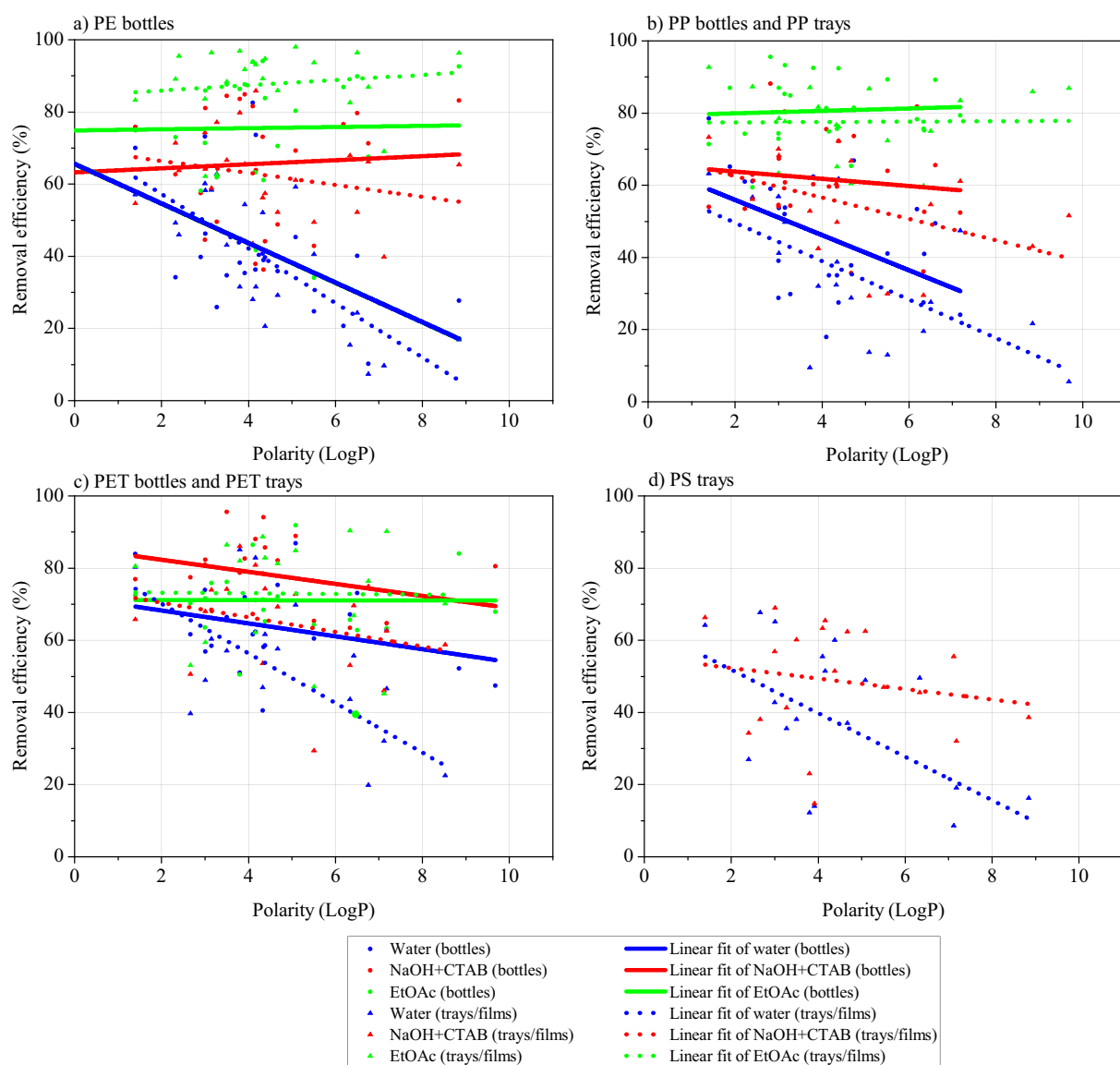


Fig. 5. Deodorization efficiency per odor compound as a function of the octanol-water partition coefficient ($\log P_{ow}$) after washing with water (indicated in blue), NaOH + CTAB (indicated in red), and ethyl acetate (indicated in green): (a) for PE bottles and PE films; (b) for PP bottles and PP trays; (c) for PET bottles and PET trays; and (d) for PS trays. Linear regression lines are shown in corresponding colors.

and ketones. This influences the deodorization efficiencies obtained with different washing media. For PET bottles, water and NaOH resulted in average deodorization efficiencies of 70–80%, which was higher than that achieved with water and ethyl acetate. For polyolefin packaging, on the other hand, water and NaOH achieved the lowest deodorization efficiencies, typically between 30 and 50%. At least a detergent (without caustic soda) or organic solvent should be applied for these materials, which results in average deodorization efficiencies up to 70 to 90%.

Thus, mimicking washing processes applied in the PET-bottle recycling industry for other packaging materials, such as PE, PP, and PS, is not the most efficient option in terms of deodorization, yet it is often the applied strategy to date. Hence, optimizing washing processes based on packaging type and physicochemical properties of the washing media and odor compounds is key to efficient deodorization; hence, it may broaden the application potential of mechanically recycled plastics.

CRedit authorship contribution statement

Martijn Roosen: Methodology, Investigation, Writing - original draft. **Lies Harinck:** Methodology, Investigation, Writing - review & editing. **Sibel Ügdüler:** Writing - review & editing. **Tobias De Somer:** Software, Data curation, Writing - review & editing. **Amaury-Gauvain Hucks:** Methodology, Investigation. **Tiago G.A. Belé:** Writing - review & editing. **Andrea Buettner:** Writing - review & editing. **Kim Ragaert:** Writing - review & editing, Funding acquisition. **Kevin M. Van Geem:** Writing - review & editing, Funding acquisition. **Ann Dumoulin:** Writing - review & editing, Supervision. **Steven De Meester:** Conceptualization, Resources, Writing - review & editing, Supervision, Project administration, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.scitotenv.2021.152467>.

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