H2020-NMBP-HUBS-2019

FlexFunction2Sustain

Open Innovation Ecosystem for Sustainable Nano-functionalized Flexible Plastic and Paper Surfaces and Membranes

Starting date of the project: 01/04/2020
Duration: 48 months

= Deliverable D3.3=

Upgraded REMIX recycling pilot facility: 100 kg/h recycling capacity and 30% improved sorting accuracy with PRE certification

Dissemination level		
PU	Public	x
PP	Restricted to other programme participants (including the Commission Services)	
RE	Restricted to a group specified by the consortium (including the Commission Services)	
CO	Confidential, only for members of the consortium (including the Commission Services)	

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 862156

DISCLAIMER

Any dissemination of results reflects only the authors' view and the European Commission, or the European Health and Digital Executive Agency (HaDEA) is not responsible for any use that may be made of the information Deliverable D3.3 contains.

Executive Summary

FlexFunction2Sustain project aims to develop a network of upgraded lab-to-fab facilities, able to tackle challenges such as the shift from non-recyclable multilayers to recyclable mono material structures, with high barrier properties. This is part of the “Circularity by design” services offered through the FlexFunction2Sustain Open Innovation Test Bed (OITB).

This Deliverable 3.3 (D3.3) addressed the upgrades made to the REMIX recycling pilot facility available at IPC; as shown in Figure 1.

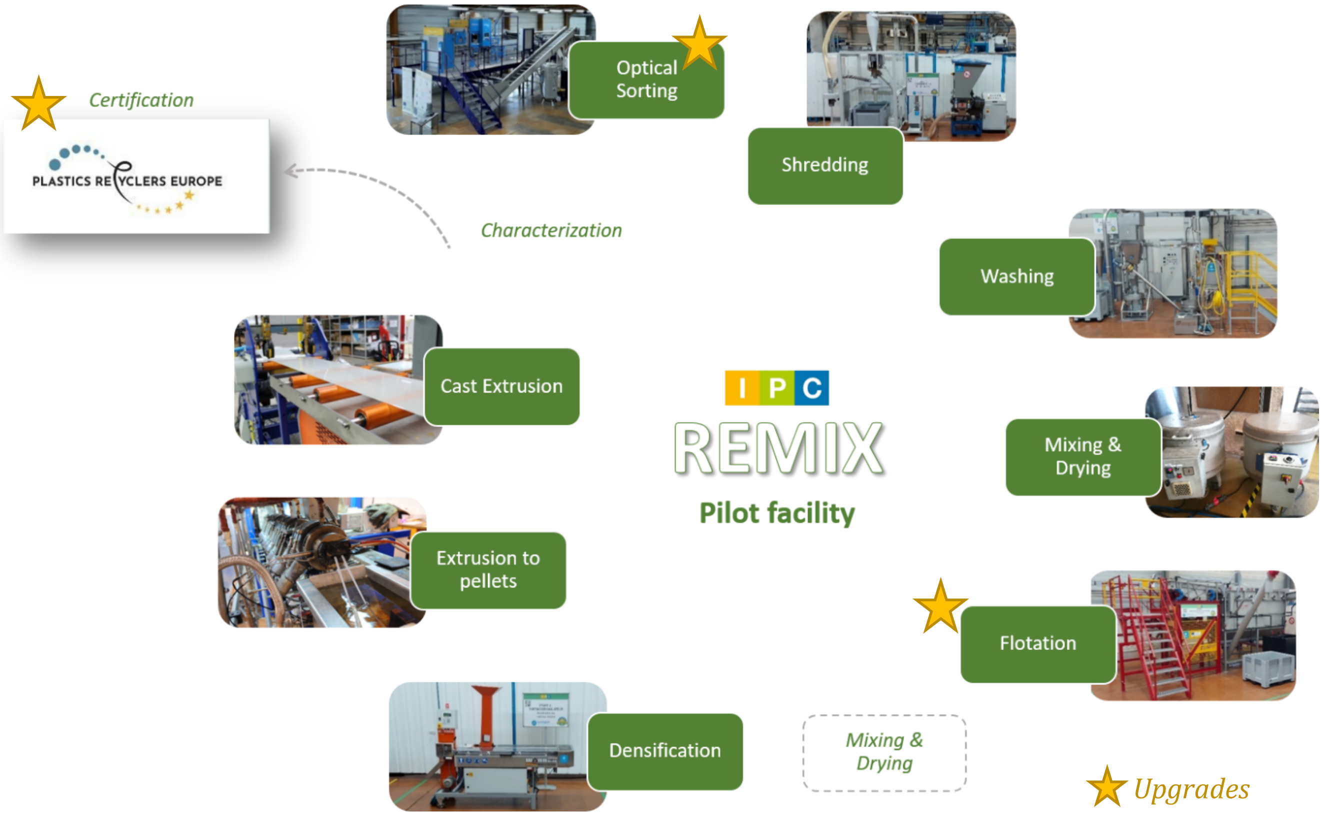


Figure 1: Remix pilot facility diagram

The upgrades described in this deliverable focus on:

- Installation of NIR optical sorting machine and optimization of the conveyor belts configuration in order to increase the sorting accuracy to 30%.
- Installation of Semi-automatic high volume sink-float (described in section 2.1.2) to increase the speed from 10 to 100kg/h
- Accreditation of the pilot facility for PP film recyclability evaluation and certification by Plastic Recyclers Europe (PRE).

Sorting and recycling tests were performed on mono PP drink pouches. A first recycling trial was performed on the innovative pouch structures in order to receive PRE accreditation. Then a second recycling trial was performed on the final product. Moreover, the improved sorting accuracy in a closed loop configuration was verified. It was observed that the presence of straws on the final packaging does not disturb its sorting. The recycling trial was conducted following PRE protocol for PP films. Figure 2 illustrates the recycled structures at each step of the recycling trial. In the output, films were obtained with different percentages of recycled material. Even if the haze of the films was affected by the presence of innovation material, the processability was not significantly affected. A large number of surface defects, mostly black spots, were also observed on the recycled films (blends B25 and B50).

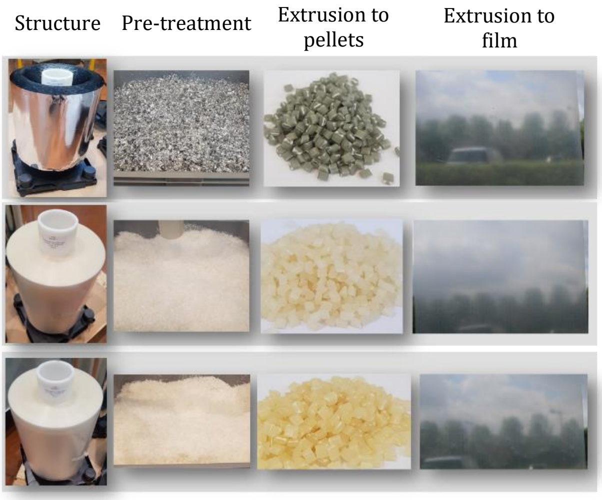


Figure 2: Recycling steps of mono PP drink pouches recycling

Table of Contents

1. Introduction

In order to support the targeted Open Innovation Test Bed (OITB) clients, Work package (WP) 3 aims at implementing a holistic Circular Economy approach, to serve the customer needs for the development of innovative and more sustainable products.

This deliverable addresses the up-grading of the REMIX recycling pilot facility at IPC. The targeted upgrades are demonstrated in this report, including the increased recycling capacity to 100kg/h and 30% improved sorting accuracy with PRE certification. This deliverable is linked to Task 3.3 of WP 3, pilot facilities for product recyclability test. This task aims to optimise the IPC's REMIX pilot line.

Task 3.3. demonstrate the recyclability of the new materials developed in the frame of the OITB following the four conditions defined by Plastic Recyclers Europe [1]:

- **Compatibility with existing sorting technologies** can be assessed at a pilot scale with a Near-Infrared optical sorting (NIR) machine and a semi-automated high volume-specific sink-float technology dedicated to films.
- **Regeneration** of products and materials can be evaluated on the whole REMIX Pilot Line. After shredding, washing, and drying along with stat-of-the-art industrial protocols, the plastic fluff can be filtered and regenerated through a twin-screw extruder and transformed into standard pellets.
- **Reprocessing of the recycled materials** into new products can be validated, from the pellets obtained after the regeneration through their re-processability via cast-film extrusion, injection moulding, extrusion of tubes, and extrusion blowing of films.
- Last but not least, **physico-chemical characterisation** of the regenerated pellets and the final products enables to confirm of the recyclability of the developed structures.

The recyclability evaluations are conducted in such a way that the recyclability testing protocols published by the Plastics Recycler Europe for Technology Approval are fully applicable using the REMIX pilot line. IPC applied for, and recently received, the accreditation of the REMIX pilot line by Plastics Recyclers Europe. Moreover, IPC is already recognized as a Certification Body regarding the Recyclability Certifications delivered by auditors.

Within task 3.3 of FlexFunction2Sustain, which seeks to set up an OITB, the involved partners AMCOR, FHG-IVV, COA, FHG-FEP, INL, and IPC have set up an experimental study to assist Capri-Sun in the development of a single polymer fully recyclable drink pouches.

In this deliverable, the upgrades are described in Chapter 2, Section 2.1. Section 2.2 describes comparative sorting tests performed on the multilayer and mono PP based drink pouches with the NIR optical sorting machine at IPC. Finally, section 2.3 describes the recyclability assessment of mono PP based drink pouches structures that lead to the accreditation of the REMIX line. The production of the 3 novel structures, made by FHG-IVV and AMCOR are detailed in deliverable 3.1. This section will focus on the recyclability trials held at IPC on the REMIX pilot line.

The present document will be publicly available, as the executive summary of the Remix recycling pilot facility available at IPC and offered through FlexFunction2Sustain.

2. Results and discussion

2.1. Recyclability assessment facility: REMIX pilot line description

The REMIX Pilot line is dedicated to plastic product evaluation at a semi-industrial scale, exploring the technical and economic balance between recovery in the pure streams and mixed ones, with a special focus on mechanical recycling.

The REMIX pilot line's main steps for the assessment of recyclability are:

- **Sorting,** with a near infrared optical sorting machine;
- **Pre-treatments**, including grinding, washing, flotation, and drying steps;

- **Extrusion**, successively with a thermogranulator, dedicated to converting the shredded film flakes into an agglomerated form, then with an extruder equipped with a filter, to allow a molten state filtration and remove impurities;
- **Converting** back into films, with a cast-film extrusion line.

The REMIX pilot line upgrades achieved through flexfunction2sustain have focused on:

- Installation of Near Infrared Optical sorting (described in section 2.1.1) to improve the sorting accuracy by 30%;
- Installation of Semi-automatic high volume sink-float (described in section 2.1.2) to increase the speed from 10 to 100kg/h;
- Recyclability evaluation following Plastic Recyclers Europe protocol testing for accreditation.

2.1.1. Near infrared sorting

Near Infrared Optical sorting

The REMIX pilot line has been upgraded with the installation of a Mistral + Connect Near-Infrared (NIR) Optical sorting machine (supplier: Pellenc ST), as seen in

Figure 3. This last generation machine was selected to be representative of modern sorting centers while offering the versatility to perform several types of testing.

Equipped with a multi-stream spectrometer, its detection covers near infrared to visible spectra. A sample will be detected from its visible upper surface material by analysis of the reflected spectra within the covered wavelengths. Thus, any sample reflecting light within the near-infrared to visible spectra can be analysed and recognized.

Linked remotely to the Pellenc ST database, including most polymers, the machine settings can be adjusted to sort by air-jet blowing one or several detected materials. It counts two exits: one for the ejected fraction, i.e. the sorted fraction, and the other for the rejected fraction.

Figure 3: Mistral + Connect Near Infrared Optical sorting (Pellenc ST)

Static detection tests can be performed to assess if a structure under development is detectable by the machine. Upon success, such a structure can be evaluated in its final form through dynamic tests. These dynamic tests can occur either with several units of the sample alone or within a waste stream, to mimic their behaviour in a real-life situation.

Several steps can then be studied. The first optical sorting step in a sorting center occurs on a mix of plastic packaging, paper/cardboard, and aluminum wastes. Later on, plastic wastes are sorted amongst each other through an optical sorting step to isolate each stream regarding its composition (PET, PP, PE…).

The key main results of dynamic tests are the efficiency of the stream sorting and the purity of the sorted stream. The efficiency measures the ability to gather all the aimed streams, while the purity covers the ability to gather specifically the aimed stream.

This machine can be fed with either flexible or rigid samples, smaller than 350 mm and with an area density up to 10 kg/m^2, and is equipped with a high resolution sorting bar, to enable the sorting of smaller elements, down to 20 mm.

Detection of widely used petroleum-based plastic (PET, PP, PE…) is possible, as well as the detection of biopolymers. The exceptions to the machine's detection and sorting abilities are non-exhaustively summed up in the next Table.

Table 1: Main exceptions to the Mistral + Connect detection and sorting abilities

Plastic films with a thickness below 15 µm
Black and very dark objects
Materials containing carbon black
Objects constituted of different inseparable materials (including multilayers, such as PE/cardboard)
Rolling objects
Packaging containing more than 100 mL of liquid
Synthetic textiles (polyesters…) cannot be sorted from a plastic stream
Natural fibres textiles cannot be sorted from a wood, paper and cardboard stream
Wet paper, cardboard, wood & natural fibres textiles are detected as the same material
PVC containing less than 20% chlorine
Inorganic chlorine (salts)

An original design of conveying systems has been implemented by IPC, with the objective to set up an optical sorting line representative of state-of-the-art sorting facilities. This upgrade of IPC's optical sorting line allows sorting in a loop, either to enable refined sorting, with the same stream being purified through each pass, or to simulate a cascade of several NIR sorting machines, each pass sorting a different stream. Therefore, several conveyors form a loop (Figure 4 & Table 2) around the machine.

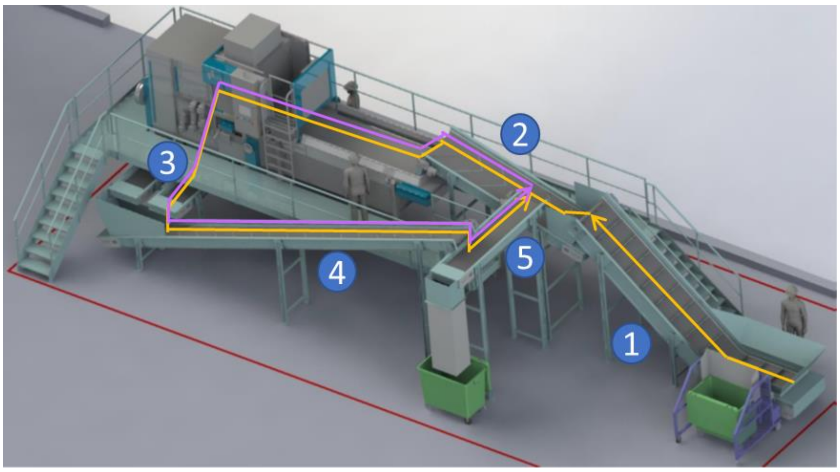


Figure 4: Conveyors loop around the Mistral+ sorting machine

Table 2: Conveyors installation specifications

Conveyor	*Role*	*Width (mm)*	*Length (mm)*	*Area (m^2)*	*Speed (m/min)*	*Course duration (min)*
1	Feeding	1000	5695	32	2 - 6	0.32 – 0.95
2	Spreading	1000	3900	15	30	0.13
Mistral +	Sorting	1200	4770	23	180	0.03
3	Exit	600	4200	18	30	0.14
4	Loop	600	9750	95	30	0.33
5	Transfer	600	3050	9	30	0.10

Once the machine will be set up, sorting tests could take place following different protocols, depending on the needs. For example, Recyclass (European) or COTREP (French) protocols regarding optical sorting of packaging could be performed. The base principle of those protocols is to test a known number of packaging units – typically 50 to 100 units, depending on their size - within a waste stream, as shown in Figure 5.

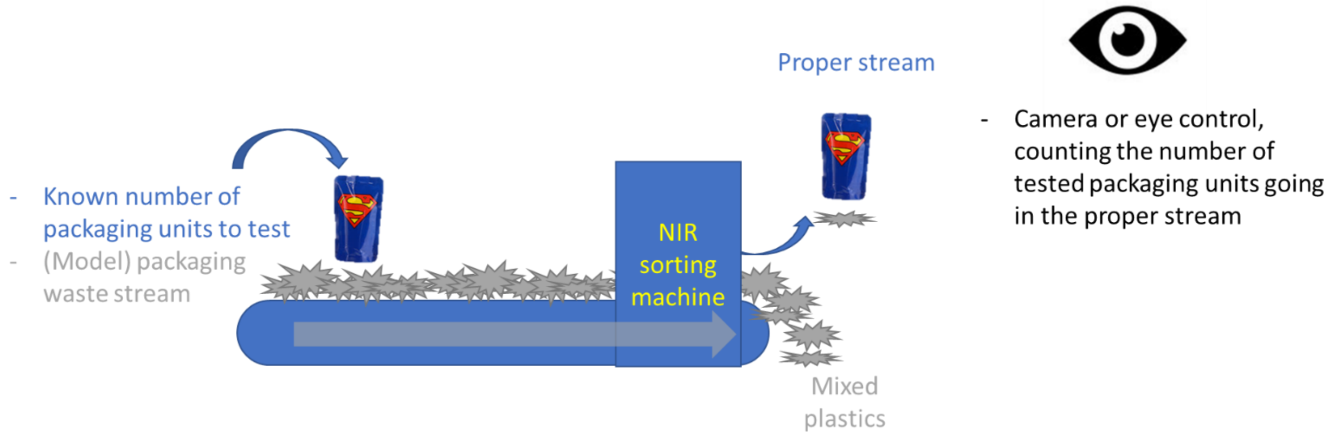


Figure 5: Sorting protocols principle

Plastics Recyclers Europe, an association representing the European plastics recyclers, was contacted, to advise on the best sorting assessment possibilities, and they recommended being as representative of reality as possible, hence, to privilege the protocols involving waste streams collected from sorting facilities.

In a nutshell, depending on the samples to be assessed, their optical sortability can be tested: by static testing, to screen the most detectable structures, and/or dynamic testing, either alone, within a sampled or model stream waste, and/or with a refined sorting by looping through the optical sorting machine for several passes.

Table 3: Waste stream types possibilities for sorting tests

*PRE : Plastics Recyclers Europe

Type	*Source*	*Pro*	*Cons*
SAMPLING	From sorting facility (pre-sorted films)	- Closer to real life waste stream	- Procurement difficulty - Limited repeatability - Odours & sanitary risks
	From sorting facility (fully sorted materials)	- Closer to real life waste stream - Validated by PRE*	- Limited repeatability - Odours & sanitary risks
MODEL	Commercial films samples	- Repeatability	- Not validated by PRE - Needs information about relevant composition & proportion
	Emptied commercial packaging	- Repeatability	- No validated by PRE - Wastage or months in the making (consumption of the products)

2.1.2. Pretreatments

The recyclability evaluation for a Technology Approval of an innovative product starts at this point. The steps and equipment are described below.

Shredding

The shredding is performed by a **single shaft shredder** (Figure 6). The equipment consists of a shaft with a dozen moving cutting tools, and various calibrated grids of 10, 20, and 30 mm.

The shredder is equipped with a vacuum emptying system, its flow rate varies depending on the shape and material of the products to be processed.

Figure 6: Shredder

Washing step

The washing is carried out in a **washing module** equipment (Figure 7) with a temperature-regulated tank where the sample is washed by friction under the effect of a rotating agitator and a set of fixed blades.

Then, the tank is unloaded onto a vibrating screen to collect as much water as possible that can be reused for a new wash. To finish, the washed wastes are conveyed by an endless screw to a dryer before being evacuated by a cyclone.

Figure 7: Washing module (left: dryer & cyclone, right: washing tank)

Wet sorting

Sink and float sorting is well used at the industrial level. Our pilot line was upgraded with a **dynamic flotation bath** (Figure 8), equipped with several rollers on the water surface, conveying the materials through the vessel. Regular feeding over the entire width of the vessel is provided by a vibrating conveyor and a spray bar. All flakes are immersed several times before being evacuated by overflow for the floating fraction. This allows sorting by different density and also remove contaminants and impurities like wood and metals (making it possible for less dense materials to float and the heavier to sink). This upgrade allows to go from a waste treatment of 10kg (static flotation bath) to 100kg per hour.

Figure 8: Dynamic flotation bath (left: outside view, right: inside view)

Drying

Drying steps are carried out in an **industrial oven** (Figure 9). If necessary, the drying is performed by a flow of heated air distributed over the entire surface of the oven for uniform drying. The films to be dried are packaged in fabric bags allowing handling and avoiding cross-pollutions.

Figure 9: Industrial oven (left: outside view, right: with fabric bags)

2.1.3. Extrusion

After the pretreatments, the sorted, ground, washed, and dried samples go through the mechanical recycling step of filtering and compounding. Beforehand, if the samples are shredded film flakes, they need to be transformed into an agglomerated form through densification.

Film densification

The **thermogranulator** (Figure 10) has the particularity of being able to transform plastic films from a shredded form to an agglomerated form, its feeding system is composed of an auxiliary screw making it possible to feed the plasticizing single screw with a large volume of raw material.

The thermogranulator is used to densify the ground films to make them usable in standard processing techniques in the plastics industry.

Figure 10: Thermogranulator

Melt filtration

The **corotating twin-screw extruder** is characterized by a screw diameter of 32 mm and an L/D ratio of 44. The raw material supply is ensured by a set of weight feeders allowing the dosing of a multitude of solid and liquid materials.

Filtration of the molten material is carried out by a manual two-position **filtering system** (Maag HSC-050) located at the head of the extruder (Figure 11). The filtration diameter is 50 mm, i.e. the filtration surface is approximately 20 cm^2, a pressure sensor placed before the screen allows to monitor the clogging of the filter. This filtration system can work at a differential pressure of around 200 bars and temperature up to 300°C. The filtration fineness can be adjusted through different filters and filter combinations: 105µm, 150 µm, 300 µm, and 500 µm.

The extruder is equipped with a water cooling tank to solidify strands then a granulator cuts them into cylindrical granules.

Figure 11: Extruder and melt filter

2.1.4. Converting

The processability and conversion of the recycled material into the new plastics part can be tested and optimized at the pilot scale. IPC is equipped with a wide range of converting machines including: dosing, mixing, compounding, filtering, profile, tube extrusion, calendaring, co-extrusion, blown film extrusion as well as injection moulding machines. For the regeneration of films in particular, the same extrusion cast-line mentioned in part 2.2 can be used, directly associated with an extruder.

Cast film extrusion

The **cast film line** is equipped with a 350 mm wide flat die, the adjustable lips of which allow the thickness to be varied from 25 to 250 µm.

The die head is equipped with ten heating zones (eight control zones distributed over the entire flat die and two heating zones for the upper and lower lips) adjustable up to a maximum temperature of 300 ° C.

The pulling line is made up of a temperature-regulated 400 mm diameter chill-roll, the temperature of the roller can be controlled between 40 and 160 ° C, with oil (Figure 12).

Figure 12: Cast-film line

The rotational speed of the chill-roll allows the film to be stretched over a range of 0 to 35 m/min. Four steel rollers are arranged along the line to allow air cooling of the film. Finally, a movable roller at the end of the line not only keeps the film tension constant but also rolls up the film obtained. A cutting module allowing a “slackening” operation can be placed upstream of this roll.

2.1.5. Characterizations

Laboratory tests are performed to assess the quality of recovered plastics after mechanical recycling. Table 4 sums up, non-exhaustively, the equipment available to perform the necessary analysis.

Table 4: List of the analysis & laboratory equipment involved in recyclability assessment

Analysis	Equipment
Bulk Density (ISO 15344)	- Apparatus for the determination of bulk density - 2000 ml.
Density (ISO 1183-1)	- Precision scale METTLER TOLEDO AE240 ; - Accessories for density measurement.
Melt Index (ISO 1133)	- Melt Flow Indexer GOTTFERT Mi-40 ; - Capillary die, length 8 mm and diameter ≈ 2.1 mm ; - Precision scale METTLER TOLEDO AE200 ; - Ventilated oven HERAEUS UT 6060.
Ash content (ISO 3451-1)	- Muffle furnace NABERTHERM ; - Precision scale METTLER TOLEDO AE200

DSC (ISO 11357-3)	- DSC calorimeter Q100 TA Instruments ; - Indium reference standard ; - Lead reference standard ; - Precision scale METTLER TOLEDO AE240.
Volatiles	- Ventilated oven HERAEUS UT 6060 ; - Precision scale METTLER TOLEDO AE200 ; - Timer NOVO 82110.
Colorimetry (L*,a*,b*)	- Spectro guide BYK Gardner.
Thickness (ISO 4593)	- Heidenhain thickness gauge.
Tear Strength (EN ISO 6383-1 or EN ISO 6383-2, Elmendorf method)	- Zwick/Roell BT2-FR005TH.A50
Tensile properties (ISO 527-3)	- Dynamometer ZWICK 1.0 ; - 1 kN sensor ; - Thermo-hygrometer TESTO TERM 6010 ; - Digital micrometer 25 mm.
Dart Impact (ISO 7765-1)	- Dart Tester
Gels and Specks	- Microscope VHX-7100 KEYENCE.

2.1.6. Certification possibilities

The whole REMIX line presented above enables the OITB to apply the **Recyclability Evaluation Protocol for Films,** called **RecyClass protocol** [2], and developed by PRE (Plastics Recyclers Europe). It is a methodology that must be followed by the plastics packaging producers, at a pilot scale, in order to identify if a plastic packaging innovation is compatible with the existing post-consumer film recycling streams. IPC is now recognized by Recyclass as a laboratory to carry out these protocols for PP and PE films (certificate on Figure 13).

Figure 13: IPC's Certificate of recognition from RECYCLASS

As for the previously presented recycling steps, the protocol follows three main phases, as illustrated in Figure 14: pre-treatment, extrusion & pellet characterization, and conversion, also followed by characterization. The input required at the beginning of this protocol is around 10 kg of innovation film and 25 kg of control film. The control film should be the same base resin as the innovation, except for the specific ingredient/feature being evaluated. During the extrusion step, 3 blends of control and innovation flakes will be prepared, containing 0%, 25%, and 50% of innovation; and during the conversion step, the batches will be diluted again, with 50% of virgin pellets, in order to prepare 3 blends of control and innovation pellets, containing 0%, 12.5% and 25% of innovation. This protocol will be described in detail in the next section, applied to one of the FlexFunction2Sustain use cases.

Based on the processability and characterizations of the recyclates, the difference between the control film and the innovation film is assessed. If the innovation packaging is successfully recycled through the protocol, a Technology Approval can be delivered by Recyclass.

Complementary to this technology approval, packaging or semi-finished packaging can also be certified through Recyclass [3] by IPC, a Recyclass-recognized Certification Body. There are three types of recyclability assessments for plastic packaging, answering the needs of the entire plastic value chain. Packaging entirely covered by the Design for Recycling guidelines can be evaluated as follows:

- **Design for Recycling Certification** classifies qualitatively from A to F the technical recyclability of a final plastic packaging on the EU market. All the recognized Certification Bodies are accredited for this assessment.
- **Recyclability Rate Certification rates** the effective recyclability of a final plastic packaging in the specific geographical area for which the assessment is conducted. IPC is able to deliver the Recyclability Rate Certification for France.
- **Letter of compatibility** evaluates the recyclability of semi-finished packaging qualitatively and based on the Design for Recycling Certification. All the recognized Certification Bodies are accredited to deliver a Letter of Compatibility.

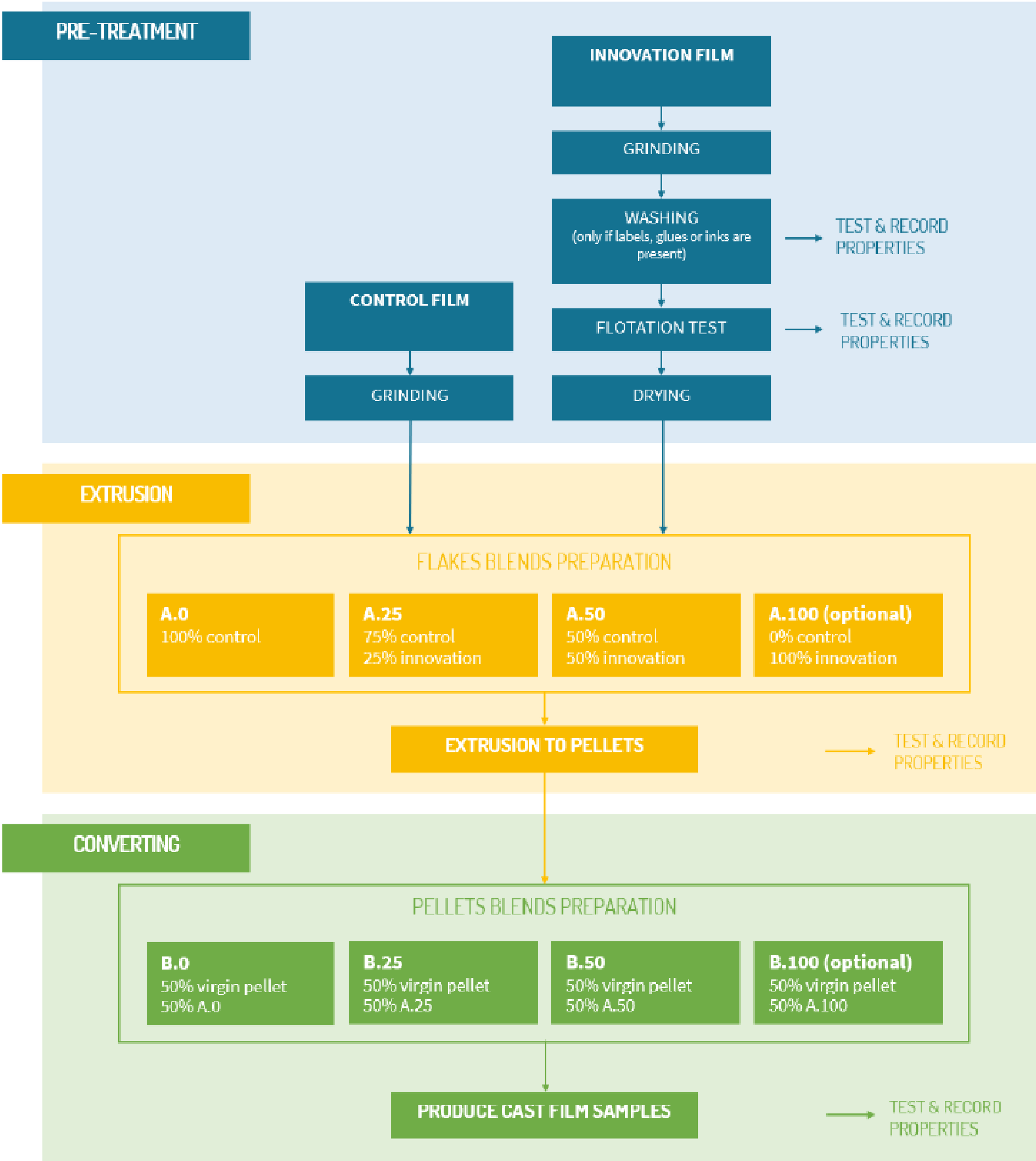


Figure 14: The Reclyclass protocol for flexible PP (v2.1) [2]

FlexFunction2Sustain offers those services and high added value expertise by using the upgraded REMIX pilot line. We are now fully equipped to provide specific indications and recommendations to the European packaging producers, for the Eco-design of a new film structures that will allow the flexible packaging industry to switch from non-recyclable laminates multilayer to fully recyclable mono material flexible packaging. The REMIX pilot line is ready to be used to support our clients in developing sustainable packaging and reducing their time-to-market.

2.2. Recyclability evaluation

The REMIX pilot line upgrades have been verified through different tests: recycling and sorting trials. The case studied in these trials is flexible PP drink pouches, from project partner Capri Sun (WP5).
Three novel PP-based structures, described in Figure 14, have been developed within the framework of FlexFunction2Sustain. The full study is described in the deliverable D3.1, "Upgrades of Circularity by design facilities demonstrating possessing of 95% Polyolefin based films" which is focusing on the SOA, life cycle assessment, production conditions and characterization results.

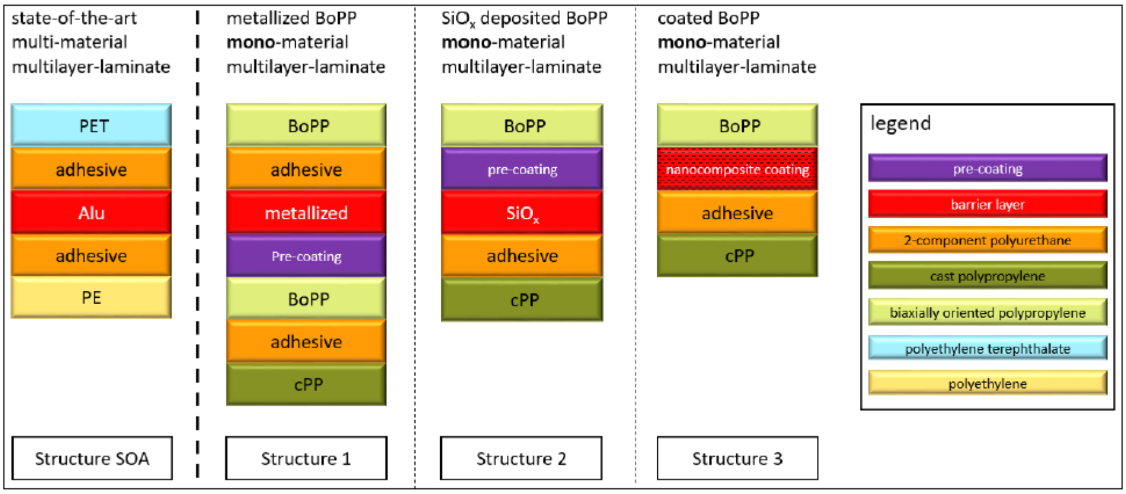


Figure 15: Structure of a State-of-the-Art multi-material laminate (the very left picture) in comparison to the novel mono material based laminates, i.e. Structure 1, Structure 2, and Structure 3 with different thin barrier technologies [5]

A first recycling assessment was held on the 3 novel post-production films (presented in Figure 15). This test followed the Recyclass recycling protocol (Figure 14) in order to receive the accreditation of the recycling line for flexible PP. Then, a new recycling campaign was held on the post-consumer product (made with structure 1). The sorting test was held on the mono material pouches from Capri Sun (structure 1) following the sorting protocol (Figure 3).

The main goal of these studies is to verify that the new mono material structures can be sorted and recycled without disturbing other streams and degrading the quality of recycled material.

2.2.1. NIR sorting test

The sorting tests were held on both multilayer structure and novel structure pouches, as shown in Table 5. The sorting of the pouch with and without straws was also tested in order to observe any influence of its presence on the pouch sorting.

Table 5: Samples used for the sorting test

Original structure *(Structure SOA)*	**Novel structure** *(Structure 1)*	
		
Multilayer structure	Mono PP pouch without straws	Mono PP pouch with straws

According to the Recyclass sorting protocol (*methodology diagram* in appendix 1), the flexible plastic stream is going through 3 NIR sorting machines. First, the drinking cardboards are ejected, then LDPE films, then PP films. After those steps, the remaining stream is considered as a mixed flexible waste.

Figure 16: NIR ejection order for plastic films streams in sorting centers

It is important to reproduce the same conditions as in a sorting center. This is why the pouches will go through each of the following steps mentioned above. We are expecting the pouches to be only ejected in the last step of the sorting, when PP is ejected. Each test is held with 50 pouches mixed in a representative stream.

Figure 17: Left: mixed plastic stream; Right: flexible plastic stream

Table 6: Sorting test results for each scenario

	Multilayer pouches	**Mono PP pouch without straws**	**Mono PP pouch with straws**
Mixed stream, drinking cardboard ejection	No ejection	No ejection	No ejection
Flexible stream, LDPE ejection	Less than 15% ejection	Less than 10% ejection	Less than 10% ejection
Flexible stream, PP ejection	14% ejection	88% ejection	90% ejection

The ejection percentage is calculated as follows:

$$\frac{Number\ of\ ejected\ pouches}{Number\ of\ pouches\ in\ input}$$

It was observed that the ejection percentage could vary from one test to the other depending on the loading rate of the conveyor belts. If the loading rate is higher than 40%, the sorting accuracy is most likely to decrease. Indeed, waste will overlap and be less detectable or accidentally blown. Therefore, the loading rate should be between 25 to 35% (rate calculated by the machine).

Figure 18: Loaded conveyor belts with mixed stream

During the sorting of mono PP pouches, it was observed that the sorting errors were mostly due to plastic overlap. The presence of straws did not disturb the sorting. However, the sorting test in a close loop condition allowed us to gain in accuracy. In this arrangement, the ejected waste stream is set aside (flexible PP) and the non-ejected stream is sent back to the sorting machine for further sorting (same sorting : PP films). This configuration allows non-blown PP waste to be detected during the 2nd passage in the sorting machine. After 3 loops, we obtained a 100% sorting accuracy for the flexible PP pouches (table 7). This result is very promising since it confirms that working in a close loop configuration could indeed increase the global sorting accuracy by more or less 30%. A cascade installation of two or three NIR machines could be considered in sorting centers to increase their sorting efficiency.

Table 7: Sorting test in a close loop configuration (mono PP pouch)

# Loops	% of pouch ejection
1	88%
2	94%
3	100%

2.2.2. Post-production film recycling

This recycling test is following the guidelines of the PRE recycling protocol for PP films (Figure 14). The first recycling trial was held on the film structure presented in the table below. A second recycling test is currently ongoing on post-consumer material (Table 5).

Table 8: Samples used for the first recycling trials

Innovation sample		
		
Structure 1: Metallized BoPP mono material multilayer laminate	**Structure 2**: SiOx deposited BoPP mono material multilayer laminate	**Structure 3**: coated BoPP mono material multilayer laminate

Control film	*Raw material*
	
Cast PP used in the innovation material	**Virgin PP**

- ## Pre-treatment

Figure 19: Pre-treatment steps

Grinding is carried out in our single-shaft shredder with a calibrated 20 mm grid, below is a summary of the flakes resulting from this treatment:

Table 9 : Picture of materials after grinding

Control material	**Structure 1**	**Structure 2**	**Structure 3**
			

The **washing** step is carried out according to the following procedure in our washing module. Once the flakes are washed, they are separated from the water with a vibrating screen and then dried.

Table 10: Picture of washing and drying steps

Structure 3 washed water	**Structure 3 on vibrating screen**	**Structure 3 after drying**
		

The **flotation** stage is carried out in our tank equipped with several rollers on the water surface, these allow the material to move. All flakes are immersed several times before being evacuated by overflow for the floating fraction. Regular feeding over the entire width of the vessel is provided by a vibrating conveyor and a spray bar.

Table 11: Picture of flotation step

Structure 1 during flotation	Structure 1 after flotation
	

No sinking fraction nor fractions in suspension were observed. The flakes were dried after the flotation test.

- **Extrusion to pellets**

The second main part of the recycling protocol is the compounding step. 3 blends have to be prepared for each structure according to the Recyclass protocol. The control film is cast polypropylene used in each structure (cPP). In accordance with the quantities to be prepared, the blending is carried out by a rotary mixer.

Table 12: Flakes blends compositions

Blends	A.0	A.25	A.50
wt% control	100	75	50
wt% innovation	0	25	50

F**ilm densification** is required with the flexible products so that they can be used in standard processing equipment such as extrusion for pellet production. A thermogranulator is used to agglomerate the flake.
Granulation is carried out with our 32 mm twin screw extruder equipped with a melt-filter system, run time extrusion is at least 30 minutes per sample. The samples are dried in a ventilated oven with hot air before processing.
Remarks: The granulation took place in good conditions; no change of screen was needed, despite the presence of relatively large quantities of black spots in the filters. In general, A.25 and A.50 blends had a higher starting pressure, this is confirmed by the MFI measurements where those blends are more viscous than the control (A.0). The innovation brings a significant color change, and almost halves the fluidity of the materials, which can affect the processability.

Table 13: Pictures of blends after extrusion to pellets

A0	**A25**	**A50**
	Structure 1	
		
	Structure 2	
		
	Structure 3	
		

- **Converting**

The different blends are prepared for each structure. This last step of the recycling protocol consists of converting the pellets into cast film samples for the final analysis.

Table 14: Blend preparation for converting

Blends	B.0	B.25	B.50
Wt% A blends	50 *(A0)*	50 *(A25)*	50 *(A50)*
Wt% virgin material	50	50	50

For the conversion, there are small differences in processability but this is not significant. From a mechanical properties point of view, the main difference is observed for the elongation at yield, which doesn't respect the requested benchmark. In addition, the coloring already observed during extrusion influences the haze, then a large number of surface appearance defects on samples B.25 and B.50 (mainly black spots) is observed.

Figure 20: Cast extrusion of B.25 (structure 2)

The characterization results for the pellets and the films are displayed in appendix 2.

Figure 21: Cast extruded film for each structure and composition

2.2.3. Final eco-designed packaging recycling

The second recycling trial is focusing on the post-consumer product. In this case, the drink pouch is made from the novel structure #1. This trial is following the same protocol as the first trial. The main purpose of this assessment is to observe if the novel structure, the inks or straws will have an impact on the recycled PP stream. Indeed, it was considered that the consumer will through away the pouch with the straw inside. The straw will eventually end up in the PP recycling stream; therefore, it is important to evaluate its impact. For comparison, a stream with and without paper straws will be studied in parallel. The recycled material (at different dilutions; see protocol Figure 14) will be compared to virgin PP and to the 1st recycling results (structure without inks and straws).

Table15: Final packaging pre-treatment steps

	Pouch	Pouch + straw
Input		
After shredding		
After flotation		

The semi-automatic flotation bath was used to test the behavior of the final product in an industrial environment with a treatment rate of 100 kg/h. By visual inspection, the PP flakes were well separated from the shredded paper straws after sinking in the bath. The inks did not bleed during the washing and flotation tests.

Figure 22: Samples in semi-automatic flotation bath

The compounding and extrusion steps will take place in the next weeks.

3. Conclusions

Deliverable 3.3 presented the recyclability evaluation service involved in a "Circularity by design" approach, available in Facility Clusters 6 of the Open Innovation Test Bed project FlexFunction2Sustain.

The Remix pilot line has been fully upgraded and is now accredited to perform recyclability assessments according to PRE protocol for PP films. Thanks to the upgrades, the sorting means of the recycling facility have been improved with the installation of a semi-automatic flotation bath and a NIR optical sorting machine. It is now possible to process over 100kg/h of material in the sink-float separation tank. Moreover, the NIR sorting machine can separate a specific material according to its composition from a mixed stream. The configuration of the conveyor belts has been optimized in order to be able to reproduce the same configurations of sorting centers. It is possible to sort the waste in an open loop (only one detection) or in a closed loop (multiple detections). It was proven that the last configuration improved the sorting accuracy after 2 loops.

An executive summary and a more detailed description of all the "Circular by design" services and certification possibilities have been assembled in report D3.5, available on the FlexFunction2Sustain website (https://flexfunction2sustain.eu).

4. Degree of progress

Deliverable 3.3 is fulfilled by 100 %.

5. Dissemination level

Deliverable D3.3 " Upgraded REMIX recycling pilot facility: 100 kg/h recycling capacity and 30% improved sorting accuracy with PRE certification" is a public document, and version 1.0 is available for download.

References

[1] Plastics Recyclers Europe, «Glossary,» [En ligne]. Available: https://www.plasticsrecyclers.eu/glossary#:~:text=Plastics%20must%20meet%20four%20conditions,by%20a%20legislatively%20mandated%20program..

[2] «Recyclability Evaluation Protocol for PP films,» [En ligne]. Available: https://recyclass.eu/wp-content/uploads/2022/02/RecyClass-Recyclability-Evaluation-Protocol-for-PP-films_v2.0-FINAL.pdf.

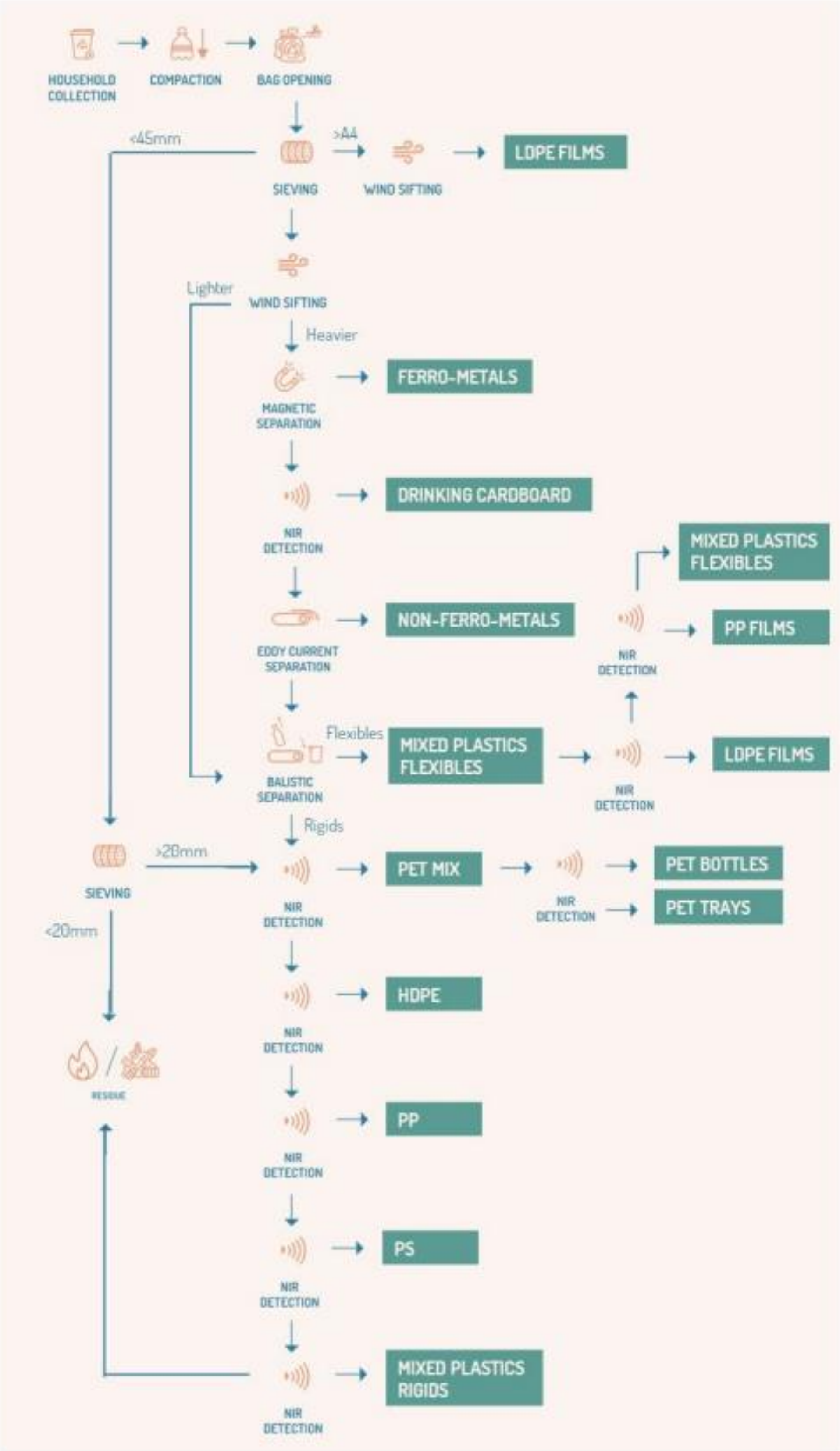
[3] «Recyclass website,» [En ligne]. Available: https://recyclass.eu/.

[4] Recyclass, «Sorting evaluation protocol for plastic packaging,» [En ligne]. Available: https://recyclass.eu/wp-content/uploads/2021/10/SORTING-EVALUATION-PROTOCOL-FOR-PLASTIC-PACKAGING_FINAL-V1.0.pdf.

[5] Fraunhofer IVV, IPC, OET, «Upgrades of "Circularity by design" facilities demonstrating (D3.1),» 2022.

6. Appendix I

Recyclass Sorting methodology Diagram [4]



7. Appendix 2

Structure 1 characterizations results

Structure 1

Pellets Properties		A.0	A.25	A.50
Density [kg/m³]		887	898	919
MFI [g/10min]		11,2	5,89	5,52
Ash content [%]		0,22	0,2	0,28
Reflection colours	L*	75,77	47,51	43,11
	a*	-0,92	-1,74	-1,53
	b*	3,73	3,64	2,08
	ΔE		28,27	32,71
Filtration (visual)		Some build-up	Some build-up	Some build-up
Moisture [%]		0,06	0,02	0,03
DSC melt temperature [°C]		158	161	162
Impurities		None	None	None
Surface appearance		Clean	Clean	Clean
Volatiles [%]		0,02	0,046	0,066
PE content [%]		None	None	None
Average Pressure [MPa]		10	32	33
Delta pressure [MPa]		6	9	8
Extrusion Process		Pressure increase	Pressure increase	Pressure increase

Conversion - Cast Film	B.0	B.25	B.50
Thickness [µm]	65	58,9	58,8
Tear Strength (TD*) [kN/m]	381	387	364
	± 30,00	± 25,00	± 33,00
Tear Strength (MD**) [kN/m]	344	288	334
	± 39,00	± 27,00	± 21,00
Tensile Strength (TD) [MPa]	21,9	21	17,7
	± 1,80	± 0,80	± 2,10
Tensile Strength (MD) [MPa]	23,5	21,4	21,7
	± 1,30	± 0,90	± 0,50
Elongation at Yield (TD) [%]	9	5,6	6,7
	± 0,70	± 2,80	± 1,00
Elongation at Yield (MD) [%]	10	9,6	9,7
	± 1,00	± 0,40	± 0,50
Haze [%]	19	21,4	27,7
	± 1,30	± 0,60	± 0,50
Gels and Specks	5	29	51
Surface Appearance	Clean	Gels & Specks	Gels & Specks

Structure 2 characterizations results

Structure 2

Pellets Properties		**A.0**	**A.25**	**A.50**
Density [kg/m³]		887	889	898
MFI [g/10min]		11,2	8,13	8,47
Ash content [%]		0,22	0,24	0,31
Reflection colours	L*	75,77	69,77	65,66
	a*	-0,92	-0,67	0,49
	b*	3,73	17,06	20,41
	ΔE		14,62	19,56
Filtration (visual)		Some build-up	Some build-up	Some build-up
DSC melt temperature [°C]		158	159	160
Impurities		None	None	None
Surface appearance		Clean	Clean	Clean
Volatiles [%]		0,02	0,039	0,063
PE content [%]		None	None	None
Extrusion Process		Pressure increase	Pressure increase	Pressure increase

Conversion - Cast Film	**B.0**	**B.25**	**B.50**
Thickness [µm]	65	70,7	73,6
Tear Strength (TD*) [kN/m]	381	341	353
	± 30,00	± 17,00	± 17,00
Tear Strength (MD**) [kN/m]	344	295	275
	± 39,00	± 42,00	± 28,00
Tensile Strength (TD) [MPa]	21,9	16,2	19,3
	± 1,80	± 2,30	± 1,00
Tensile Strength (MD) [MPa]	23,5	25,2	17,9
	± 1,30	± 4,70	± 1,30
Elongation at Yield (TD) [%]	9	6,8	10
	± 0,70	± 0,60	± 1,00
Elongation at Yield (MD) [%]	10	10	9,5
	± 1,00	± 1,00	± 0,60
Haze [%]	19	37	34
	± 1,30	± 0,30	± 1,20
Surface Appearance	Clean	Gels & Specks	Gels & Specks

Structure 3 characterizations results

Structure 3

Pellets Properties		A.0	A.25	A.50
Density [kg/m³]		887	886	899
MFI [g/10min]		11,2	6,29	5,44
Ash content [%]		0,22	0,26	0,34
Reflection colours	L*	75,77	65,33	61,77
	a*	-0,92	1,51	3,96
	b*	3,73	20,97	24,91
	ΔE		20,30	25,85
Filtration (visual)		Some build-up	Some build-up	Some build-up
DSC melt temperature [°C]		158	160	161
Impurities		None	None	None
Surface appearance		Clean	Clean	Clean
Volatiles [%]		0,02	0,032	0,05
PE content [%]		None	None	None
Extrusion Process		Pressure increase	Pressure increase	Pressure increase

Conversion - Cast Film	B.0	B.25	B.50
Thickness [µm]	65	56,8	59,5
Tear Strength (TD*) [kN/m]	381	383	379
	± 30,00	± 28,00	± 24,00
Tear Strength (MD**) [kN/m]	344	327	297
	± 39,00	± 35,00	± 32,00
Tensile Strength (TD) [MPa]	21,9	23,6	22,6
	± 1,80	± 0,90	± 1,30
Tensile Strength (MD) [MPa]	23,5	21,6	19,4
	± 1,30	± 2,90	± 1,80
Elongation at Yield (TD) [%]	9	8,7	10
	± 0,70	± 2,50	± 1,00
Elongation at Yield (MD) [%]	10	7,7	8,2
	± 1,00	± 0,20	± 1,30
Haze [%]	19	23,8	26,7
	± 1,30	± 0,70	± 0,30
Surface Appearance	Clean	Gels & Specks	Gels & Specks

* TD = Transverse Direction

** MD = Machine Direction