COSMETIC **PACKAGING** GUIDANCE

HOW TO EVALUATE POST-CONSUMER POLYOLEFIN RECYCLATES IN COSMETIC PACKAGING?

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How to Evaluate Post-consumer Polyolefin Recyclates in Cosmetic Packaging?

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Abstract

Circular economy requires that post-consumer packaging materials should be recycled into new packaging materials. The application of post-consumer recyclates in cosmetic packaging should not raise any safety or health issue for the consumer. This document summarizes the knowledge on post-consumer contaminants in HDPE and PP recyclates as well as consumer safety evaluation and gives practical guidance and reliable criteria for the safe use of post-consumer HDPE and PP recyclates in cosmetic packaging applications.

Key Words

Cosmetic packaging; Polyolefin Recycling; Circular Economy; Post-consumer recyclates; Risk Assessment; Safety

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1 Introduction

Nowadays, nearly all European countries have established recollection systems for post-consumer packaging waste. These recollection systems guaranty the availability of large amounts of post-consumer plastics and led to increasing recycling capacities, which were installed all over Europe. With increasing circular economy and environmental demands, used plastic (post-consumer) packing are considered to be reused in packaging applications, in order to assure that packaging materials are kept in circulation as long as possible. This requires that post-consumer packaging materials be recycled into new packaging materials. The major challenge for the recycling of plastic waste into new cosmetic packaging applications is to ensure the safety of the consumer. Post-consumer substances or degradation products from the polymer or from polymer additives substances could migrate into the cosmetic product and endanger consumer health.

Different packaging polymers have different material properties, which includes also the diffusion behaviour of substances in the polymer. From a consumer safety perspective, it is important that the migration of polymer constituents (like polymer additives or monomers) into the filling is minimized to an unavoidable level, which as well could be evaluated as safe for the intended use conditions. Low diffusive polymers like polyethylene terephthalate (PET) with the associated low mass transfer (migration) into the filling is therefore the most promising polymer for circular economy. For more than two decades PET post-consumer recycling into new PET bottles had been established in Europe (Welle 2011). The post-consumer PET recyclate has been shown to be safe in the very sensitive area of food packaging materials (Welle 2013). On the other hand, high diffusive polymers like polyolefins (high density polyethylene HDPE or polypropylene PP) are processed to post-consumer recyclates which are typically going into less critical applications. However, in recent years increasing progress had been made to recycle post-consumer HDPE into new packaging applications for example into cosmetic packaging. The application of post-consumer recyclates in cosmetic packaging still should raise no safety or health issue for the consumer. Therefore, the recycling of post-consumer plastics into cosmetic packaging applications need knowledge about contamination levels from the first use of the packaging materials as well as knowledge about the migration from the packaging material into the filling in order to evaluate the risk for consumer's health.

The intention of this document is to summarize the knowledge on post-consumer contaminants in HDPE and PP recyclates and consumer safety evaluation. This should give practical guidance and reliable criteria for the safe use of post-consumer HDPE and PP recyclates in cosmetic packaging applications. Here in particular, the maximum concentrations of post-consumer contaminants in cosmetic packaging should be given which can be used in routine control to assure the high-quality standards of cosmetic packaging materials.

2 Classification of Input Streams

The input materials for the recycling processes play an important role in the risk assessment of the recyclates. As expected, the recollection system has an influence on the contamination levels of the recyclates. The input materials for a recycling process can be divided into the following different categories (EU 2005):

Class 1 are post-industrial materials, which are remaining from production. The history of the material is known, and the material was always under control of the packaging manufacturer. Provided, that contamination can be excluded or has been evaluated already by scientific groups, e.g. EFSA or FDA, this material can be reused in cosmetic packaging materials like virgin materials. Class 1 materials corresponds to FDA's primary recycling category (pre-consumer scrap).

Class 2 are post-consumer materials for well-known applications, which are recollected as pure grade by the recycler. Due the post-consumer character this material is usually not under complete control over the time period from its first use up to its return. Due to the pure grade sorting the contamination levels are typically low and the variation in the contamination levels is small. Examples for class two materials are the UK milk bottles or pure collected shampoo bottles. Also, all kinds of packaging materials, which are recollected by use of deposit systems fall into this category (e.g. PET bottles).

Class 3 are post-consumer materials recollected from mixed plastics collections and contain all kinds of packaging. Typically, such recollection systems are "green dot" collections or mixed plastics materials. Sometimes such materials are derived also from all-waste collections. Due to the unknown history and potential cross-contamination from non-food fillings as well as from non-cosmetic fillings, these input materials show a high contamination level with a broad range of different contaminants and concentrations. The contamination level may also vary from source to source. Sorting efficiency may be an important criterion to establish homogenous fractions with a low contaminant level and low variation range of these class 3 materials.

Class 2 and 3 correspond to US FDA category "physical reprocessing, secondary recycling" (US FDA 1992).

Class 4 are post-consumer materials, that had been chemically reprocessed by depolymerisation into monomers or oligomers. The monomers or oligomers are used after purification as input materials for re-polymerisation from which new packaging polymers can be manufactured. Class 4 materials correspond to US FDA's category "chemical reprocessing, tertiary recycling".

Class 1 and class 4 materials are typically considered as safe and can be re-manufactured together with virgin polymers into new packaging for food and cosmetic products. Class 2 and class 3 materials, however, might be contaminated and need to be further controlled. This document deals with class 2 and class 3 materials.

3 Recycling Process Description

The recycling processes for HDPE and PP mixed plastic collection in Europe typically have the following main recycling steps (Figure 1):

- Pre-sorting by polymer type.
- Grinding of the recollected post-consumer HDPE or PP bottles and trays for so-called flakes followed by an intense hot washing step.
- Surface drying of the washed flakes.
- Colour and polymer sorting on flakes level (optional step).
- Re-extrusion with vacuum degassing and filtration.
- Further deep cleaning of the re-extruded pellets by use of hot air or vacuum (optional step).

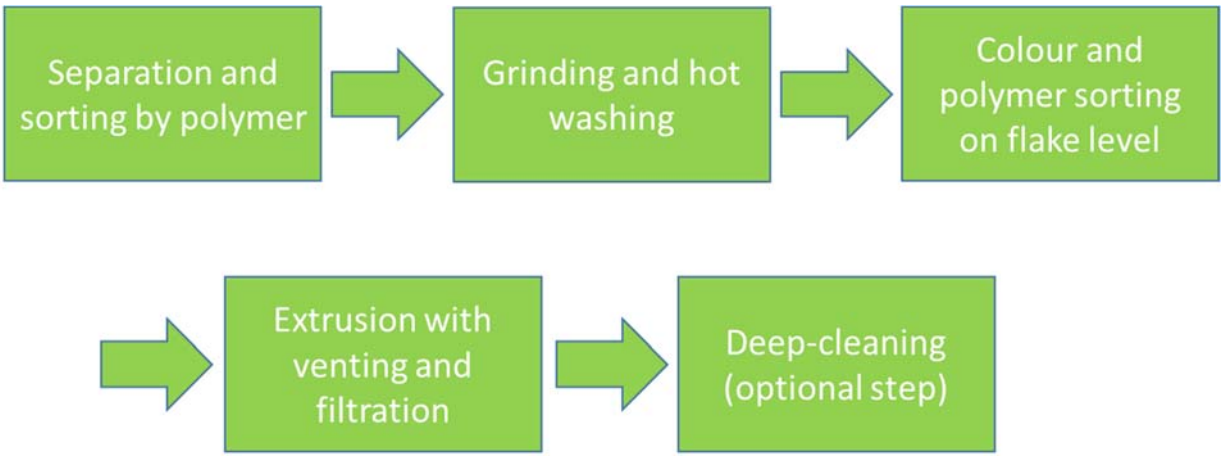

Figure 1: Recycling steps for HDPE and PP mixed plastic collections

The main decontamination steps for post-consumer substances are the warm or hot washing process, the extrusion as well as the (optional) further deep cleaning of the pellets. In addition, the washing process removes also dust and dirt, labels as well as printing inks on the surface of the flakes. The washing process step is therefore mandatory. Subsequently the washed flakes are surface dried e.g. by use of hot air. The cleaning efficiency of the washing process depends on the residence time of the flakes in hot water, the washing temperature as well as on process conditions of the drying step. Such a washing procedure is the so-called "conventional recycling step".

In principle, washed flakes can be directly used for the production of new cosmetic packaging. However, the flakes are typically extruded to pellets, because this allows a further decontamination with melt degassing and allows also a better performance of the cosmetic packaging compared to flakes as an input material. A further deep-cleaning step is optional. Within the last decade further recycling steps had been developed, which additionally reduces the contamination levels in post-consumer HDPE recyclates. Such steps consist in using high temperatures, stripping with air or inert gas, or vacuum in order to remove volatile substances from the recyclates. Due

to this additional treatment, deep-cleaned polyolefin recyclates show a significantly lower amount as well as lower concentrations of post-consumer substances. Therefore, the deep-cleaning process is an important step in the decontamination process of post-consumer polyolefins. Additionally, deep-cleaned polyolefin recyclates also show a lower off-smell.

The cleaning efficiencies of the conventional recycling processes are in most cases not available, because the unwashed inputs are difficult to characterise due to surface contamination. Therefore, the determination of the cleaning efficiency is typically started on ground and washed polyolefin flakes as input material for the recycling process. Consequently, cleaning efficiencies are available only for deep-cleaning processes (see Chapter 5.3). The cleaning efficiencies are determined in a "challenge test" by use of artificial contamination with worst-case concentrations of model contaminants (so-called surrogates).

4 Food Law Compliance of HDPE and PP Recyclates

All packaging materials (manufactured from post-consumer recyclates or from virgin materials) have to comply with Commission "Regulation (EC) No 1935/2004 on materials and articles intended to come into contact with food". Article 3 of this Regulation requires that food contact materials and articles do not transfer their constituents to food at harmful levels to human health. Furthermore, the packaging material must not change food composition or the organoleptic properties of the packed food in an unacceptable way.

In addition to these general requirements from Article 3, specific requirements are laid down in Regulation (EU) No 10/2011. Annex I of this Regulation contains a list of substances that are authorized to be intentionally used in the manufacturing process of plastic materials and articles. The "positive list" comprises (a) monomers or other starting substances; (b) additives excluding colorants; (c) polymer production aids excluding solvents; and (d) macromolecules obtained from microbial fermentation. Within this positive list also specific migration limits (SML) are given in order to ensure the safety of the final material or article.

The use of post-consumer recycled polymers in direct food contact is regulated by Commission Regulation (EU) 282/2008. Regarding the Recycling Regulation, the recycler has to provide a dossier to the European Food Safety Authority (EFSA). In principle, the evaluation of post-consumer plastics by EFSA for direct food contact is based on the three main criteria:

- Determination of the input concentration of post-consumer substances in recollected and washed flakes.
- Determination of the cleaning efficiency of the applied (deep cleaning) recycling process by use of artificial contaminated material (challenge test).

- Calculation of the exposure of the consumer towards residual post-consumer substances depending on the packaging application.

To date, EFSA has evaluated six recycling processes for multi-use crates. For more sensitive direct food contact, EFSA evaluated only two (more or less identical) recycling processes for the recycling of HDPE milk bottles into new fresh milk bottles. PP recyclates in food contact are not evaluated by EFSA to date.

As mentioned above, the EFSA approach for HDPE milk bottles (EFSA 2015) is based on the input contamination of the post-consumer recyclate, the cleaning efficiency of the recycling process as well as on the exposure to the consumer, which is discussed in the following section in more detail.

4.1 Input Contamination Levels in Washed Post-consumer HDPE Flakes

The input contamination of post-consumer contaminants in recycled HDPE flakes can be determined in a non-target screening of washed rHDPE flake samples. Such a screening should include different recollection and production days, regional differences in the recollection systems as well as seasonal differences due to higher sorption of post-consumer substances in summer times.

EFSA assumes contaminants of the input, which are necessary for the derivation of the input concentrations as:

- Not authorized substances for food contact.
- Incidental contaminants from previous uses including possible misuses.

Regarding not authorized substances for food contact: This means that substances are intentionally introduced in food packaging materials used for the input, which are not listed in Regulation (EU) 10/2011. Since most of the input materials are food packaging, all intentionally added substances are in compliances with Regulation (EU) 10/2011. However, an analysis of the input material (post-consumer washed HDPE flakes) should be made where non-authorized substances should be monitored.

Regarding incidental contaminants from previous uses including possible misuses: this represents all other substances found in the input material of the deep-cleaning recycling process, which are not detectable in virgin HDPE. These substances should be also monitored in a screening of the input materials.

Such a non-target screening was performed in a public funded project in UK. Within this project, washed post-consumer HDPE flakes from milk bottles were screened for any HDPE untypical substances. It was found (see Chapter 5) that two out of 24000 HDPE bottles show hints for misuse. The highest concentrations of contaminants in such flake samples were determined to approx. 6500 mg/kg. This leads to a mean input concentration of substances from misuse of 0.54 mg/kg in a washed HDPE flake sample. Based on the data given in the public funded project, EFSA assumes that a

concentration of 0.5 mg/kg of an unknown substance in the HDPE milk packaging material is the worst case.

4.2 Cleaning Efficiencies of the Applied Recycling Process

In the second step EFSA evaluates the cleaning efficiency of the recycling process. This cleaning efficiency is determined by use of artificial contamination in a worst-case scenario. Usually the input levels of the model contaminants (surrogates) are in the range of 500 mg/kg to about 1000 mg/kg. The initial concentrations in the washed and contaminated flakes are experimentally determined. The contaminated flakes are subsequently reprocessed with the recycling process in pilot or small-scale production plant. After each individual process step, samples are drawn and analysed regarding their residual levels of the model contaminants. From the difference between the input levels and the output levels, the cleaning efficiency can be calculated for each individual process step as well as for the overall recycling process. Due to the fact, that the initial concentrations for the applied surrogates are different in the challenge test (as well as between different challenge tests), the cleaning efficiencies are normalized to the worst-case initial concentration of 0.5 mg/kg. Together with the cleaning efficiencies, the normalized residual concentrations (c_{res}) after the deep-cleaning recycling can be calculated.

4.3 Consumer Exposure to Post-Consumer Substances

Subsequently EFSA looks into the exposure of the consumer with contaminants coming from recycled packaging materials. EFSA uses the Threshold of Toxicological Concern Concept (TTC) to evaluate consumers' health risk towards post-consumer substances from the recycled polymer materials. Within this TTC Concept, it is concluded that 0.0025 µg per kg body weight per day for an unknown contaminant represents a negligible risk for the consumer. This value includes genotoxic compounds. For the evaluation of the HDPE milk bottles with post-consumer recyclates, EFSA assumes that a toddler with 10 kg body weight (b.w.) consumes 90 g milk products per kg b.w. per day, so overall 900 g milk products per day. The maximum safe concentration in the milk is assumed to 0.028 µg/l. To evaluate the migration of a post-consumer substance, EFSA calculates the migration by use of diffusion modelling. Due to the fact, that the applied diffusion models overestimate the migration by a factor of at least two, the maximum tolerable migration into food is set to 0.06 µg/l. The contact conditions for diffusion modelling was set to 15 d at 5°C, which are applied typically for fresh milk bottles. By use of these storage conditions, EFSA calculated the maximum bottle wall concentration (c_{mod}) for all applied surrogates which corresponds to a migration value of 0.06 µg/l. The diffusion coefficients for this migration calculation derive from the general accepted A_P migration model (JRC 2010, JRC 2015).

4.4 Risk Assessment Post-consumer Recyclates in Food Contact

In the last step, EFSA compared the experimentally determined c_{res} value with the calculated c_{mod} value for each of the applied surrogates. EFSA considers a recycling process as safe if the residual concentration c_{res} of the challenge test is lower than the modelled concentration c_{mod} in the packaging material for all applied surrogates.

Regarding the milk bottle recycling processes, EFSA concluded that "the Panel noted the limited decontamination efficiency under the conditions of testing and concluded that the processes do not satisfy criteria set for HDPE. Uncertainties and consequent conservatism of the selected criteria could allow the conclusion that a process is safe when these criteria are met but not when they are not met. Therefore, the CEF Panel considered that, for the manufacture of recycled bottles for milks and fruit juices and trays for animal products, additional data should be provided before it can conclude on the safety assessment." (EFSA 2015). Therefore, the recyclates from post-consumer milk bottles cannot be used for direct food contact.

Assuming that food grade HDPE recyclates approved by EFSA will be on the market in future, it should be noted, that the recyclates are not necessarily safe also for cosmetic packaging. For example, according to the EFSA evaluation, a post-consumer recyclate is in any case assessed with a short-term migration contact scenario of only 15 d at 5°C. Cosmetic packaging has a much longer storage time which increases the migration into the product. On the other hand, the exposure of the consumer is much lower in case of cosmetic applications, especially for rinse-off products.

5 Contaminants in recycled HDPE and PP

5.1 European Overview on Migration Relevant Substances in rHDPE

Within a feasibility study of Beiersdorf and Werner & Mertz together with Fraunhofer IVV as packaging research laboratory, post-consumer rHDPE samples commercially available in Europe were investigated. Four categories of samples were analysed to facilitate the comparison and discussion of results:

- HDPE bottles made from different recycled resin grades and with different recycled content, from 0 to 100%.
- HDPE pellets (virgin) from different suppliers.
- HDPE pellets (recycled) from different suppliers.
- HDPE pellets (recycled) from different batch numbers for the same supplier.

The samples were analysed by use of headspace gas chromatography (detection of volatile substances) and by gas chromatographic analysis (medium and low-volatile substances) after extraction of the recyclate samples. By use of these two methods the most suitable recyclate sources can be qualified. The methods are also suitable for routine control in the bottle manufacturing process. Most of the detected substances are HDPE typical substances like linear oligomers as well as identified post-consumer

substances mostly from previous cosmetic and food fillings. Detection of unknown substance peaks are rare but were found in the investigated samples. The highest concentrations of an unknown substances found in the screening was 19 mg/kg.

Example chromatograms are given in Figure 2 to Figure 4 for bottles made with virgin HDPE (reference), post-consumer rHDPE pellets sample after conventional recycling as well as a post-consumer pellets after additional deep-cleaning. In the virgin HDPE sample (Figure 2) the typical HDPE oligomers were detected as well as some other polymer impurities. The post-consumer rHDPE samples (Figure 3 and 4) contain additional substances peaks, which are not detectable in the virgin HDPE sample. These additional substance peaks are linked to post-consumer substances identified mainly as flavours and cosmetic ingredients from previous fillings. The highest concentration were found for isopropyl myristate (670 mg/kg). In the deep-cleaned rHDPE sample only trace amounts of substance peaks were detectable (Figure 4). The post-consumer as well as the HDPE typical substance were significantly reduced in concentration to levels below the analytical detection limits. Such recyclate samples are most suitable input materials for the cosmetic packaging.

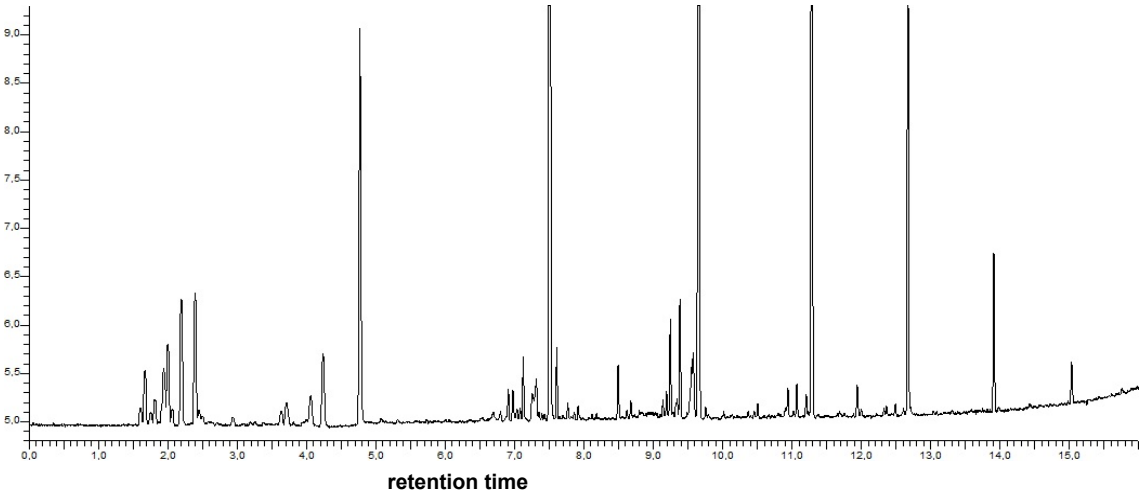

Figure 2: Example headspace gas chromatogram of virgin HDPE bottle

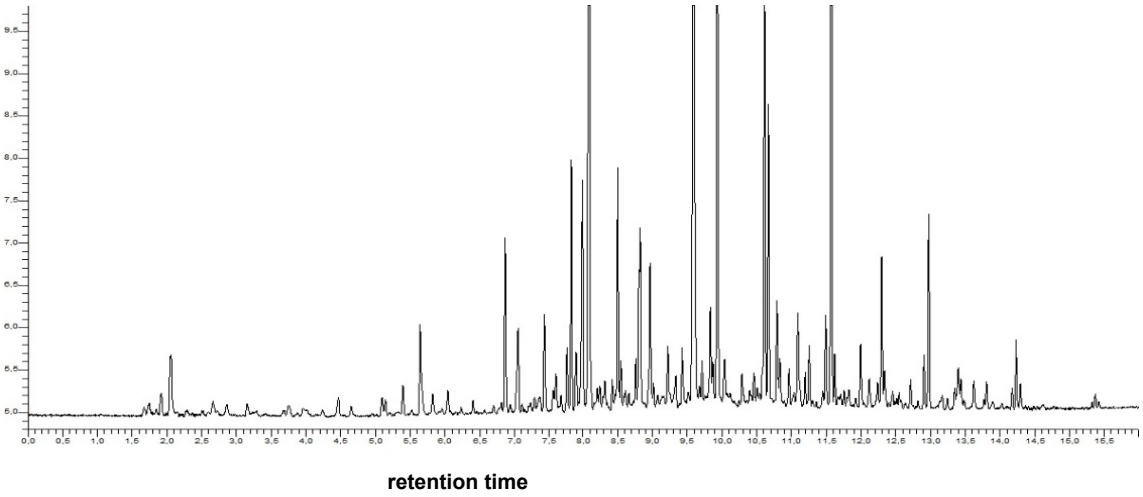

Figure 3: Example headspace gas chromatogram of post-consumer rHDPE from mixed plastic collections

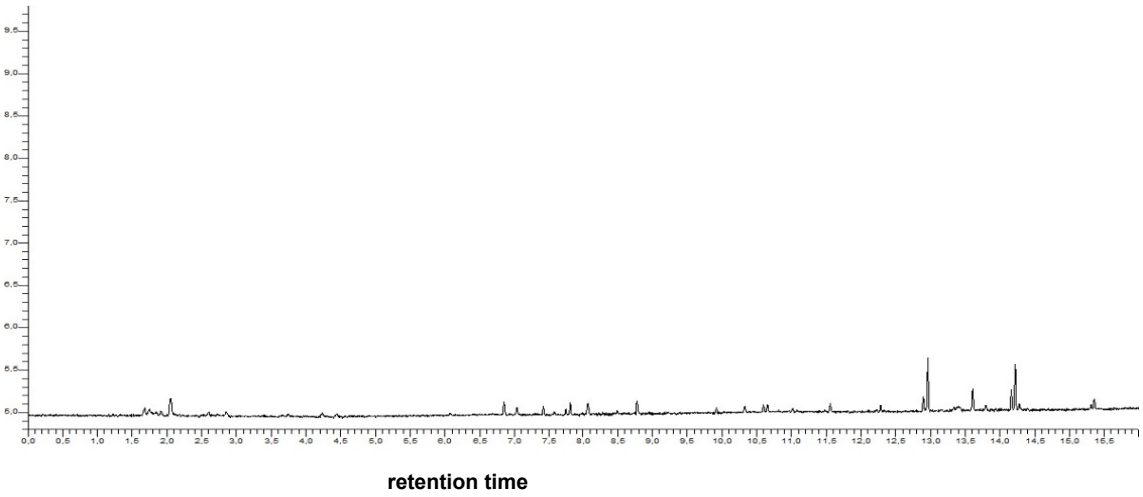

Figure 4: Example headspace gas chromatogram of post-consumer rHDPE from mixed plastic collections after additional deep-cleaning

5.2 Literature Review on Contamination Levels in Post-consumer Polyolefin Recyclates

Comprehensive studies on the contamination levels in recycled polyolefin recyclates are rare in the scientific literature. Only a couple of studies are available, which identified and quantified post-consumer substances in polyolefine recyclates.

In a first study, 21 rHDPE pellets samples of the bottle fraction of household waste collections from five different sources were investigated towards post-consumer substances (Huber and Franz 1997a). 74 substances were identified in the samples in concentrations above 0.5 mg/kg, which are not detected in virgin HDPE. The main substances are saturated fatty acid esters and phthalate esters, as well as hydrocarbons, preservatives, monoterpenes and sesquiterpenes. Most of the substances are identified as constituents from personal hygiene products, cosmetics and cleaning agents. The highest concentrations were found for limonene, diethylhexyl phthalate and the isopropyl esters of myristic and palmitic acid, which are present in

the concentration range of 50 to 200 mg/kg. Many odour compounds and preservatives were determined in concentrations from 0.5 to 10 mg/kg. In a follow-up study seven rHDPE and eight rPP samples were analysed (Huber and Franz 1997b). The main substance detected in the polyolefin samples was limonene in concentrations up to 100 mg/kg. It is interesting to note that the differences in the limonene concentration are going along with the diffusion behaviour of the polymers. In addition to limonene they found phthalates esters, alkanes, 2,6-di-*tert*-butyl-4-hydroxytoluene and oligomers but no hints for misuse of the bottles, e.g. for the storage of toxic chemicals.

A much more comprehensive study had been published in 2005 on the contamination levels of post-consumer rHDPE flakes from milk bottles. The milk bottles were only recollected in the UK (WRAP 2005, Welle 2005). As a result, the predominant contaminants in washed rHDPE flakes were unsaturated oligomers (also found in virgin HDPE pellet samples). The concentrations of both decene and dodecene were around 20 mg/kg, which is a similar concentration range as found in virgin HDPE. Also, small amounts of saturated oligomers like decane and dodecane were found. The concentrations of the saturated oligomers were detected in slightly higher concentrations as found in virgin HDPE. The most predominant post-consumer substance was the flavour compound limonene. As degradation product of antioxidant additives di-*tert*-butylphenol was detected in the post-consumer rHDPE flakes. One sample contained unknown substances at around 130 mg/kg and 40 mg/kg, respectively, which could be caused by the presence of a non-milk HDPE bottle or by misuse. This leads to a mean contamination of post-consumer rHDPE milk bottle flakes of 6500 mg/kg HDPE calculated based on a concentration of 130 mg/kg in 50 flakes as well as under the assumption that two milk bottles out of every 24000 bottles show hints for contaminants from misuse (0.008%). This results in a mean concentration level from misuse of 0.5 mg/kg in rHDPE milk bottle flakes (EFSA 2015) (see Chapter 4.1).

In conclusion, data on the contamination levels of post-consumer recyclates are very rare in the scientific literature and most of the data are more than 15 years old. The most comprehensive study on contamination levels in post-consumer HDPE recyclates was published in 2005 (WRAP 2005) on the contamination levels of rHDPE flakes from milk bottles recollected in the UK. As a consequence, EFSA concluded in their milk bottle opinion that "the monitoring of post-consumer HDPE bottles before recycling, especially misused milk bottles, could provide useful data on the nature/identity of the chemicals involved. This helps to refine the contamination scenario and possibly the set of surrogates used for the challenge test. This analysis should cover potential polar and non-polar contaminants with molecular weights up to 1000 Da using an analytical method of adequate performances at low detection limits" (EFSA 2015).

5.3 Literature Review on Cleaning Efficiencies of Deep-Cleaning Processes for Polyolefins

Cleaning efficiencies of deep-cleaning processes have been published in the scientific literature. The cleaning efficiencies were determined by use of artificial contamination

of washed post-consumer flakes (challenge test). The contaminated flakes were subsequently decontaminated with the investigated recycling process. The difference between the input contamination level and the residual concentrations after decontamination can be expressed as cleaning efficiency. The cleaning efficiencies of the HDPE milk bottle recycling processes are given in Table 1. As a result, the high volatile substances like toluene and chlorobenzene were efficiently removed from the recyclates by use of deep-cleaning processes. Low volatile surrogates like benzophenone, methyl palmitate and methyl stearate are still detectable in significant amounts in the deep-cleaned recyclates. The most challenging contaminants in recycled rHDPE samples are therefore low volatile substances.

For the recycling processes of polypropylene, cleaning efficiencies for deep-cleaning processes are to date not available in the scientific literature.

Table 1: Cleaning efficiencies of HDPE Recycling processes

Surrogate	Cleaning efficiency in %					
	Biffa (EFSA 2015)	CLR (EFSA 2015)	WRAP two steps without washing (Welle 2005)	WRAP three steps without washing (Welle 2005)	WRAP two steps with washing (Welle 2005)	WRAP three steps with washing (Welle 2005)
Toluene	>99.96		99.2	99.8	99.7	99.9
Chlorobenzene	>99.99		99.1	99.8	99.6	99.9
Butyl salicylate	45.28					
Phenyl cyclohexane	71.34	24.5	78.2	94.5	71.0	95.0
Benzophenone	68.95	64.7	11.2	38.1	34.5	54.3
Methyl palmitate	32.23					
Methyl stearate		0	33.7	75.6	>33.7	>75.6

6 Compliance Testing of Virgin HDPE and PP for Cosmetic Applications

Prior to being placed on the market, a cosmetic product must have undergone a safety assessment based on all relevant information. A safety report must be set up in accordance with the Annex I of the Cosmetic Regulation (EC) No 1223/2009.

Guidelines of the European Commission on the Annex I of the Cosmetic Regulation mention the following aspects on packaging materials in direct contact with the formula which must be considered in the risk assessment:

- Interactions between the product and the packaging material, barrier properties of the packaging material and substance migration from / to packaging material.
- Information on the relevant characteristics of packaging material.
- Composition of the packaging material, including technical substances (i.e. additives) and technically unavoidable impurities.

Packaging material means the container (or primary packaging) that is in direct contact with the formulation. The relevant characteristics of packaging materials are important for the safety of the cosmetic product. Reference to the Regulation (EC) No 1935/2004 on Food Contact Materials could be useful (see Chapter 4). Materials that have been developed for food packaging have often already been tested, so relevant information on stability and migration may be available. Additional testing may not be required.

Depending on the nature of materials, it is necessary to check which substances are susceptible to migrate and could have an impact on the finished product safety. Particular attention should be given to substances either prohibited or restricted by the cosmetic regulation. Studies on interactions/suitability between formulation and packaging allow testing of the potential migration of small amounts of substances from the primary packaging material into the product. These tests are performed under specific and relevant test conditions. There are no standard procedures for cosmetic products. An appropriate assessment may be made based on knowledge of the formulation and primary packaging materials and experienced expert judgment.

6.1 Relevant Regulations for Packaging Materials for Cosmetics

All packaging material used for packaging components for cosmetics, independently if they are used for direct or indirect formula contact, must comply with the following Regulations and Directives:

- Regulation (EC) No 1223/2009 of the European Parliament and of the Council of 30 November 2009 on Cosmetic Products.
- Commission Regulation (EC) No 1907/2006 of the European Parliament and of the Council of 18 December 2006 concerning the Registration, Evaluation, Authorization and Restriction of Chemicals (REACH). Dangerous Substances of Annex XVII.
- LFGB, §2, Nr. 2: Bedarfsgegenstände; Abschnitt 5, §30: Verbote zum Schutz der Gesundheit.
- Regulation (EC) 1272/2008 of the European Parliament and of the Council on Classification, Labelling and Packaging of Substances and Mixtures, amending and repealing Directives 67/548/EEC and 1999/45/EC, and amending Regulation (EC) No 1907/2006.
- European Parliament and Council Directive 94/62/EC on Packaging and Packaging Waste.

6.2 Initial Testing for Harmful Substances

Harmful substances are analysed based on migration tests according to Regulation (EU) 10/2011. The following tests are typically applied for the testing of virgin packaging materials:

- Overall migration into 10%, 50% and 95% ethanol, isooctane and 3% acetic acid as simulants
- Phthalate ester (10 d, 60°C) into 95% ethanol as simulant (10 d, 40°C)
- Heavy metals (Pb, Cd, Cr, Hg)
- Specific migration of metals into 3% acetic acid as simulant (10 d, 60°C)
- Specific migration of poly aromatic hydrocarbons (PAH) into isooctane as simulant (10 d, 60°C)
- Specific migration of primary aromatic amines (PAA) into 95% ethanol as simulant (10 d, 60°C)

6.3 Non-Intentional Added Substances (NIAS) Screening and Analysis

In addition to the tests on harmful substances a screening study on non-intentionally added substances (NIAS) according to Regulation (EU) 10/2011 should be done based on the following tests and methods:

- Material screening studies on volatile substances (Headspace GC MS, Thermodesorption TD-GC MS)
- Migration studies on semi-volatile substances: GC-MS: migration with 95% ethanol (10 d, 60°C)
- Migration studies on non-volatile substances: LC-MS: migration with 95% ethanol (10 d, 60°C)

6.4 Toxicological Risk Assessment

The toxicological risk assessment on virgin polymers should be made as follows: Each single identified substance has to be evaluated regarding the relevant toxicological endpoints skin sensitization, mutagenicity and systemic toxicity. The Threshold of Toxicological Concern (TTC) approach can be applied in those cases where no specific data are available. A different exposure scenario for rinse-off and leave-on products has to be considered.

In general, the exposure assessment should be based on the application conditions mentioned in the "The SCCS Notes of Guidance for Testing of Cosmetic Ingredients and their Safety Evaluation (SCCS 2018)" for aggregate exposure of rinse-off and leave-on products. For leave-on products the exposure scenario of sunscreens is, as a worst-case assumption, representative. A dermal penetration rate for the detected substances of 50% should be applied. The following aggregate exposure scenario is used for exposure calculation:

Parameter	Rinse-off	Leave-on
daily product amount	540 mg/d/person	18000 mg/d/person
dermal absorption	50%	50%

Remark: retention factor for rinse-off already included; the sunscreen amount is similar to aggregated amounts for leave-on.

7 Risk Assessment Recycled Packaging Materials for Cosmetics

The safety evaluation of post-consumer HDPE and PP packaging in contact with cosmetic fillings is based on the substance found in the post-consumer polyolefins. There are three types of substances:

- Identified substances from the polymer, which were also found in the reference packaging made from virgin HDPE and PP.
- Identified substances from previous fillings or from cross-contamination during recollection or recycling.
- Non-identified or unknown substances found in the rHDPE and rPP.

For the identified substances a specific toxicological evaluation should be done on each of the substances. Every toxicological endpoint has to be addressed in the expert judgement, considering the exposure scenario of the cosmetic product e.g. a rinse-off or a leave-on application. For by-products coming from the polymer, such an evaluation is most probably already available, because these substances are also found in packaging manufactured from virgin polymers. For other contaminants, not yet evaluated by official expert panels (e.g. EFSA), the Point Of Departure (POD) has to be defined during risk assessment and a safe concentration for each substance should be defined.

Regarding the non-identified substances, a specific toxicological evaluation is not possible. For these substances the evaluation should be done on the Threshold of Toxicological Concern Concept (TTC) (Kroes et al. 2007). As a worst-case assumption the unknown substances should be evaluated as potential genotoxic substances. Even if the substances cannot be identified, the concentration in the recycled polymers can be estimated from analytical tests. However, it is possible that some genotoxic substances are present in the recycled material but in concentrations below the analytical detection limit. In this case, the analytical detection limit should be used for evaluation.

Cosmetic products can be divided regarding their normal and foreseeable use into two principle product types:

- Rinse-off products (like shampoos and shower gels), where it is assumed that only 1% of the applied product amount remains on the skin, which could be absorbed into the body, 99% of the applied product amount is washed off.

- Leave on products (like lotions and creams), where the complete product amount remains on the skin and could be absorbed into the body.

Based on these exposure scenarios a risk assessment of the recycled cosmetic packaging material can be achieved. The following evaluation and examples are used for showing the methodology.

The concentrations of substances and contaminants in post-consumer recycled polyolefins are available from screening tests during recycling or packaging manufacturing. Genotoxic substances should be non-detectable in the recyclate samples within these screening tests. In most cases, an analytical screening of post-consumer substances or other impurities are achieved on post-consumer flakes and pellets. Therefore, analytical data are typically available for input materials for the production of cosmetic packaging materials.

7.1 Risk assessment for Rinse-off Products

The evaluation of post-consumer recyclates in cosmetic applications presented in this document is similar to the evaluation of post-consumer substances in direct food contact, which is evaluated by EFSA (see Chapter 4). This evaluation considers that an unknown contaminant possibly present in post-consumer polymers should be below a threshold of 0.0025 µg per kg b.w. per day. In addition, worst-case assumptions should be applied in the risk assessment of post-consumer recyclates in cosmetic packaging materials. For an adult person a body weight of 60 kg has been taken into account, whereas for an infant a body weight of 8 kg has been assumed (Cosmetic Europe 2015).

As a first worst-case assumption, the concentrations determined in the post-consumer flake or pellet samples (input materials for cosmetic packaging manufacturing) are considered the same as in the final recycled HDPE or PP packaging. This means, that the packaging manufacturing process will not reduce the concentration of post-consumer substances and will not provide a part on the cleaning efficiency of the recycling and packaging manufacturing process. Due to the fact, that any thermal process applied to a post-consumer material reduces the concentration of volatile organic compounds in the material, this assumption can be considered as a worst-case scenario. As a second worst-case assumption, the total amount of all post-consumer substances is migrating from the packaging material into the cosmetic product and is independent from any partitioning between the polymer and the cosmetic product. It is important to note, that using the total migration approach, the risk assessment gets independent from the shelf life and the storage conditions of the cosmetic product. The other parameters, e.g. the packaging wall thickness, the weight of the packaging material or the percentage of post-consumer recyclate in the packaging application should be assumed on realistic or only worse-case assumptions. The risk assessment approach is illustrated by the following examples.

Example 1

A typical application of the post-consumer recyclate is a HDPE bottle for a cosmetic product (shower gel or shampoo) with a bottle volume of 300 ml. The bottle weight is 20 g. Assuming a concentration of a post-consumer substance (e.g. limonene) of 10 mg/kg in the HDPE bottle manufactured with 100% recyclate and assuming a total migration of the substance results in a concentration of 0.667 mg/kg in the shower gel. A typical portion of 18.67 g of shower gel (SCCS 2018) for an adult person once per day used by the consumer for body wash event will then contain 12.5 µg of a post-consumer substance like limonene and much lower for other terpenes. Assuming that 99% of the shower gel is rinsed off with water and 1% of the shower gel constituents will remain on the skin then 0.125 µg of the substance is available for dermal absorption. For an adult person with 60 kg body weight the internal exposure is 0.00207 µg per kg body weight per day. This value is far below the safe threshold of 1.5 µg per kg body weight per day for Cramer Class III substances (Kroes et al. 2007).

However, post-consumer substances might be also below the analytical detection limit of the applied analytical screening methods. Non-detectable substances need a separate consideration of the detection limits. In addition, non-detectable substances are unknown and might be also genotoxic substances. Assuming a detection limit of 1 mg/kg in the post-consumer polymer, under the same conditions as mentioned above, 0.0125 µg is available for dermal absorption. An adult person with 60 kg body weight will have an internal exposure of 0.000207 µg per kg body weight. This value is below the lowest TTC threshold value for genotoxic carcinogens which is 0.0025 µg per kg body weight per day. In conclusion, the so-derived internal exposure of 0.000207 µg per kg body weight per day is a factor of 12 below this lowest TTC threshold. Vice versa, the detection limit should be below of 12 mg/kg.

Example 2

The second example is based in the same bottle weight to filling ratio as used in Example 1 and a detection limit of 1 mg/kg. Based on these assumptions, the maximum concentration in the product is 0.0667 mg/kg. This example is a shampoo for infants with a body weight of 8 kg. The daily shampoo amount for adult is 10.46 g. Using this amount for babies too, as a worst-case assumption, leads to an internal exposure of 0.000872 µg per kg body weight which is still below the threshold of 0.0025 µg per kg body weight per day, but the safety factor is now only 3.

Example 3

The third example uses a small shampoo bottle with 50 ml content and a bottle weight of 6 g. The concentration of an unknown substance is 10 mg/kg in the post-consumer recyclate. This results under the same conditions as for Example 1 to an internal exposure of 0.00373 µg per kg body weight per day for a person with 60 kg body weight. This exposure is above the safe threshold of 0.0025 µg per kg body weight per day. For an infant with 8 kg b.w. the threshold is of course exceeded as well. Therefore,

the use of 100% of this material in a small packaging volume could not be evaluated as safe within this exposure scenario. A recyclate amount of 100% will be not possible from safety perspective for such an application. However, assuming a recyclate content of <67% (shampoo for adults) and <8.9% (infants) will result in an internal exposure below the threshold of 0.0025 µg per kg body weight per day.

7.2 Risk assessment for Leave-on Products

In principle, the risk assessment of leave-on products is very similar to rinse-off products, but as a main difference the whole amount of the cosmetic product remains on the skin and the resorption into the body will be assumed as 100%. This will lower the internal exposure scenario by a factor of 100 compared to the risk assessment approach for rinse-off products given above.

Example 4

A typical application of the post-consumer recyclate is a HDPE bottle for a leave-on product (body lotion) with a bottle volume of 300 ml. The bottle weight is 20 g. Assuming a concentration of post-consumer substances of 10 mg/kg (e.g. limonene) in the HDPE bottle manufactured with 100% recyclate and assuming a total migration of the substance into the body lotion results in a concentration of 0.667 mg/kg. A typical daily portion of 7.82 g of body lotion (SCCS 2018) used by the consumer will then contain 5.21 µg of a post-consumer substance like limonene and much lower for other terpenes. Assuming that 100% of the body lotion will remain on the skin this amount of limonene is available for dermal absorption. For an adult person with 60 kg body weight the internal exposure is 0.0869 µg per kg body weight per day, assuming 100% skin absorption. This value is far below the safe threshold of 1.5 µg per kg body weight per day for Cramer Class III substances.

Assuming genotoxic substances below an analytical detection limit of 1 mg/kg, under the same conditions as mentioned above 0.521 µg is available for dermal absorption. An adult person with 60 kg body weight will have an internal exposure of 0.00869 µg per kg body weight. In conclusion, the so-derived internal exposure of 0.0087 µg per kg body weight per day is a factor of 3 above this lowest TTC threshold of 0.0025 µg per kg body weight per day. In conclusion, if it is not excluded that impurities in the post-consumer recyclates have no mutagenic or carcinogenic potential, the packaging material is not considered as safe for cosmetic leave-on body lotions.

8 Organoleptic evaluation

Sensory properties of the recyclate containing packaging materials are not a critical issue regarding compliance evaluation, but maybe an important point for marketing and consumer acceptance. Therefore, appropriate sensory testing of the cosmetic packaging made from deep-cleaning products is recommended. As the properties

depend very much on the application and on the smell of the finished product, this sensory test should be done by or together with the cosmetic manufacturer.

9 Good Manufacturing Practice and Routine Control

In the same way that for cosmetic packaging with virgin polymers, packaging with recyclates should be manufactured under "Good Manufacturing Practice" standards. A Quality Management (QM) and Quality Assurance System (QAS) should be established as well. In addition, routine control devices for the detection of the contaminant levels in post-consumer recyclates should be established. This is important, because the variation of the contamination levels of post-consumer recyclates might be (significantly) higher as for virgin polymers. Recyclate lots with a too high contamination level, should be detected effectively in an early state of the packaging production and should be able to be sorted out.

The applied methods are similar to the methods used in virgin polymer testing (see Chapter 6.3). The frequency of testing depends on the risk to exceed the maximum levels in the packaging material for the individual applications. A protocol should be defined together with the risk assessment as given in the Examples in Chapter 7.

10 Conclusions

Post-consumer polyolefin recyclate contain substances from the previous filling and include also non-identified substances. Therefore the recyclates should be monitored by used of analytical, non-target screening methods. Headspace gas chromatography as well as extraction followed by gas chromatographic evaluation are suitable and complimentary methods to determine the differences in the input materials. Deep-cleaning methods reduce significantly the concentrations in the post-consumer recyclates and make such recyclates more suitable as input materials for cosmetic packaging.

In general, manufacturers of cosmetic products consider the packaging to be safe if food compliance can be confirmed according to Regulation (EU) No 10/2011 and Regulation (EC) No 1935/2004. However, since this is usually not possible for post-consumer recyclate materials, the company has to prove the safety of these materials for cosmetics by evaluating all toxicological endpoints of any migrating substance. A batch-related control of the post-consumer materials by means of analysis and the evaluation of the substances found, either directly in the material or after migration tests in the respective product, is mandatory. Based on the concentrations found in the materials, a toxicological evaluation can be applied e.g. by evaluating the specific toxicity of any impurity or by applying the threshold limits of the TTC approach.

As a conservative assumption, evaluation of the exposure is based on a total mass transfer of any post-consumer substance from the packaging material into the cosmetic

product. This is a worst-case assumption for any product. On the other hand, by using this assumption, the shelf life of the product does not necessarily have to be taken into account separately. In addition, by use of the total mass transfer approach also the packaging materials and their specific migration behaviour have not been taken into account, which makes the risk assessment less work intensive. However, on the other hand, the safety of a post-consumer recyclate cannot be confirmed in general. The safety assessment must always take into consideration the specific application and packaging geometries of the recyclate containing packaging. As a consequence, safety evaluation of post-consumer polyolefins in cosmetic packaging is a case by case evaluation.

Quality assurance systems and methods for the determination of post-consumer impurities at the recycling and/or on the production side should be installed in order to monitor the contamination levels of the recyclate batches. Recyclate batches with too high levels of contaminants should be sorted out efficiently and should not be used for the production of cosmetic packaging. The concentration of post-consumer substances in recyclates determined in day by day routine testing can be used to assess the exposure of the consumer towards substances from the recyclate.

Based on the examples given in this guidance document the detection limits for the routine test methods can be derived. E.g. for example 1 and 2 in Chapter 7.1 the detection limits should be below of 12 mg/kg and 3 mg/kg, respectively. Non-identified substances should be below of these threshold limits. It's important to note, that a general threshold limit cannot be applied, because the detection or threshold limits depend on the exposure scenario. In addition, the necessary detection limits, however, depend on the recyclate amount in the final packaging, the volume of the packaging and the packaging weight. Therefore, the detection limits necessary for compliance evaluation depends on exposure of the consumers with the specific cosmetic product.

The lack of statistical concentrations of post-consumer substances in different recollection and recycling streams is the major drawback. Due to this data gap, an evaluation with the EFSA approach is hardly possible. This is also concluded by EFSA in the evaluation of the milk bottle recycling processes (see Chapter 7).

The use of analytical detection limits and the non-detectability of genotoxic substances in post-consumer recyclates in routine – batch to batch - analytical screenings overcomes the main issue in the safety evaluation of polyolefin recyclates for cosmetic packaging: the lack of statistical input contamination levels in polyolefin recyclate streams in Europe.

11 Author Contributions

Conceptualization: F.W; methodology: F.W; original draft preparation: F.W.; Chapter Compliance Testing of Virgin HDPE for Cosmetic Applications: E.G; writing, review and editing: M.P., E.G., I.S. and F.W.

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