CONFERENCE 2024
“Towards circularity of PET thermoformed packaging”

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Functional Barrier Updates

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Objectives of the Functional Barrier Project (main)

- Demonstrate the ability of the process to produce materials in compliance with Article 3 of Regulation (EC) No 1935/2004

- Exert monitoring of input and output materials; control and report contamination

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Demonstrate the ability of the process to produce materials in compliance with Article 3 of Regulation (EC) No 1935/2004

Regulation (EU) 2022/1616- Art. 10(3)(c)

The notification by the developer shall […] provide detailed information concerning the following:

[…]

extensive reasoning, and scientific evidence and studies, compiled by the developer, demonstrating that the novel technology can manufacture recycled plastic materials and articles that comply with Article 3 of Regulation (EC) No 1935/2004 […]

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Compliance with Art. 3 of R. (EC) No 1935/2004 ➔ RPET use behind a Functional Barrier

- *'functional barrier' means a barrier consisting of one or more layers of any type of material which ensures that the final material or article complies with Article 3 of Regulation (EC) No 1935/2004 […]* (R. (EU) 10/2011-Art. 3(15))
- For post-consumer PET, EFSA conservatively sets a reference contamination level to 3 mg/kg PET. In this scenario EFSA assumes that all possible contaminants are genotoxic substances
- EFSA set limits of migration at 0.1 µg/kg food for infants, 0.15 µg/kg food for toddlers and 0.75 µg/kg for adults. The toddlers' scenario is usually adopted for RPET applications-when a conservative migration scenario is applied

Scientific Opinion on the criteria to be used for safety evaluation of a mechanical recycling process to produce recycled PET intended to be used for manufacture of materials and articles in contact with food | EFSA (europa.eu)

Proof of migration ➔ Migration modelling

Migration is calculated through Software AKTS 365SML v.6.7

Based on Diffusion Model Equations

The calculated migration depends on

- Food Type (or Food Simulant)
- Temperature and Time of contact with food
- Geometry: thickness; surface/volume
- Molecular weight of the migrating substance
- Polarity of the migrating substance
- Solubility of the migrating substance in food/food simulant (Partition Coefficient)
- The Diffusion Coefficient (rate of "travelling" across the multilayer structure)
- Morphology and density of the material from which the migration takes place (parameters identified as Ap' and τ)

The Model may use different equations, and the parameters for calculation may be set as to result into more ***realistic*** or a migration scenario, whereas the conservative scenario is often applications to EFSA

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Applied to VPET/RPET/VPET trays

Migation will also depend on

- Total thickness: 120 μ; 150 μ; 300 μ; 700 μ; 1400 μ
- Partial thickness: 5/90/5; 7.5/85/7.5; 10/80/10; 15/70/15
- Percent RPET in the inner layer: 100%; 75%; 50%; 30%
- Thermoforming conditions: temperature, time and draw ratio
- Extrusion technology: single screw vs. twin screw, vacuum level

The calculation results in a sequence of steps, that simulate all manufacturing and storage phases, up to the contact with food

The calculation is carried out starting from the concentration of the surrogate contaminants, as resulting after the application of the decontamination efficiency determined by the Challenge Test of the relevant technology

Each calculation consists of five steps, each step considers the actual or conservative conditions at which the contaminants' diffusion takes place

temperature(°C)	time	contact with food	Density	Tau	Ap'	equation	thickness
280	0.33 min	NO	1.2	1577	3.2	realistic PET > 70°C	total
25	180 days	NO	1.375	1577	-1.5	realistic PET <70°C	total
125	10 sec	NO	1.375	1577	3.2	realistic PET > 70°C	total /2.5
25	180 days	NO	1.375	1577	-1.5	realistic PET <70°C	total/2.5
25	365 days						
40	10 days	YES	1.375	1577	3.1	upper bound (worst case) PET <70°C	total/2.5
20	10 days						

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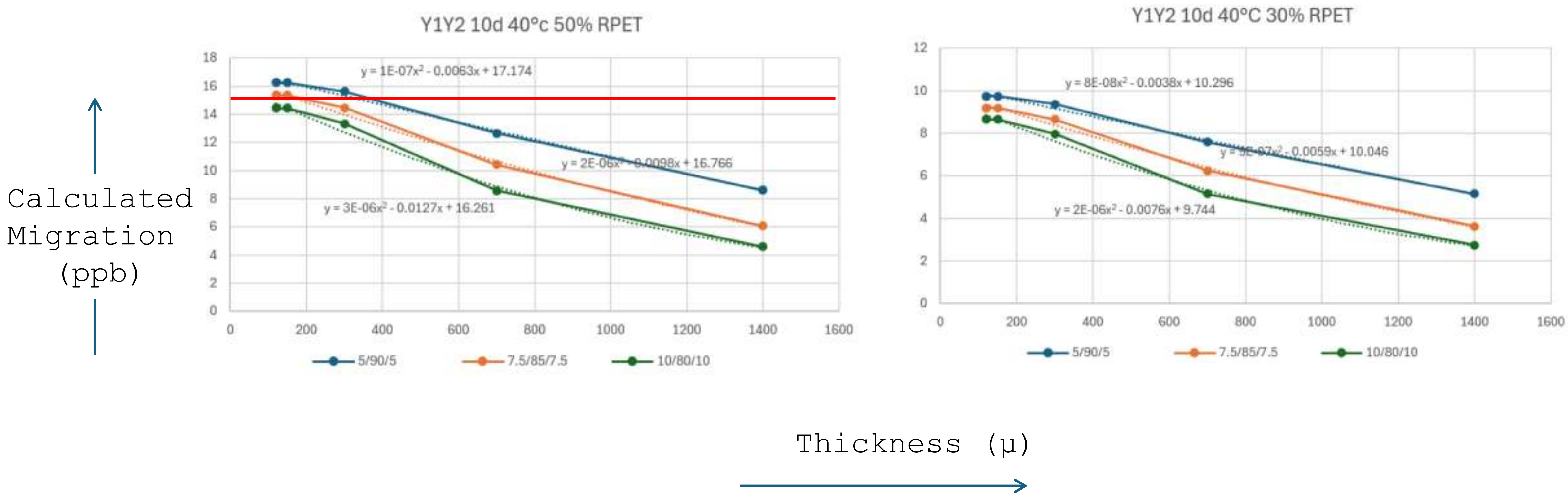
The sequence is repeated for combinations of thickness/structures/conditions/%RPET, leading to a large number of calculations

We are applying an updated version of the software and calculation routine, with enhanced capacity of simulation

We are considering tools to be developed in the software, for automation of the calculation, to speed up the process

Simulation tests	
Total thickenesses	120 um 150 um 300 um 700 um 1400 um
s/v	0.6
Simulation	sequential- see xls
Simulant	D2
Layer structure	5/90/5 7.5/85/7.5 10/80/10 15/70/15
conditions	365D, 25C 10D, 40C 10D, 20C
rPET %	100 75 50 30

Example of outcomes: simulation of migration at 10 days/40°C



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Exert monitoring of input and output materials; control and report contamination

Regulation (EU) 2022/1616- Art. 13(5): monitoring and reporting of the contamination level

The report shall contain at least:[…]

(c) a list of all substances with a molecular weight below 1 000 Dalton found in the plastic inputs to each of the decontamination installations and in the recycled plastic output thereof, sorted in descending order by their relative occurrence and of which at least the first 20 detected incidental contaminants in the input have been identified, and their amounts specified as weight fraction of the input and output; :[…]

[…]

(f) a measurement or estimation of the migration levels to food of contaminants present in the recycled plastic

Monitoring of contamination ➔ NIAS analysis

- 60 samples tested via non-targeted screening analysis
- 12 laboratories across EU
- Three analytical approaches
  - Headspace GC➔ volatile substances
  - GC-MS➔ semi-volatile substances
  - LC- MS (QTOF)➔ non-volatile substances
- Different test methods applied, with potential different outcomes
- 1.000+ substances detected
  - Decreasing with decontamination, or
  - Formed during processing

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Most occurring substances

Table 4: possible origin of substances for technologies X1/X2

substance	possible origin
limonene	food constituent
isophthalic acid	PET constituent
xylenes	degradation product
benzaldehyde	degradation product
toluene	degradation product
styrene	contaminant/degradation product
2,2-bis(4-hydroxyphenyl)propane	contaminant
2-methyl-1,3-dioxolane	degradation product
acetic acid, ethyl ester	degradation product
1,3-ethanediol, monoacetate	
acetone	degradation product
acetic acid	degradation product
tpa-eg oligomers	PET constituent
ethyleneglycol	PET constituent
3,6,13,16-tetraxoatricyclo[16.2.2.2.(8,11)]tetracosa-8,10,18,20,21,23-hexane-2,7,12,17-tetrone	PET constituent
2-[2-hydroxy-3,5-bis(1,1-dimethylbenzyl)phenyl]benzotriazole	contaminant
terephthalic acid	PET constituent
benzene	degradation product
formic acid	degradation product
acetaldehyde	degradation product

Table 5: possible origin of substances for technologies Y1/Y2

substance	possible origin
aibn	plasticizer
limonene	food constituent
xylenes	degradation product
2-pentyl-furan	contaminant
benzaldehyde	degradation product
acetophenone	degradation product
toluene	degradation product
2-methyl-1,3-dioxolane	degradation product
acetic acid, ethyl ester	degradation product
formic acid	degradation product
unknown	
acetic acid	degradation product
2-[2-hydroxy-3,5-bis(1,1-dimethylbenzyl)phenyl]benzotriazole	contaminant
ethyleneglycol	PET constituent
acetone	degradation product
tpa-eg oligomers	PET constituent
pet oligomers	PET constituent
2,2-bis(4-hydroxyphenyl)propane	contaminant
terephthalic acid	PET constituent
hydrocarbon	contaminant
acetaldehyde	degradation product
benzene	degradation product

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Initial comments on NIAS analysis

- Many contaminants are removed to a high extent (>90%), such as limonene, 2-methyl-1,3-dioxolane and others
- Some contaminants are generated in the process, such as acetaldehyde. This is an expected behaviour caused by thermal degradation
- We have a complex pattern of oligomers, which are both removed and generated; they should in principle be regarded as constituents rather than contaminants
- We will focus on selected contaminants which are more critical and representative, and organize a focused check of these substances

Table 2: first 20 most occurring substances- equipment configuration X1and X2 (microgram/kg)

Substance	Average IN (ppb)	Average OUT (ppb)	Average IN Perc 5 < Value < Perc 95 (ppb)	Average OUT Perc 5 < Value < Perc 95 (ppb)	Diff (%)
limonene	1378	89	1323	87	-93%
isophthalic acid	498	72	1041	75	-93%
xylenes	1109	81	954	72	-92%
benzaldehyde	2495	235	1606	254	-84%
toluene	3647	548	1137	407	-64%
styrene	353	142	288	134	-53%
2,2-bis(4-hydroxyphenyl)propane	558	799	758	515	-32%
2-methyl-1,3-dioxolane	3156	2514	2910	2062	-29%
acetic acid, ethyl ester	847	688	821	589	-28%
1,2-ethanediol, monoacetate	520	267	332	269	-19%
acetic acid	6934	41788	6702	6040	-10%
acetone	2559	14680	2098	2370	13%
tpa-eg oligomers	405746	624321	390172	538020	38%
ethyleneglycol	1185	1476	986	1382	40%
pet oligomers	1749586	2833294	1691542	2628518	55%
3,6,13,16-tetraoxatricyclo[16.2.2.2(8,11)]tetracosa-8,10,18,20,21,23-hexaene-2,7,12,17-tetrone	10664	17445	10806	17283	60%
2-[2-hydroxy-3,5-bis(1,1-dimethylbenzyl)phenyl]benzotriazole	8226	60965	9228	14880	61%
terephthalic acid	14819	24334	14117	24088	71%
benzene	809	1568	559	1079	93%
formic acid	4124	13865	3871	7558	95%
acetaldehyde	3753	14031	3470	12094	249%

Lesson learned

- Not all labs have sufficient capabilities for a proper analysis; pro and cons of different approaches
  - Dissolution vs. extraction
  - Temperatures 70-200°C
  - Extraction solvent: DCM, acetonitrile, hexane mixtures
- Decision was taken to carry out a round robin test comparing vPET with a structure containing 100% RPET in the inner layer , to determine lab performances and inter-laboratory repeatibility
- Results obtained recently, at the moment under evaluation

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Next steps

- Completion of the calculation with the updated software
- Conclusions on labs proficiency
- Specific migration of identified selected substances
- Benchmark experimental and simulated levels of migration
- 3rd monitoring report, 10th October 2024

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