1st Six Month Report according to Article 13(4) of the Commission Regulation (EU) 2022/1616

Version 1: 09.04.2024

Recycling of Post-consumer HDPE Closures and Pallets / Crates into new Pallets for Direct Food Contact Application

Developer Craemer GmbH
Brocker Straße 1
33442 Herzebrock-Clarholz
Germany

Relevant for the site in

33442 Herzebrock-Clarholz, Germany

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Description of Novel Technology

This chapter describes briefly the Novel Technology.

The company Craemer, Herzebrock-Clarholz Germany, introduce post-consumer HDPE into new pallets / creates. The process uses HDPE bottle closures and discarded or damaged multi-use HDPE pallets / crates as input material.

The Craemer recycling process for HDPE flakes comprises the following main process steps:

- Step 1a: Grinding of collected post-consumer HDPE closures into flakes followed by a washing step with surfactants and by surface drying (done by flake suppliers).

- Step 1b: Grinding of HDPE pallets / crates into flakes followed by a washing step and by surface drying (mechanical step).
- Step 2: Extrusion of the flakes from steps 1a and 1b to pellets with vacuum degassing, with melt filtration and underwater pelletizing system, followed by surface drying of the pellets.
- Step 3: Manufacturing of palettes from recycled material from step 2.

The Craemer recycling process pallet to pallet with similar process conditions got an approval from the US Food and Drag Administration FDA (PNC 2679, October 21, 2021)

The intended food contact applications are pallets / crates for agricultural products like dry food, whole food, vegetables, fruits and pre-packed meat. The contact conditions are 7 days in maximum at room temperature.

The recycled HDPE is intended to be used for the production of new pallets / crates with up to 100% of recyclate content.

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

Craemer provided on 26.09.2023, an initial report on the Novel Technology in accordance with Article 10 of Commission Regulation (EU) No. 2022/1616. This report includ extensive reasoning, scientific evidence, and studies that demonstrate that the recycled HDPE complies with Article 3 of Regulation (EC) No. 1935/2004.

List of substances in plastic input and recycled output

Within the last six months five batches of recycled HDPE 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-1877-23 from 08.04.2023 investigates the following samples:

- Sample 1: HDPE bottle flakes, different colors, input 27.11.23 9:45
- Sample 1o: HDPE pellets, output 27.11.23 9:45
- Sample 2: HDPE bottle flakes, different colors, input 07.12.23 13:30
- Sample 2o: HDPE pellets, output 07.12.23 13:30
- Sample 3: HDPE bottle flakes, different colors, input 18.12.23 11:45
- Sample 3o: HDPE pellets, output 18.12.23 11:45
- Sample 4: HDPE bottle flakes, different colors, input 20.12.23 12:50
- Sample 4o: HDPE pellets, output 20.12.23 12:50
- Sample 5: HDPE pallet flakes, different colors, input 03.01.24 10:45
- Sample 5o: HDPE pellets, output 03.01.24 10:45

The results of the identification and semi-quantification are given in Table 1 to Table 10. Substances which are also determined in virgin HDPE are marked with "*" in Table 1 to Table 10.

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

R_t [min]	Identification	Semi-quantification [mg/kg]	
		Sample 1	Sample 1o
1.72	too small for clear identification	<5	5.2
1.82	too small for clear identification	<5	6.1
2.08	branched alkane	18.9	8.7
2.14	branched alkane	26.6	
2.22	branched alkane	14.5	
2.38	methyl cyclopentane	13.2	
2.43	branched alkane	<5	5.7
2.68	dimethylbutadiene	<5	79.2
3.32	methyl cyclohexane	23.1	26.1
4.52	3-octene	34.0	32.0
4.72	1-octene	16.3	15.6
4.78	branched alkane	5.2	<5
6.96	terpene	5.7	<5
7.89	decane*	10.9	9.9
8.05	cumene	5.5	<5
8.17	limonene	155.9	91.4
8.49	terpene	12.8	<5
9.99	dodecane*	15.0	12.9
11.63	tetradecane*	**	**
13.04	hexadecane*	**	**
14.29	octadecane*	**	**

* substance also detectable in virgin HDPE, ** quantified based on the extracts, see below

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

R_t [min]	Identification	Semi-quantification [mg/kg]	
		Sample 2	Sample 2o
1.72	too small for clear identification	<5	10.0
1.82	too small for clear identification	<5	9.6
2.07	branched alkane	12.6	15.8
2.14	branched alkane	16.9	<5
2.22	branched alkane	12.2	<5
2.38	methyl cyclopentane	8.2	<5
2.43	branched alkane	<5	11.6
2.68	dimethylbutadiene	9.3	36.7
3.32	methyl cyclohexane	9.2	6.6
4.31	branched alkane	8.3	5.3
4.76	1-octene	14.3	12.5
5.30	branched alkane	6.6	5.6
6.96	terpene	9.5	<5
7.88	decane*	14.6	16.7
8.05	cumene	<5	7.0
8.17	limonene	199	160
9.99	dodecane*	18.4	19.1
11.63	tetradecane*	**	**
13.04	hexadecane*	**	**
14.29	octadecane*	**	**

* substance also detectable in virgin HDPE, ** quantified based on the extracts, see below

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

R_t [min]	Identification	Semi-quantification [mg/kg]	
		Sample 3	Sample 3o
1.72	too small for clear identification	<5	18.5
1.82	too small for clear identification	<5	10.4
1.89	too small for clear identification	<5	6.1
2.08	branched alkane	14.4	9.9
2.14	branched alkane	17.9	<5
2.22	branched alkane	9.0	<5
2.38	methyl cyclopentane	8.0	<5
2.43	branched alkane	<5	6.6
2.68	dimethylbutadiene	<5	10.0
3.32	methyl cyclohexane	<5	22.8
4.52	3-octene	11.9	21.3
4.72	1-octene	6.0	10.2
4.78	branched alkane	<5	6.0
7.89	decane*	12.6	15.4
8.17	limonene	143.0	110.8
8.49	terpene	10.6	6.7
9.99	dodecane*	18.4	17.3
11.63	tetradecane*	**	**
13.04	hexadecane*	**	**
14.29	octadecane*	**	**

* substance also detectable in virgin HDPE, ** quantified based on the extracts, see below

Table 4: Results of the identification / characterisation and **semi-quantification** of volatile substances in the investigated recyclate samples

R_t [min]	Identification	Semi-quantification [mg/kg]	
		Sample 4	Sample 4o
1.72	too small for clear identification	<5	11.4
1.82	too small for clear identification	11.2	6.4
2.08	branched alkane	16.9	7.9
2.14	branched alkane	14.8	<5
2.22	branched alkane	11.2	<5
2.38	methyl cyclopentane	9.3	<5
2.43	branched alkane	<5	5.5
2.68	dimethylbutadiene	<5	7.0
3.32	methyl cyclohexane	<5	19.2
3.86	toluene	5.2	<5
4.52	3-octene	<5	19.5
4.72	1-octene	<5	8.9
5.19	branched alkane	9.0	<5
5.49	branched alkane	5.9	<5
7.89	decane*	11.0	11.4
8.05	cumene	11.5	5.8
8.17	limonene	228.1	199.2
8.49	terpene	10.9	9.8
8.82	terpene	5.3	<5
9.99	dodecane*	19.5	17.3
11.63	tetradecane*	**	**
13.04	hexadecane*	**	**
14.29	octadecane*	**	**

* substance also detectable in virgin HDPE, ** quantified based on the extracts, see below

Table 5: Results of the identification / characterisation and **semi-quantification** of volatile substances in the investigated recyclate samples

R_t [min]	Identification	Semi-quantification [mg/kg]	
		Sample 5	Sample 5o
1.72	too small for clear identification	<5	39.7
1.82	too small for clear identification	<5	11.3
2.08	branched alkane	22.7	<5
2.14	branched alkane	20.2	<5
2.22	branched alkane	17.8	<5
2.38	methyl cyclopentane	7.5	<5
6.96	terpene	<5	14.8
7.89	decane*	11.7	8.8
8.49	terpene	5.1	<5
9.25	branched alkane	<5	5.4
9.99	dodecane*	10.4	7.8
11.63	tetradecane*	**	**
13.04	hexadecane*	**	**
14.29	octadecane*	**	**

* substance also detectable in virgin HDPE, ** quantified based on the extracts, see below

Table 6: Results of the identification / characterisation and **semi-quantification** of medium and non-volatile substance substances in the investigated recyclate samples

Peak	Identification	Concentration [mg/kg]	
		Sample 1	Sample 1o
1	Limonen	134 ±12	93 ±1
2	n-Alkane C14*	105 ±17	65 ±3
3	n-Alkane C16*	233 ±24	148 ±2
4	n-Alkane C18*	290 ±12	187 ±6
5	n-Alkane C20*	303 ±4	192 ±1
6	n-Alkane C22*	285 ±5	171 ±3
7	n-Alkane C24*	235 ±8	129 ±5
8	Monopalmitine CAS 234700-00-0	15 ±2	57 ±7
9	n-Alkane C26*	39 ±27	49 ±2
10	Monostearone CAS 123-94-4	173 ±6	90 ±4
11	Erucamide* CAS 112-84-5	1 ±1	31 ±4
12	Behenamide* CAS 3061-75-4	463 / 190	355 ±10
13	n-Alkane C28*	489 / 310	200 ±2
14	n-Alkane C30*	152 ±6	79 ±2
15	n-Alkane C32*	83 ±4	42 ±1
16	Irgafos 168* CAS 31570-04-4	52 ±1	30 ±3
17	oxid. Irgafos 168* and traces of Irganox 1076* CAS 2082-79-3	360 ±53	346 ±7
18	not identified	261 ±10	410 ±5

* substance also detectable in virgin HDPE

Table 7: Results of the identification / characterisation and **semi-quantification** of medium and non-volatile substance substances in the investigated recyclate samples

Peak	Identification	Concentration [mg/kg]	
		Sample 2	Sample 2o
1	Limonen	177 ±11	156 ±2
2	n-Alkane C14*	86 ±9	77 ±5
3	n-Alkane C16*	182 ±20	152 ±2
4	n-Alkane C18*	210 ±27	178 ±2
5	n-Alkane C20*	213 ±32	175 ±1
6	n-Alkane C22*	205 ±37	145 ±1
7	n-Alkane C24*	173 ±32	99 ±2
8	Monopalmitine CAS 234700-00-0	30 ±5	39 ±4
9	n-Alkane C26*	63 ±19	53 ±8
10	Monostearone CAS 123-94-4	132 ±31	66 ±1
11	Erucamide* CAS 112-84-5	4 ±2	17 ±3
12	Behenamide* CAS 3061-75-4	610 ±7	388 ±20
13	n-Alkane C28*	244 ±52	161 ±22
14	n-Alkane C30*	122 ±24	58 ±9
15	n-Alkane C32*	70 ±18	34 ±1
16	Irgafos 168* CAS 31570-04-4	50 ±14	27 ±4
17	oxid. Irgafos 168* and traces of Irganox 1076* CAS 2082-79-3	356 ±94	265 ±27
18	not identified	304 ±33	381 ±6

* substance also detectable in virgin HDPE

Table 8: Results of the identification / characterisation and **semi-quantification** of medium and non-volatile substance substances in the investigated recyclate samples

Peak	Identification	Concentration [mg/kg]	
		Sample 3	Sample 3o
1	Limonen	139 / 360	108 ±1
2	n-Alkane C14*	99 ±7	84 ±2
3	n-Alkane C16*	213 ±45	168 ±2
4	n-Alkane C18*	262 ±81	196 ±2
5	n-Alkane C20*	360 / 180	188 ±3
6	n-Alkane C22*	141 / 165	160 ±3
7	n-Alkane C24*	282 / 134	114 ±4
8	Monopalmitine CAS 234700-00-0	8600/ 18	28 ±1
9	n-Alkane C26*	57 / 88	76 ±2
10	Monostearone CAS 123-94-4	213 / 98	76 ±3
11	Erucamide* CAS 112-84-5	9855 / 1	12 ±1
12	Behenamide* CAS 3061-75-4	209 ±6	298 ±2
13	n-Alkane C28*	249 ±70	211 ±2
14	n-Alkane C30*	122 ±37	71 ±1
15	n-Alkane C32*	76 ±33	27 ±14
16	Irgafos 168* CAS 31570-04-4	48 ±19	21 ±1
17	oxid. Irgafos 168* and traces of Irganox 1076* CAS 2082-79-3	230 ±8	181 ±3
18	not identified	217 ±5	426 ±4

* substance also detectable in virgin HDPE

Table 9: Results of the identification / characterisation and **semi-quantification** of medium and non-volatile substance substances in the investigated recyclate samples

Peak	Identification	Concentration [mg/kg]	
		Sample 4	Sample 4o
1	Limonen	189 ±4	198 ±1
2	n-Alkane C14*	113 ±2	94 ±4
3	n-Alkane C16*	222 ±13	189 ±2
4	n-Alkane C18*	260 ±28	224 ±1
5	n-Alkane C20*	261 ±32	223 ±1
6	n-Alkane C22*	245 ±34	193 ±2
7	n-Alkane C24*	200 ±30	141 ±2
8	Monopalmitine CAS 234700-00-0	21 ±1	26 ±2
9	n-Alkane C26*	106 ±51	72 ±1
10	Monostearone CAS 123-94-4	149 ±26	95 ±1
11	Erucamide* CAS 112-84-5	3 ±2	11 ±2
12	Behenamide* CAS 3061-75-4	429 ±96	449 ±8
13	n-Alkane C28*	281 ±62	196 ±15
14	n-Alkane C30*	134 ±21	69 ±12
15	n-Alkane C32*	72 ±14	41 ±3
16	Irgafos 168* CAS 31570-04-4	47 ±8	28 ±2
17	oxid. Irgafos 168* and traces of Irganox 1076* CAS 2082-79-3	322 ±36	199 ±2
18	not identified	222 ±1	484 ±6

* substance also detectable in virgin HDPE

Table 10: Results of the identification / characterisation and **semi-quantification** of medium and non-volatile substance substances in the investigated recyclate samples

Peak	Identification	Concentration [mg/kg]	
		Sample 5	Sample 5o
1	n-Alkane C10*	6 ±1	5 ±1
2	1-Alkene C12*	7 ±1	8 ±2
3	n-Alkane C12*	19 ±2	18 ±1
4	1-Alkene C14*	18 ±4	12 ±1
5	n-Alkane C14*	29 ±3	26 ±1
6	2,4-Di-tert-butylphenol CAS 96-76-4	6 ±4	7 ±1
7	1-Alkene C16*	33 ±12	20 ±1
8	n-Alkane C16*	35 ±8	35 ±2
9	1-Alkene C18*	67 / 32	27 ±1
10	n-Alkane C18*	37 ±10	32 ±1
11	1-Alkene C20*	57 ±28	27 ±1
12	n-Alkane C20*	32 ±13	25 ±1
13	1-Alkene C22*	57 ±33	24 ±1
14	n-Alkane C22*	22 ±10	15 ±1
15	1-Alkene C24*	51 ±32	20 ±2
16	n-Alkane C24*	17 ±8	10 ±1
17	2,4-bis(1-methyl-1-phenylethyl)-phenol CAS 2772-45-4	1 ±1	134 ±18
18	1-Alkene C26*	40 ±27	16 ±2
19	n-Alkane C26*	11 ±5	10 ±2
20	not identified	<1	<1
21	not identified	34 ±26	10 ±1
22	1-Alkene C28*	<1	95 ±28
23	n-Alkane C28*	5 ±1	6 ±1
24	Irgafos 168* CAS 31570-04-4	242 / 8	73 ±2
25	oxid. Irgafos 168* and traces of Irganox 1076* CAS 2082-79-3	516 ±3	401 ±5
26	not identified	32 ±3	27 ±1

* substance also detectable in virgin HDPE

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 extrusion 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 to Table 10 show that many of the substances determined in the recyclate samples are also found in virgin HDPE. These substances are generally found at similar concentrations in both virgin and recycled samples. Other substances (e.g., limonene, terpenes, branched alkanes, …) are oligomers or flavouring substances from the first use of the HDPE input.

Estimate of migration levels of contaminants to food

Within the initial safety report, the exposure to consumers was evaluated based on migration modelling. Following this calculations, the maximum migration into food in 12.5 µg/kg, which is very low. Assuming a toddler (10 kg b.w.) and a consumption of 100 g agricultural products per day, will result in an exposure of 0.125 µg per kg body weight per day.

It should be noted, that typically foodstuffs that are stored on the pallet are in most cases already packed in primary packaging and have no direct contact to the pallets / crates. Therefore, the low amount of migration is considered as critical. Only vegetables or fruits might be stored without primary packing on the pallets / crates. However, these agricultural products are washed and often peeled before they are consumed. This washing and peeling reduces or eliminates the contaminants from the agricultural products.

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 were drawn. Within the six months period, five lots were produced and therefore five input/output pairs were analysed using the method described below.

<u>Screening for volatile substances</u>

Based on the accredited Fraunhofer IVV Method 1.334:2021-11 (quantification not accredited)

For each test 1.0 g of sample material was weighed into a headspace vial and analysed by headspace GC/FID under normal atmosphere. Gas chromatograph: 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. Headspace Autosampler: oven temperature: 120 °C, equilibration time: 1 h. Quantification of limonene was achieved by external calibration.

Identification was achieved by mass spectrometry. Mass spectrometer 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.

<u>Screening for medium and low volatile substances</u>

Based on the accredited Fraunhofer IVV method 1.337:2018-02

1.0 g of sample material was extracted with 10 ml of dichloromethane and stored for 3 days at 60 °C. An internal standard of butylated hydroxyanisole (BHA) und Tinuvin 234 was added to an aliquot of the extracts, and analysed by gas chromatography with flame ionisation detection (GC-FID) for semi-volatile compounds. Gas chromatograph: capillary column DB-1 (length 30 m, inner diameter 0.25 mm, film thickness 0.25 µm), temperature programme: 50 °C (2 min up to 340 °C (10 min) with a heating rate of 10 °C/min. Peaks of interest were semi-quantified using the internal standard BHA. This method is valid for organic components with a molecular weight of approximately 150 to 700 g/mol.

The identification of the main compounds was done by GC analysis coupled with mass spectrometry. GC/MS-System: column: Optima-5-MS (length 30 m, inner diameter 0.25 mm, film thickness 0.25 µm), temperature programme: 50 °C (2 min up to 340 °C (10 min) with a heating rate of 10 °C/min. Full scan mode, mass range m/z 40 - 800. The identification of the 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.

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.

Discussion of differences with previous reports

This is the first 6-month report on this technology, and thus, no differences are observed.