

Post-consumer Polystyrene behind a Co-extruded Functional Barrier of Virgin PS

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Description of Novel Technology

This chapter describes briefly the Novel Technology.

The company Fernholz, Meinerzhagen and Schkopau Germany, introduce post-consumer polystyrene (PS) in food packaging. The recyclate is used in PS behind a functional barrier of virgin high impact polystyrene (HIPS) in an ABA sheet structure. The functional barrier is applied in a co-extrusion process. In one alternative the ABA structure is replaced by a ABK structure. Whereas the B layer is containing partially PS from post consumer resources. "A" is a layer of virgin PS applied by coextrusion and "K" ("Kaschierung" or lamination process) is a laminated layer of virgin PS applied as film during the cooling of the sheet in the roll stack (calander) process (after the extrusion die and before winding). The K layer is the non-food contact layer and has a typical thickness of 45 µm. The initial function of the K layer is a decoration item, as the K layer film is printed before lamination.

The ABA sheet has 80% post-consumer recyclate content in the core layer (B layer) with 10% of virgin HIPS as functional barrier on both sides (2x A layers). The overall post-consumer recyclate content is 48%. The overall thickness is typically >0.65 mm. The intended application of the sheet is the production of cups for yoghurt and dessert for storage of maximum 40 days at 8 °C. Other application maybe covered by this initial safety report on demand.

The Fernholz process includes the following process steps:

- step 1: sourcing suitable post-consumer PS material
- step 2a: ABA sheet extrusion with vacuum degassing
- step 2b: ABK sheet extrusion with vacuum degassing and lamination of K layer

Compliance with Article 3 of Regulation (EC) No 1935/2004

Fernholz provided on April 10, 2023, an initial report on the Novel Technology in accordance with Article 10 of Commission Regulation (EU) No. 2022/1616. This report included extensive reasoning, scientific evidence, and studies that demonstrate that the recycled polystyrene behind the Functional Barrier complies with Article 3 of Regulation (EC) No. 1935/2004.

The functional barrier properties of the virgin layer was tested in a worst-case scenario by used of contamination of the core layer (B) similar to a challenge test. Within this challenge test the whole sheet manufacturing process was investigated by use of artificially introduced model substances within a worst-case scenario with initial concentrations in the flake between 390 mg/kg and 1410 mg/kg. The manufactured sheet was further investigated in migration experiments (simulants 10% ethanol and 50% ethanol at 20 °C and 40 °C for 10 days as well as migration kinetics with 360 kinetic points for 10 days at 40 °C.

In conclusion, it was shown that high concentrations of artificially introduced contaminants into the feedstock material lead to a migration of the ABA multilayer sheet far below of 10 µg/kg. This is due to the cleaning efficiency of the extrusion process / manufacturing process as well as due to the functional barrier (ABA structure), which reduces significantly the migration.

List of substances in plastic input and recycled output

Within the last six months three batches of ABA sheets were produced. According to Regulation 2022/1616 input and output samples were tested according to potential contaminants. The samples were analysed by the Fraunhofer IVV in Freising, Germany.

Fraunhofer Test report PA-1823-23 from 27.03.2024 investigates the following samples:

- Sample 1: rPS Granulat, CO2RE 687 DI30 Charge DE08N8I002, 29.08.2023, input

- Sample 2: recyclathaltige ABA Folie, FRE11, Auftrag 23007150, Herstellungsdatum 24.10.2023 08:15, CO2RE 687 DI30 Charge DE08N8I002 43% in der Mittelschicht, Charge Rolle P0005968847 (R136.1.2), output

Fraunhofer Test report PA-1174-24 from 27.03.2024 investigates the following samples:

- Sample 3: rPS Granulat, CO2RE 687 DI30 Charge DE08NAP002, Wareneingang 12-2023, input
- Sample 4: recyclathaltige ABA Folie, internes Kürzel HA7, Auftrag 23008427, Herstellungsdatum 14.12.2023 7:00, CO2RE 687 DI30 Charge DE08NAP002 33% in der Mittelschicht ergibt 23.1% bezogen auf Gesamtfolie, Charge Rolle P0006417603 (R____), output

Fraunhofer Test report PA-1175-24 from 27.03.2024 investigates the following samples:

- Sample 5: rPS Granulat, CO2RE 687 DI30 Charge DE08O1H002, Wareneingang 01-2024, input
- Sample 6: recyclathaltige ABA Folie, internes Kürzel HA7, Auftrag 24000249, Herstellungsdatum 12.02.2024 14:15, CO2RE 687 DI30 Charge DE08NAP002 33% in der Mittelschicht ergibt 23.1% bezogen auf Gesamtfolie, Charge Rolle P0006559944 (R58), output

The results of the identification and semi-quantification are given in Table 1 (Fraunhofer Test report PA-1823-23), Table 2 (Fraunhofer Test report PA-1174-24) and Table 3 (Fraunhofer Test report PA-1175-24) and Table 3. Substances which are also determined in virgin PS are marked with "*" in Table 1 to Table 3.

It is important to note, that all substances above 1 mg/kg were identified and genotoxic compounds were not detected in the input or output material of the Novel Technology.

Table 1: Results of the identification / characterisation and **semi-quantification** of volatile substances in the investigated recycle samples

R _t [min]	Identification	Semi-quantification [mg/kg]	
		Sample 1	Sample 2
1.72	2-methyl-1-propene (isobutene)	<1	22.9
1.82	ethoxy ethene*	<1	14.2
2.06	tert-butanol	<1	2.1
3.11	dimethylfurane	<1	2.3
3.53	1-heptane	<1	1.7
3.89	toluene*	3.9	2.8
4.57	4-ethenylcyclohexene	<1	1.0
5.64	ethylbenzene*	27.7	41.9
5.79	1,3-dimethylbenzene	2.2	3.2
6.08	styrene*	216	270
6.16	2-heptanone	2.2	3.4
6.42	heptanal	<1	1.1
6.56	alpha-pinene	<1	1.6
6.68	isopropylbenzene*	2.4	6.8
6.95	benzaldehyde	<1	3.4
7.11	n-propylbenzene*	1.5	4.3
7.22	2-methyl-1-ethylbenzene	<1	1.6
7.36	1-decene	<1	1.1
7.45	2,2,4,6,6-pentamethylheptane	<1	1.1
7.67	beta-mycrene	<1	3.1
7.84	1-propenylbenzene	<1	1.3
8.00	1-methylpropylbenzene*	<1	1.6
8.21	1-octene-3-one	<1	1.0
13.71	styrene dimer*	/	/
13.90	styrene dimer*	/	/

* substance also detectable in virgin HIPS

Table 2: Results of the identification / characterisation and **semi-quantification** of volatile substances in the investigated recycle samples

R _t [min]	Identification	Semi-quantification [mg/kg]	
		Sample 3	Sample 4
1.72	2-methyl-1-propene (isobutene)	<1	4.7
1.82	ethoxy ethene*	<1	4.8
3.89	toluene*	3.4	2.5
4.53	hexanal	<1	1.0
5.23	1,3-propandiol	<1	1.7
5.64	ethylbenzene*	25.8	46.9
5.79	1,3-dimethylbenzene	2.5	4.2
6.08	styrene*	194	252
6.16	2-heptanone	2.7	4.4
6.68	isopropylbenzene*	3.1	7.8
6.95	benzaldehyde	<1	3.1
7.11	n-propylbenzene*	1.9	5.2
7.22	2-methyl-1-ethylbenzene	<1	1.8
7.67	beta-mycrene	<1	1.3
7.84	1-propenylbenzene	<1	1.2
8.00	1-methylpropylbenzene*	<1	1.6
13.71	styrene dimer*	/	/
13.90	styrene dimer*	/	/

* substance also detectable in virgin HIPS

Table 3: Results of the identification / characterisation and **semi-quantification** of volatile substances in the investigated recycle samples

R _t [min]	Identification	Semi-quantification [mg/kg]	
		Sample 5	Sample 6
1.72	2-methyl-1-propene (isobutene)	<1	2.3
1.82	ethoxy ethene*	<1	4.3
2.62	cyclohexane	<1	2.6
3.89	toluene*	3.2	2.0
4.53	hexanal	<1	1.1
5.23	1,3-propandiol	<1	1.1
5.64	ethylbenzene*	25.2	55.1
5.79	1,3-dimethylbenzene	2.4	4.8
6.08	styrene*	187	222
6.16	2-heptanone	2.7	5.6
6.68	isopropylbenzene*	3.0	10.1
6.95	benzaldehyde	<1	2.6
7.11	n-propylbenzene*	1.9	4.7
7.22	2-methyl-1-ethylbenzene	<1	1.3
13.71	styrene dimer*	/	/
13.90	styrene dimer*	/	/

* substance also detectable in virgin HIPS

List of contaminating materials regularly present in plastic input

The input materials used for the production of the above-mentioned lots were produced with a dissolution process. Foreign materials as well as impurities were filtered. Foreign polymers or other impurities are therefore not detected in the input materials.

Analysis of the most likely origin of the identified contaminants

The results given in Table 1 and Table 2 show, that many of the substances determined in the recycle samples are also found in virgin PS. These substances are generally found at similar concentrations in both virgin and recycled samples. Other substances (e.g., limonene, alpha pinene, ...) are flavouring substances from the first use of the PS packaging. Aldehydes and ketones are most probably oxidation products or degradation products which are most probably generated during extrusion and ABA manufacturing. It should be noted, that these substances might be also generated during analysis of the polymer at high temperatures. In order to reduce this oxidation during analysis the headspace gas chromatograms were made under nitrogen atmosphere.

Estimate of migration levels of contaminants to food

Within the initial safety report, the functional barrier properties of the virgin layer was tested in a worst-case scenario by used of contamination of the core layer (B) similar to a challenge test. Within this challenge test the whole sheet manufacturing process was investigated by use of artificially introduced model substances within a worst-case scenario with initial concentrations in the flake between 390 mg/kg and 1410 mg/kg. The manufactured sheet was further investigated in migration experiments (simulants 10% ethanol and 50% ethanol at 20 °C and 40 °C for 10 days as well as migration kinetics with 360 kinetic points for 10 days at 40 °C. The results of this migration testing showed that the migration of the investigated model substances was below of 10 µg/kg. The concentrations of potential contaminants are far below of the concentrations used for the challenge test. Therefore it can be concluded that migration of the detected contaminants will be far below of 10 µg/kg.

Description of applied sampling strategy

In accordance with Article 13(1) of Commission Regulation (EU) No. 2022/1616, samples from each batch of input and the corresponding output (ABA sheet) were drawn. Within the six months period, two lots were produced and therefore two input/output pairs were analysed using the method described below.

Description of analytical procedures and methods used

Samples were analysed according the accredited Fraunhofer IVV Method 1.334:2021-11

For each test 1.0 g of sample material was weighed into a headspace vial and analysed by headspace GC/FID under nitrogen atmosphere. Gas chromatograph: Perkin Elmer Clarus, column: ZB 1 (length 30 m, inner diameter 0.25 mm, film thickness 0.32 µm), temperature program: 50°C (4 min) to 320°C (15 min) with a heating rate of 20°C/min, pre-pressure: 50 kPa helium, split: 10 ml/min. Headspace Autosampler: Perkin Elmer TurboMatrix, oven temperature: 150 °C, needle temperature: 160 °C, transfer line: 170 °C, equilibration time: 1 h, pressurization time: 3 min, injection time: 0.02 min, withdrawal time: 1 min. Quantification of benzene, ethylbenzene, styrene, limonene and acetophenone was achieved by external calibration.

Identification was achieved by mass spectrometry. Mass spectrometer: Perkin Elmer Clarus GC-MS-System with electric ionization (EI), in full scan mode with mass range m/z 35-300. The identification of the mass spectra was done by comparison with the NIST spectra library (NIST/EPA/NIH Mass Spectral Library 2017). A confirmation of the suggested spectra by analysis of a respective standard was not done.

The analytical methodology was useful in identifying low molecular weight substances (i.e., less than 250 Daltons). Higher molecular weight substances would

not be expected to migrate at any significant level from rPS because it is a low diffusive polymer^[1].

Analysis and explanation of discrepancies

No discrepancies have been observed between the contaminant levels expected in the input and output of the installation and its decontamination efficiency. The data above supports a finding that the decontamination process adequately removes contaminants from the waste stream and the ABA functional barrier reduces sufficiently migration of potential contaminants.

Discussion of differences with previous reports

No differences are observed compared to the previous 6-month report(s) on this technology.

^[1] F. Welle, Diffusion coefficients and activation energies of diffusion of organic molecules in polystyrene below and above glass transition temperature, *Polymers*, 2021, 13(8), 1317. doi:10.3390/polym13081317