Fraunhofer IVV

Initial Report on the Novel Technology

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

Petitioner:

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Technology:
Craemer plastic pallet recycling

Contains Confidential Information

This technical dossier contains information about process steps and process parameters. The process parameters are fundamental to decontamination efficiency. The process parameters form part of the intellectual property of the technology manufacturer of the recycling process. Therefore the process parameters should be kept confidential. Confidential information is marked in red.

Characterisation of the Super-clean Recycling Technology

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 at temperatures of [redacted] (residence time [redacted]) with surfactants followed by surface drying (done by flake suppliers).
- Step 1b: Grinding of HDPE pallets / crates into flakes followed by a washing at temperatures of [redacted] (residence time [redacted]) with surfactants followed by surface drying (mechanical step).
- Step 2: Extrusion of the flakes from steps 1a and 1b to pellets at an extruder temperature of approx. [redacted] with vacuum degassing [redacted], with melt filtration [redacted] and underwater pelletizing system, followed by surface drying of the pellets at [redacted] for approx. [redacted]
- Step 3: Manufacturing of palettes from recycled material from step 2 (temperature [redacted]) for [redacted]

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)

Description of the Super-clean Recycling Technology (Step 2)

The extruder design allows to control the following parameters, which are important to guaranty the decontamination efficiency:

- Temperature
- Vacuum
- Residence time

A flow chart of the investigated super-clean recycling process is shown in Figure 1.

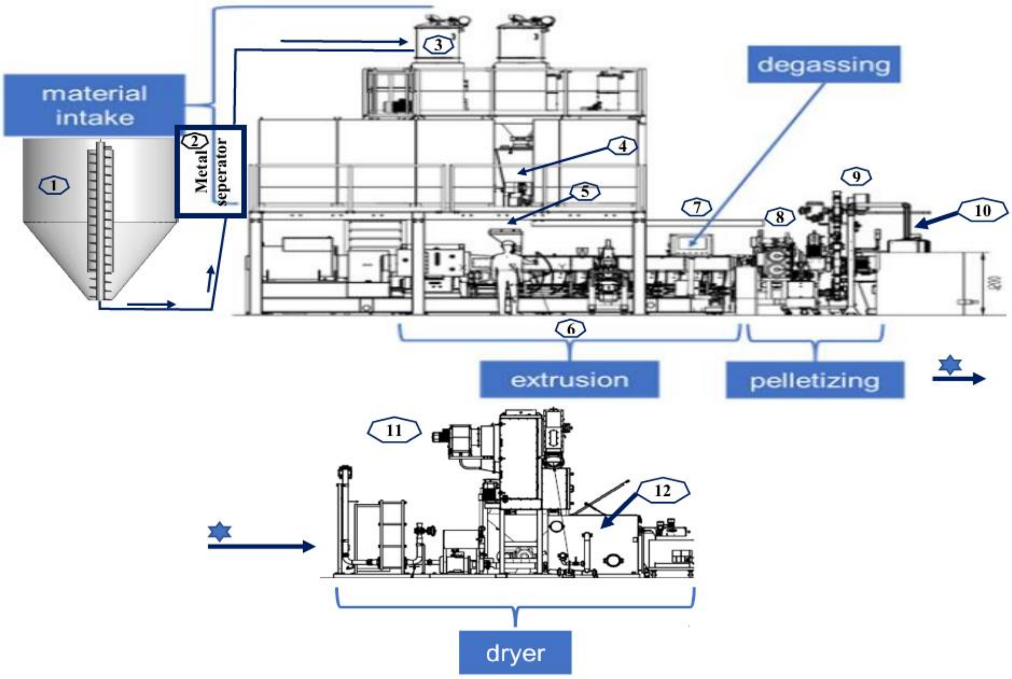

Figure 1: Flow chart of the investigated recycling process (non-confidential)

<u>Material Intake</u>

(1) To prevent colour fluctuations in the final product, the material is stored in a mixing silo with a capacity of [redacted] Here, a "volcano effect" is created with the help of a dosing screw, whereby the material is repeatedly transported from the bottom to the top and thus mixed.

(2) Before the material enters gravimetric dosing (3), the material is sent through a metal detector to sort our metals from the material.

(4) The material is conveyed with vacuum into the corresponding collection containers. After the vacuum is switched off, the material is fed into the designated dosing units. The metering unit is only filled again when the fill is [redacted]

(5) Filler funnel for the preset the mixture.

<u>Extrusion</u>

(6 and 7) The polymer to be processed is conveyed longitudinally through a heatable barrel in the extruder by means of rotating screw shafts and homogenised. With barrel temperature of [redacted], a melt temperature of [redacted] and a screw speed of about [redacted], the melted material reaches step (6). The throughput of the extruder is [redacted].

<u>Degassing</u>

(8) Degassing takes place in three degassing steps. The first atmospheric degassing takes place via a vacuum of [redacted], whereby the moisture is degassed in the first step. The second degassing takes place via a side degassing, which degasses at a

speed of [redacted]. In the third step, degassing is carried out with a vertical degasser at [redacted].

Pelletizing

(9) With the help of [redacted], the material is filtered with [redacted], whereby the screen changers reach a temperature of [redacted]. The material is then filtered with the help of two pressure sensors, which are located before and after the filtering process. A differential pressure is generated by two pressure sensors, which are located before and after the filtering process. At a maximum differential pressure of [redacted], a backwash is automatically requested. This rinses the screens and keeps the melt pressure constant at [redacted]. After approx. [redacted] backwash cycles, a screen change is carried out. The machine switches from four to three sieves so that one can be changed. The previously melted material is pressed through a perforated plate with [redacted]. Here, the perforated plate and the start-up diverter have temperature [redacted].

Dryer

(10) After the material has been cut to the correct grain size with the help of a granulator with six knife edges, the material is cooled and conveyed with an underwater pelletiser. In the underwater pelletiser, a circuit with a volume of about [redacted] of water, which is pumped into the circuit at about [redacted], runs continuously. The water temperature (cooling water) here is about [redacted].

(11) In order to remove the surface moisture from the material, the material enters the dryer in the next step, where the moisture is removed with a centrifugal movement of [redacted]. The time between the "granulation step" and the exit of the compound from the dryer is approx. [redacted].

(12) Water filters are used keep the water flow for underwater pelletising clean. These are to filter the water from microplastics, sand and any solid contaminants.

Manufacturing of Pallets (step 3)

The pallets are produced on a standard injection moulding machine. The material is processes at a temperature of [redacted] for [redacted]. During this process a possible microbiological contamination is removed.

Input Material Characterisation

The Craemer recycling process uses the following input materials:

- re-collected post-consumer HDPE closures from PET beverage bottles.
- multi-use pallets / crates used for the transport and storage of foodstuff, like meat, fruits or vegetables. The pallets / crates typically were used in a multi-use system for several times, approximately 20 to 50 cycles.

The materials from pallets / crates are at the end of their service life due to mechanical damages.

All input materials (closures, pallets / crates) were previously used for food contact and are in compliance with EU Regulation 10/2011. This is confirmed by the input material suppliers.

Input closure materials: The input material originates from PET bottle collections systems such as curbside and deposit collections. The PET bottles were sorted before recycling. This sorting step guaranties that only (>99%) PET beverage bottles were introduced into the recycling process. Therefore, the HDPE closures from these PET bottles were in contact with food in the first application. This input material is therefore in compliance with EU Regulation 10/2011 and amendments.

The PET bottles are grinded to so-called flakes together with the PET bottles. The HDPE closure flakes are separated from the PET flake stream by use of a swim/sink separation step. Subsequently the HDPE flakes are washed at temperatures of [redacted] with surfactants. During this washing step dust on the surface of the HDPE flake were removed. The overall residence time of the HDPE flakes in the washing line is [redacted]. Subsequently the flakes are mechanically dried in order to remove moisture on the surface of the flakes. This step is done by flake suppliers.

Input material (HDPE closure flakes) is only acceptable from certified suppliers with valid documentation complying with EU regulations. Delivered material are inspected on arrival upon integrity of packaging and visual appearance. From each lot a sample is drawn and stored for 6 months after material processing for tracing supplier. Internal test procedures at the processing company are applied regularly for achieving reliable quality assurance.

Input pallets / crates: The pallets / crates are coming from known and controlled closed-loop systems. The crates are originally used as multi-use agricultural transport or storage crates for meat, vegetables, fruits etc. which are sorted out due to scuffing and/or other damages. As far as we know, all multi-use pallets / crates originally are manufactured from food grade material as used also for food packaging purposes and are therefore in compliance with EU Regulation 10/2011 and amendments.

All pallets / crates are visually inspected before recycling. The pallets / crates are therefore not visibly contaminated. In addition only pallets / crates from Craemer with glass and fork sign are used as input material for the recycling process. That means the pallets / crates to be recycled were food contact pallets / crates in the first use. In addition the pallets / crates have been originally produced and are used by exactly specified and controlled pooling systems. This means that food pallets / crates (with glass and fork sign) are only used for food transport. From the controlled pooling systems it is excluded, that the pallets / crates are used for non-food transport.

The HDPE pallets / crates flakes were washed at temperatures of [redacted] with surfactants are used. During this step dust on the surface of the HDPE flake were removed. The overall residence time of the HDPE flakes in the washing line is [redacted]. Subsequently the flakes are mechanically dried in order to remove moisture on the surface of the flakes.

Incoming Inspection of Input Material (closure material, pallets / crates) – quality assurance:

1. General visual inspection for obvious contamination.
2. Measuring the Melt Flow Index (MFI).

3. Measuring the Moisture.
4. Analyze the material with a flake scanning method for material quality and quantity of foreign (solid) particles (like metal, stones, dust or wood).

Intended Use of the Recycled Material

Typical 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 1935/2004

Characterisation of Input Contamination Levels

Input HDPE flakes from closures

According to the scientific opinion on HDPE milk bottles[1] EFSA has defined the maximum contamination level in post-consumer HDPE recyclates to 0.5 mg/kg. The input material of the actual petition, however, is closures from PET bottles. For the following reasons, we concluded, that an input contamination of 0.5 mg/kg also is applicable in the evaluation of post-consumer closures from PET bottles.

The post-consumer HDPE milk bottles were collected from curbside and bring scheme collections in UK. The composition of the input material was only 20% of HDPE milk bottes. The other materials were PET bottles (50%), other HDPE bottles (20%), PS, PP, other plastic bottles (6%) and residue (4%). The maximum contamination level was found in the milk bottle fraction from the above mentioned input after washing was determined to 0.5 mg/kg, which was used by EFSA as worst-case input contamination level for the evaluation of milk bottle recycling[1].

The input material of this Novel Technology originates from PET bottles from deposit systems as well from curbside collections. The closures are separated from the PET bottles in the washing process. Therefore it can be concluded, that only closures are entering the process, which were on PET bottles. The input material (washed closure flakes) is therefore much more controlled as the milk bottle collection in UK. Therefore the input contamination level should be lower as 0.5 mg/kg as found for the UK milk bottles.

Regarding the incidence of misuse of PET bottles (with closures) for storage of household chemicals, it was found within the Recyclability Project that the incidence

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[1]Scientific Opinion on the safety assessment of the processes ‘Biffa Polymers’ and ‘CLRrHDPE’ used to recycle high-density polyethylene bottles for use as food contact material, EFSA Journal 2015;13(2):4016 (25 pages)

of misuse of PET bottles in Europe is 0.03-0.04%[2][. This means, that 3-4 closures from 10000 closures might be contaminated with chemicals from misuse. Assuming a closure weight of 3 g, 9-12 g of contaminated HDPE flakes might be found in 30000 g HDPE flakes (or 300-400 g of contaminated flakes in 1 t). Assuming a concentration of 1000 µg/g (ppm) in the contaminated flake, the absolute amount of 300-400 mg chemicals from misuse can be found in 1 t of HDPE closure flakes, which is 0.3-0.4 mg/kg (ppm) in washed flakes from closures. This value is from a pure statistical point of view and is in good agreement with the 0.5 mg/kg of the HDPE milk bottles.

It should be noted here, that the input contamination of 0.5 mg/kg used for the calculation of c_{res} includes exclusively genotoxic compounds. However, it is most unlikely, that the input material from post-consumer PET bottle closures contains a homogenous contamination of 0.5 mg/kg of genotoxic compounds. The contamination of genotoxic compounds of 0.5 mg/kg in the input stream must be, if at all, much lower. This assumption is supported by the fact that the consumer has no genotoxic compounds, which can be brought into contact with the PET bottles and HDPE closures. Therefore, the genotoxic compounds have to be built during recycling, for example as degradation products from the polymer or from polymer additives. The screening of the washed flake sample gave no hints that degradation occurs in concentrations of 0.5 mg/kg. In addition, also in the challenge test samples no hints for degradation are given even if the challenge test samples are artificially contaminated with high amounts of chemicals.

<u>Input pallets / crates from controlled closed loop systems</u>

The pallets / crates are coming from known and controlled closed loop systems. The pallets / crates are originally used as multi-use agricultural transport or storage crates for meat, vegetables, fruits etc. which are sorted out due to scuffing and/or other damages. These input material are defined pallets / crates from reliable manufacturers, which are approved and certified (e.g. acc. ISO 9001:2015). The crates are controlled by identification of the types.

All HDPE crates originally are manufactured from food grade material as used also for food packaging purposes and are therefore, before first use, in compliance with EU Regulation 10/2011 and amendments. The pallets / crates have the glass and fork sign.

The pallets / crates are visually inspected before grinding. Pallets / crates which are contaminated (with e.g. oil, paints, etc.) are not introduced into the recycling process. In addition, the multi-use pallets / crates are cleaned by the pooling provider after each turn, so that potential contaminants have only short time contact to the multi-use pallets / crates.

Potential contaminants of pallets / crates before recycling are:

- stones, dust etc.
- bio-residues (from the vegetables and fruits)
- microbiological contamination (rotten foodstuffs, remains from fungi)
- chemical contamination

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[2]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 Journal 2011;9(7):2184 (25 pages)

Stones, dust, dirt, etc. are only surface contaminants with are removed from the pallets / crates completely during the washing process. According to a statistical evaluation at the recycling plant, the incidence of extraordinary stone, dust and dirt contamination is one pallets / crates of about 100 pallets / crates, when coming back in the pooling loop. Subsequently the pallets / crates were washed before recycling. Therefore, contamination with stones and dust etc. can be excluded.

Bio-residues are removed during grinding and washing. According to a statistical evaluation at the recycling plant, the incidence of extraordinary bio-residues contamination is one pallets / crates of about 100 pallets / crates when coming back in the pooling loop. Subsequently the pallets / crates were washed before recycling. Therefore, contamination with stones and dust etc. can be excluded.

Microbiological contamination is mostly from rotten foodstuff. In addition, remains from funghi might occur. Microbiological contamination is removed as dust during washing. In addition, during recycling and extrusion the microbiological contamination is deactivated due to the applied hot temperatures.

Chemical contamination might occur from liquids like oil. It should be noted, that visibly contaminated pallets / crates are sorted out before recycling. So only pallets / crates with a rare invisible contamination might enter the recycling process. Chemical contamination on the surface of the pallets / crates is removed during washing of the pallets / crates and of the flakes. In addition, during recycling and the two extrusion processes, potential contaminants are volatilized are removed due to the applied hot temperatures.

Due to the fact, that it is most unlikely that pallets / crates with (invisible) chemical contamination are entering the process, this contamination cannot be analysed by analysis of the flakes, because if any only a very small amount of flakes might show hints of chemical contamination. Therefore, diffusion models were used for estimation of potential contamination levels from chemical contamination. For the calculations, the commercial available AKTS software was used. The diffusion coefficients were predicted according to the A_P model[3]. Sorption of chemical compounds into the pallets / crates can be considered as predominantly controlled by the diffusion process in the polymer as well as by the partition coefficient K between the polymer and the environment. For the calculations the partition coefficient of K = 1 has been used. K = 1 assumes a good solubility of the contaminant in the polymer as well as good solubility in the contact medium during the sorption phase. The results of the prediction of the sorption are given in Table 1.

The highest concentrations are found for small molecules and a wall thickness of the pallets / crates (e.g. 677 mg/kg for a substance with a molecular weight of 100 g/mol (e.g. a solvent) and a wall thickness of 1 mm). Higher wall thickness results in lower contamination levels, because of the fact that the sorption is not homogeneous. The highest concentrations are at the surface. It should be noted here, that the minimum thickness of the pallets / crates is 3 mm, so a thickness of 1 mm can be considered as worst-case.

As mentioned above, that the pallets / crates are visually inspected and are sorted out. Consequently, chemically contaminated pallets / crates should not be come into

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[3]Practical Guidelines on the Application of Migration Modelling for the Estimation of Specific Migration, EU Report 27529 EN, ISBN 978-92-79-52790-6, 2015.

the recycling stream. However, assuming a conservative approach, that the incidence of chemical contamination in pallets / crates (which are not sorted out) is one pallets / crates of 100000 pallets / crates, the resulting average concentration of the contaminant in the recyclate containing pallets / crates is about 0.00677 mg/kg only by dilution.

Table 1: Prediction of the sorption of potential contaminants into the HDPE pallets / crates (A_P = 14.5, τ = 1577 K), sorption time 10 d at 25 °C, concentration of contaminants 1000 mg/kg in contact with the polymer surface of the crates (K = 1)

Wall thickness of the pallets / crates	Average concentration of contaminant in the polymer in relation to the molecular weight of contaminant [mg/kg]						
	100 g/mol	200 g/mol	300 g/mol	400 g/mol	500 g/mol	750 g/mol	1000 g/mol
1 mm	677	341	194	119	76.6	29.8	13.3
3 mm	230	114	64.6	39.6	25.5	9.49	4.45
5 mm	138	68.2	38.7	23.8	15.3	5.97	2.67
10 mm	68.8	34.1	19.4	11.9	7.68	2.98	1.34

Within a literature study the influence of the recycling of pallets / crates has been determined[4,5]. The authors of the study concluded, that:

- no significant increase in overall migration or the fraction of migrants with a molecular mass below 1000 g/mol could be observed for PP and HDPE with increasing recycling steps.
- no increase in specific migration of any of the additives could be observed in 15% ethanol. In *iso*-octane, only the specific migration of Irgafos 168 increased for HDPE, while for PP the levels of Irgafos 168, Irganox 1010 and Chimassorb 944 increased with increasing recycling steps.
- in all cases the specific migration limit (SML) was not exceeded. Several unlisted, migratable components were found for both PP and HDPE that increased with increasing recycling steps. All of these components could be (semi-)quantified and identified or classified as degradation products of additives.

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[4]L. Coulier, H. G. M. Orbons, R. Rijk, Analytical protocol to study the food safety of (multi-)recycled, high-density polyethylene (HDPE) and polypropylene (PP) crates: Influence of recycling on the migration and formation of degradation products, Polymer Degradation and Stability, 2007, 92, 2016-2025.
[5]Guidelines on submission of a dossier for safety evaluation by the EFSA of a recycling process to produce recycled plastics intended to be used for manufacture of materials and articles in contact with food, The EFSA Journal, 2008, 717, 1-12.

Determination of Decontamination Efficiencies

It is expected, that process parameters used in the Novel Technology will have only a slight decontamination effect. Therefore, no experimental data on the cleaning efficiency were generated. The evaluation of the consumer exposure were evaluated without considering the decontamination efficiency of the Novel Technology (see below). However, the slight decontamination efficiency will bring additional safety factors.

Exposure Evaluation

Based on the considerations in section "Characterisation of Input Contamination Levels" the initial contamination level before the super-clean recycling process is set to 0.5 mg/kg for both input materials.

Based on the input concentration of 0.5 mg/kg the migration from pallets / crates at 23 °C for a storage time of 7 d was calculated. The results are given in Table 2.

In the first approach, the migration is calculated for pallets / crates thicknesses of 1 mm, 3 mm, 5 mm, 10 mm. As a result, the migration is independent from the thickness of the pallets / crates.

The pallets / crates are washed before the first use. Therefore, the concentration on the surface of potential contaminants is reduced during this washing step. This is quite analogous to multi-use applications. In the next step, the influence of the washing step (10 min at $\geq$50 °C) has been considered. The washing step reduces the initial concentration on the surface of the pallets / crates. Consequently the migration is also reduced.

In the third step of this calculations, the sorption of potential contaminants has been considered. According to Table 1 the sorption is dependent from the molecular weight of the contaminant. This reduced the initial concentration of 0.5 mg/kg accordingly.

Following this calculation, 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.

Table 2: Migration of potential contaminants from the HDPE pallets / crates (A_P = 14.5, τ = 1577) into contact materials after storage for 7 d at 23 °C, concentration of contaminants 0.5 mg/kg in polymer (K = 1), EU cube

	Migration [µg/kg]						
	100 g/mol	200 g/mol	300 g/mol	400 g/mol	500 g/mol	750 g/mol	1000 g/mol
1 mm thickness	14.8	7.40	4.22	2.59	1.67	0.652	0.292
3 mm thickness	14.8	7.40	4.22	2.59	1.67	0.652	0.292
5 mm thickness	14.8	7.40	4.22	2.59	1.67	0.652	0.292
10 mm thickness	14.8	7.40	4.22	2.59	1.67	0.652	0.292
1 mm thickness, with washing of crates before use (10 min at 50 °C)	12.5	6.26	3.57	2.19	1.41	0.549	0.246
1 mm thickness, with washing and initial concentration considered from Table 1	8.46	2.13	0.692	0.261	0.108	0.0164	0.00327

Technology Description

Figure 2 shows a simple block diagram of all unit operations used in the decontamination process.

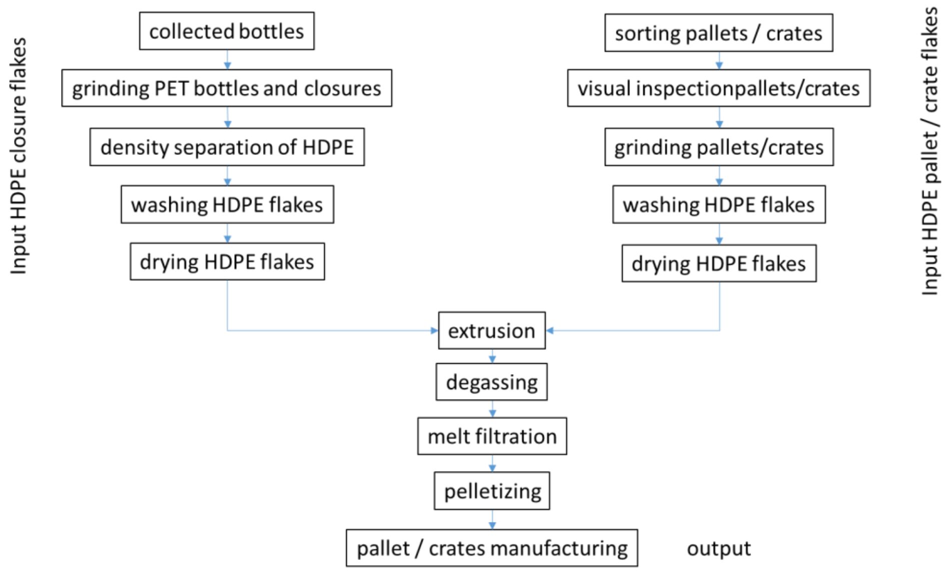

Figure 2: Block diagram showing flakes treatment, extrusion and pallets / crates production (non-confidential)

Differentiation from Existing Technologies

The super-clean technology used to produce HDPE Pallets from HDPE closures and recycled pallets to be used at up to 100% in food contact applications is a novel technology. Recycling of post-consumer HDPE packaging waste back into new food contact applications has not been established in Europe on an industrial scale to date. The only existing technologies to produce recycled plastics for food contact applications are the suitable technologies listed in Annex I of this regulation, which is PET recycling and closed loop recycling.

Proposed Evaluation Criteria

The proposed evaluation criteria are as follows:

- extruder temperature: [redacted]
- vacuum degassing: [redacted]
- surface drying of the pellets: [redacted] for approx. [redacted]
- pallets / crates extruder temperature : [redacted] for [redacted]

Decontamination Installation Operated for the Development of the Novel Technology

The development of the decontamination technology of the Novel technology is taking place at the facilities of Craemer GmbH, Brocker Straße 1, 33442 Herzebrock-Clarholz, Germany.

Due to the special characteristics and requirements of the Novel technology, it is not intended that other recyclers will use this process.

https://www.craemer.com/media/craemer_novel_technology_initial_report_v1-0-20092023.pdf