RecyClass

RECYCLABILITY EVALUATION PROTOCOL

FOR PP FILMS

STANDARD LABORATORY PRACTICE

REP-PPflex-01

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RecyClass

CONTENT

RecyClass

GLOSSARY

A.0	100 % control film flakes
A.25	Blend 75/25 control/innovation flakes
A.50	Blend 50/50 control/innovation flakes
A.100	Blend 100 % innovation film flakes
ASTM	American Society for Testing and Materials
B.0	Film made of 50 % of virgin pellets and 50 % of A.0 pellets
B.25	Film made of 50 % of virgin pellets and 50 % of A.25 pellets
B.50	Film made of 50 % of virgin pellets and 50 % of A.50 pellets
B.100	Film made of 50 % of virgin pellets and 50 % of A.100 pellets
Control Sample	Plain PP film (or PP resin that has already been thermally processed once) used as benchmark
EN	European Standard
Innovation Sample	Film containing the innovative technology
ISO	International Organization for Standardization
MFI	Melt Flow Index
PE	Polyethylene
PP	Polypropylene
PVC	Polyvinyl Chloride
TC	Technical Committee
TGA	Thermogravimetric Analysis
Virgin Material	PP resin that will for the first time be converted to a plastic product (no thermal pre-treatment)
wt%	Weight Percentage

DISCLAIMER

"RecyClass is a non-profit, cross-industry initiative advancing recyclability, bringing transparency to the origin of plastic waste and establishing a harmonized approach toward recycled plastic calculation & traceability in Europe. The Recyclability Evaluation Protocols will promote recyclability by encouraging the industry to test new plastic technologies, materials or products, providing recommendations on improving their recyclability before market launch.

The Recyclability Evaluation Protocols are freely available to download in the *RecyClass website*. Companies developing new plastic packaging concepts are encouraged to use them to self-assess the impact of their solutions on recyclability and highlight potential issues. **However, compliance with a Recyclability Evaluation Protocol is not a replacement for an official assessment and may not be used as a marketing tool.** The RecyClass Steering Board, following the recommendations of the Technical Committees, will decide on the compatibility of the innovation with recycling according to the evaluation results, granting a Recyclability Approval Letter to the Applicant.

All tests must follow the Evaluation Protocols recommended by the RecyClass Technical Committees and be conducted by an independent laboratory recognised by RecyClass which has no legal affiliation to the applicant.

More information is reported in the RecyClass Internal Procedures available on the *RecyClass website.*"

RecyClass

1. INTRODUCTION AND PURPOSE OF THE PROTOCOL

The "RecyClass[1] Recyclability Evaluation Protocol for PP Films" referred to in this document as "The Protocol" describes the methodology the applicant must follow at a laboratory scale to determine if a plastic packaging innovation is compatible with the post-consumer PP film recycling stream. The Protocol targets companies responsible for introducing a packaging product onto the market. The applicant shall proceed with the Protocol as established in the Assessment Process for Applicants of Recyclability Evaluation in the RecyClass Internal Procedures[2] and "RecyClass Recyclability Approval Quality Management & Procedures document[3].

The Protocol analyses whether an innovation will undergo the necessary pre-treatment, extrusion and conversion steps described in this methodology at a laboratory scale without negatively impacting the recycling process and the quality of the recycled PP material, tested in a PP flexible application as a reference. It aims to prove the recyclability[4] of plastics packaging while encouraging innovation in the PP film market. The overall goal is to maintain the protection of packaged goods and their marketing display functions without obstructing the proper functioning of the PP film recycling process and ensuring the highest possible quality of the recycled polymer, tested in a PP film application as a benchmark.

This document provides guidance on the testing methodology that shall be followed, including benchmark recommendations to guide the interpretation of the results.

PP film terminology, as it is used in this document, is defined as a flexible plastic whose form changes depending on whether it is filled with a substance or not. It has a thickness of up to 250 µm and at least 90 % of its weight is plastic, with up to 10 % of closely bonded or impregnated material. Printing, coatings, or plastic fillers can classify as closely bonded or impregnated materials. It includes blown, cast and biaxially oriented PP films.

Please note that all units in this protocol are expressed following the International System of Units[5], from the Bureau International des Poids et Mesures.

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1 RecyClass assesses the recyclability of a plastic package providing a ranking from A to F. RecyClass also provides specific indications and recommendations on how to improve packaging design to fit current recycling technologies. More information at https://recyclass.eu/

[2] *RecyClass Internal Procedures*

[3] *RecyClass Recyclability Approval Quality Management & Procedures*

4 Recyclability definition according to PRE & APR: Plastics must meet four conditions for a product to be considered recyclable: 1. The product must be made with a plastic that is collected for recycling, has market value and/or is supported by a legislatively mandated program. 2. The product must be sorted and aggregated into defined streams for recycling processes. 3. The product can be processed and reclaimed/recycled with commercial recycling processes. 4. The recycled plastic becomes a raw material that is used in the production of new products.

[5] SI Brochure - BIPM

2. SCOPE OF THE PROTOCOL

The scope of the Protocol covers any innovation introduced to the existing packaging solutions for PP films. Before initiating the evaluation, the applicant shall review the Design for Recycling Guidelines for clear PP films or coloured PP films[6] to confirm that the PP innovation film is compatible with the recycling stream for PP films.

The following packaging solutions and/or innovations are covered by the scope of this Protocol:

1. Non-PP layers and coatings, including PE, nylon, EVOH, and others not specified.
2. Rigid PP and non-PP attachments to the PP film tested packaging.
3. Mineral fillers and other additives that alter the density of PP film.
4. Paper and filmic labels
5. Inks and pigments, including direct, reverse, laminated, and other printing technologies.
6. Compatibilizers and other additives (impact improvers, nucleating agents, etc.) not otherwise specified.
7. Adhesives.

Following the RecyClass Recyclability Methodology[7], packaging containing aluminium layer, metal, bio/oxo-degradable plastics, black carbon surface, as well as PVC and PVDC and PET layers are considered as disqualifying criteria for PP films recyclability. Consequently, packaging containing any of these features does not fall under the scope of this Protocol.

3. DISCLAIMER

A PP film recycling process is not yet established. The Protocol aims to represent as accurately as possible how the PP recycling process should work at an industrial scale to allow PP films to get recycled in high quality products. RecyClass Technical Committee reserves the right for further testing, if necessary, to issue a final opinion on the recyclability of the tested innovation. The Recyclability Evaluation Protocol establish some benchmark recommendations to guide the decision-making process. However, only some of the properties listed in the protocol are provided with a benchmark recommendation, given that the evaluation is also based on the technical expertise of the Technical Committee (TC).

Within RecyClass, “easy-to-empty” and “easy-to-access” indexes are essential factors when considering the recyclability of a package. Washing operations at state-of-the-art PP film mechanical recycling facility uses mild conditions and no detergents or strong chemicals. Consequently, any food or product residue constitutes an impurity for the recycling stream. RecyClass encourages testing to verify that the package is “easy-to-empty” and therefore ensures the minimum amount of residue material at the end of its useful life. Nonetheless, this factor is beyond the scope of this Protocol.

4. LABORATORY TESTING METHODOLOGY

This methodology aims to reproduce the recycling process at a laboratory scale to determine the suitability of an innovation for the PP film recycling stream. The methodology described below shall be followed precisely and any modifications or problems during the testing phase must be noted. Additional tests may be requested by the PO films

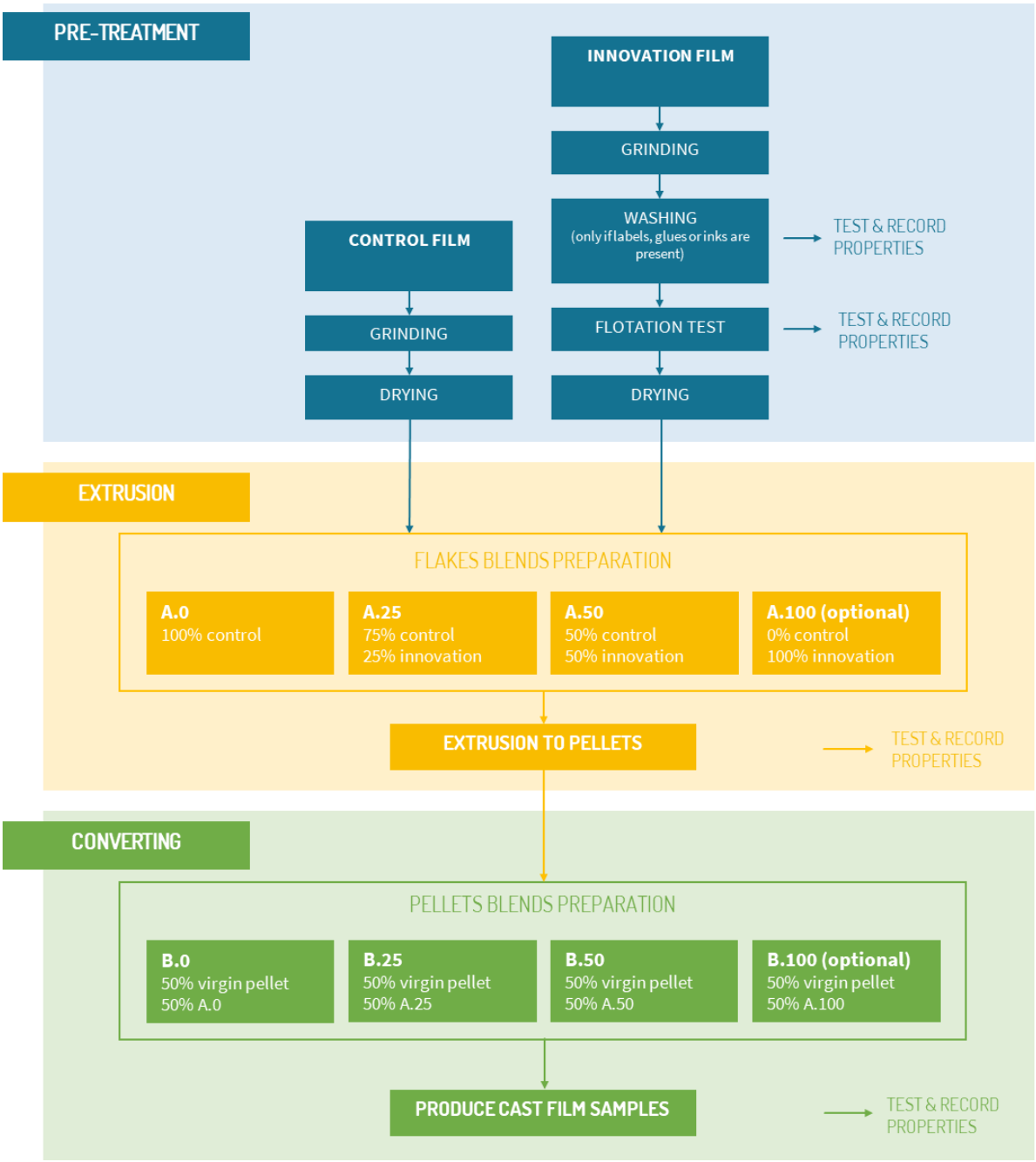
6 Design for Recycling Guidelines

7 RecyClass Methodology

TC for specific cases requiring particular attention. A Laboratory Evaluation Report compiling objectively all the results obtained shall be prepared to report to the RecyClass PO films Technical Committee (TC), which will interpret the final results. Any remarks during the laboratory tests described in the Protocol shall also be noted down.

See below in Figure 1 a diagram describing methodology.

Figure 1: Methodology diagram



4.1 CONTROL SAMPLE SELECTION

The control PP film sample for the purpose of performing the Protocol can be selected:

- **Option 1:** If there is a PP film known to be recyclable, consisting of the same base PP resin as the Innovation, except/apart from the specific ingredient/feature being evaluated, it can be selected as the control for this Protocol, with the approval of the RecyClass PO films TC.
- **Option 2:** The applicant can select a PP virgin with the typical MFI for cast film applications and copolymers structure as the innovation article, ± 10 % MFI and ± 0.005 density can be used as control for this Protocol, upon the approval of the RecyClass PO Films TC (see Annex 1). To obtain the control, the selected PP resin must be extruded once (preferably into a film), following the recommendations for extrusion present in this protocol, in order to simulate the same thermal history that an actual packaging may haveThe same physical form as the innovative material should be preferred.

These options will be used to make both the control flakes and the blends with innovation flakes that will contain the innovative feature(s) (barrier, adhesive, additive, coating, label, multilayer resin, etc.) for the recyclability study.

Since control material is used as reference to evaluate the impact of the innovation, A.0 and B.0 (see Figure 1) needs to fulfil some minimum requirements to make the recyclability evaluation valid. RecyClass recognised testing facilities are aware of these minimum requirements and will inform both the Applicant and RecyClass in case of deviations. Quick analyses can be performed before to start the recyclability test to ensure suitability of the control material (MFI, FTIR, oven test, …)

For the purpose of the tests, the amount of material that the Applicant should provide will depend upon the equipment and scale used in each laboratory. Usually, at least 10 kg of innovation material (as packaging) and 25 kg of control material (as packaging) will be requested to prepare blends of at least 8 kg. More innovation sample could be requested in case optional tests are required by the RecyClass PO Films TC. It is worth pointing out that the protocol should be used to test innovations as specific parts of the packaging, meaning that all the decorations or elements of the packaging do not need to be present. The objective is to evaluate the impact of a specific innovation on the recyclability of PP Films. Note that complete packaging (with labels, decoration, closures, etc.) can also be assessed under this present protocol.

4.2 VIRGIN SAMPLE SELECTION

The virgin PP sample to be used in this Protocol can be selected from the PP resins listed in Annex 1 or proposed by the applicant to match the target application. The choice of the virgin must be approved by the PO films TC and used as it is (i.e. without applying any thermal pre-treatment). In the case the control material selected has an MFI out of 3 to 8 g/10min, the virgin material should be selected as homopolymer : copolymer blend that can lead to an MFI for B.25 and B.50 blends in the range of 3 to 8 g/10min (230°C, 2.16 kg). A 90/10 homopolymer : copolymer ratio would be preferred.

5. LABORATORY TEST PROCEDURES

5.1 PRE-TREATMENT STEPS

5.1.1 GRINDING

Control (if provided as film) and innovation samples are separately ground in order to fit the feeding hopper of a standard laboratory extruder. In case the control is provided in form of pellets, only the innovation sample has to be ground.

Procedure:

- Report the mass of each sample before grinding as m_0.
- Grind separately control and innovation sample to flakes of 3 to 15 mm.
- Store in separate containers.
- Report the mass of each sample after grinding as m_1.

5.1.2 WASHING

Control and innovation samples are separately washed to test the impact on wet washing operations. Washing shall only be performed if paper, labels or surface printing is present in innovative films. If none of those are present, go directly to step 5.1.3.

The following procedures must be used for innovation sample only.

Procedure:

- Prepare the wash container at a 1:24 ratio (1 g flakes vs 24 ml water) with tap water at a room temperature (+/- 20 – 25 °C). No added detergents or caustic soda.
- Wash each sample separately at a 1:24 ratio (1 g flakes vs 24 ml water) at 1,000 rpm for 10 minutes.
- Rinse each sample at the same ratio with 500 rpm for 5 minutes.
- Pour the liquid-flake mix over the de-watering screen and save the wash water.
- Take photos at each step.

Save the washing and rinsing water separately for visual observation. Record the presence of suspended particles or fibers within the water as well as any water coloration. Visually check and record whether the glue has been diluted after the rinsing or whether it remains attached to film flakes.

5.1.3 FLOTATION TEST

The flotation test will determine if the flakes can be separated by density in the float/sink tank used in the recycling operation.

The following procedure must be used for innovation sample only.

Procedure:

- Homogenise the flakes collected after washing and save 50g of washed flakes to be used to estimate the efficiency of the density separation.
- Pour the washed flakes in a tank of water filled with water at a 1:24 ratio at a room temperature.
- Stir at 500 rpm for 10 minutes.

- Stop the stirrer and allow the water to rest for 2 minutes.
- Remove the Stirrer from the tank.
- Collect all particles that float on the surface with a sieve.
- Collect separately the particles that sink.

The efficiency of the sink/float separation should be measured using the 50 g of flakes of innovative samples saved before density separation and a graduated beaker filled with tap water, as described by the following procedure.

Procedure:

- Fill a 2 L graduated beaker with 1.5 L ml of tap water (pH between 7 and 8).
- Boil the water for 10 minutes, and then cool at room temperature.
- Transfer 1 L of water to a graduated beaker.
- Put the innovative sample in the water and stir at 500 rpm for 2 minutes.
- Stop the stirrer and allow the water to rest for 2 minutes.
- Take photo of the beaker.
- Remove all particles that float on the surface with a sieve.
- Take photos of the floating and sinking fractions separately.
- Save the water for visual evaluation.
- Dry the floating fraction for 1 hour at 80 °C in a bed desiccant or 3 hours at 65 °C with air.
- Cool to room temperature, weigh and record the weight of the float fraction.
- Calculate the test efficiency as:

$$\eta = \frac{W_F}{W_I} = \frac{(W_I - W_S)}{W_I} \; x \; 100 \; [\%]$$

Where:

η: Test efficiency

W_F : weight of floating fraction

W_S : weight of sinking fraction

W_I : weight of innovative sample

5.1.4 DRYING

Reduce the flake moisture at 60°C without applying vacuum to release surface moisture to less than 1%.

Procedure:

- Heat the oven to 60 °C.
- Divide the flakes evenly between at least 4 dishes. The dishes are sequentially numbered.
- Weigh the different dishes with the control or innovation material before introducing them in the oven.
- As soon as the oven has reached 60 °C, the flakes are added to the oven without applying vacuum until 1% moisture content is reached.
- Report the mass of each sample after drying as m_4.
- Record the moisture content.

According to the mass measured at the different steps of the pre-treatment, fill the table in Annex 2 and determine the pre-treatment yield for both control and innovation as follow:

$$\eta_{PT} = \frac{m_4}{m_0}$$

Where:

ɲ: Pre-treatment yield

m_0 : mass of sample before grinding

m_4 : mass of sample after drying

5.2 EXTRUSION

5.2.1 FLAKE BLENDS PREPARATION

For each sample obtained, to evaluate and record the properties of innovation PP films against control as laid out in this Protocol, a set of flake blends is prepared as described in Table 1. Blends shall be produced once the control and innovation films have separately gone through all pre-treatment steps described above.

Keep separated the control and innovation flakes obtained following the previous steps, and air dry for 24 hours in an ambient environment. Then, according to the values reported in Table 1 prepare three different dry blends with 100 % control (and 0 % innovation), 75 % control – 25 % innovation, and 50 % control – 50 % innovation, and tag them respectively as A.0, A.25 and A.50.

For the purpose of the tests the Applicant should provide enough innovation and control materials which allows for the blend preparations (cf. section 4.1.).

5.2.2 FLAKE BLENDS COMPOSITION

Three different blends 0 %, 25 % and 50 % of innovation film (optionally 100 % innovation film), will be prepared as described in Table 1.

Depending on the application, the TC can also ask the Applicant also to perform the tests with a sample of 100 % innovation (i.e., A.100, by replacing the test with A.25).

Table 1: Flake blends composition for the production of pellets

BLEND	COMPOSITION	% CONTROL FILM	% INNOVATION FILM
A.0	100 % Control film	100	0
A.25	75 % Control film 25 % Innovation film	75	25
A.50	50 % Control film 50 % Innovation film	50	50
OPTIONAL A.100	100 % Innovation film	0	100

The different blends are prepared by manually mixing the flakes in large-capacity containers and following the procedure below:

- Introduce the specified masses of the innovation flakes and control flakes into the mixing container.

- Close the container and agitate for 2 minutes in multiple orientations so as to simulate the motion of a tumble-mixer. Ensure that the container is filled to no more than 60 % of its volume and that the mixing operation yields a suitably homogeneous flake blend.
- If the total quantity of material cannot be processed in a single batch, perform multiple mixing operations with smaller quantities rather than mixing all the material at once.
- Once mixing is complete, verify that homogeneity has been achieved. If uniformity is not confirmed, repeat the mixing cycle until the flake blend is homogeneous.

5.2.3 PELLET PRODUCTION

If extrusion is not carried out directly after the previous drying stage, the flakes need to be dried under the same conditions with hot air. The level of moisture must be below 1 %. The flakes are extruded using a co-rotative twin-screw extruder at a melt temperature of 230 °C. The extrudate will be filtered with a 110 µm filter. When needed, for low bulk density materials, a densification step can be used prior extrusion to effectively feed the extruder, under the agreement of the RecyClass PO Films Technical Committee. Densification should be done following the procedure FPE-P-04 developed by APR[8].

Control flake sample A.0 has to be extruded first. Further size reduction before extrusion is acceptable if needed to allow good feeding of the material into the extruder. Nevertheless, the flake size should be kept constant between all samples.

The extruder has to be cleaned before starting the extrusion process. This involves pulling the screws out of the barrel and then mechanically cleaning them with brass brushes until they reach a glossy finish. The barrel also has to be mechanically cleaned with round brass brushes from the mandrel to the run-out zone.

Procedure:

- If a densification step was performed, report the equipment used, as well as condition and procedures followed.
- Any agglomeration of flakes during drying must be reported.
- Extrude at a preferred melt temperature from 230 ± 5°C with a suggested filtration screen at 110 µm. If the range is not optimal, record temperature and state reasons for alteration. Melt residence time should be between 1 and 6 minutes.
- Recommended throughput is between 5 and 10 kg/h and rotation speed between 100 and 200 rpm.
- Extrusion run time per variable, no less than 30 minutes.
- Extrusion load > 50% (if not possible, to be reported).
- Pellets should be between 1 and 5 mm diameter and length.
- Record a 10 second video of the extrusion for each blend, to allow observations of fumes, or volatiles. The video should be centred on the dye.
- Torque and pressure over time must be monitored and reported. If a continuous monitoring is not possible, 5 data points should be measured within the 30 min extrusion. The starting point must be considered 1 minute after material started to flow out of the extruder.
- Verify that the average pressure is less than 25 % superior to the control over a stable 15 minutes run time.

Record the resulting observations during extrusion in Table 2, and pellets properties in Table 3. The processing conditions used for all the samples must be identical. A small amount of each sample (50 g) will be retained for the RecyClass PO Films TC and the Applicant. The extruded pellets will be tested for pellet properties characterisation

[8] APR PE Film Practices FPE-P-00

(Table 3). The pellets of the test samples will be compared with the pellets of the control sample. All pellets should meet the requirements reported in Table 3.

5.2.3.1 EXTRUSION PROCESS OBSERVATIONS

Table 2: Extrusion process observations & monitoring

ASSESSMENT	STANDARD	BENCHMARK RECOMMENDATION
Odours	**0:** No noticeable odours, even right next to the extruder. **1:** Slight odour near the extruder, noticeable but not a problem for operators. **2:** Strong odour in the work area, sometimes needing ventilation but still manageable. **3:** Very strong odour making it uncomfortable, forcing operators to move away from the extruder or use protective equipment	0 or 1 If odours present, report more details Report with pictures in comparison with A.0
Fumes	**0:** No visible fumes observed near the extruder. **1:** Slight fumes observed, disappearing quickly. **2:** Moderate fumes, clearly visible and staying in the air for some time. **3:** Heavy fumes, very visible and dense, making it hard for operators to stay near the extruder.	0 or 1 If 2 or 3, report with pictures Report with pictures in comparison with A.0
Die build-ups	Visual inspection	No die build-up
Filtration (110 µm)	Visual inspection. In case of presence of build-ups, an FTIR analysis is recommended to identify the origin of the deposit.	No build-up on the screen
Average Pressure (MPa)	Average pressure after extruding through 110 µm filter for the stable 15 minutes run time, compared to 100 % control	No more than a 25 % increase to A.0
Pressure Variation (MPa)	$(\Delta P_{25\text{-}30minutes} - \Delta P_{5\ first\ minutes})$	No increase higher than 25 % compared to start

5.2.3.2 PELLET PROPERTIES CHARACTERISATION

Table 3: Pellet properties characterisation

ASSESSMENT	STANDARD	BENCHMARK RECOMMENDATION
Bulk Density (kg/m^3)	ISO 60 or EN 15345	Superior to 480 kg/m^3
Density (kg/m^3)	ISO 1183-1	A.25 and A.50 (and eventually A.100) lower than 0.920 g/cm^3 for natural films and lower than 0.950 g/cm^3 for coloured films
Melt Index (g/10 min)	ISO 1133-1 (230 °C/2,16 kg)	A.25 and A.50 (and eventually A.100) less than 15 % deviation in respect to A.0
Volatiles (wt%) before and after extrusion	Heat 10 g blends (before extrusion) and pellets (after extrusion) exposed to 160 °C for 10 minutes	± 0.1 % for A.25 and A.50 (and eventually A.100) respect to A.0 measured on cold material.
Ash content (wt%)	ISO 3451-1 (muffle) up to 750 °C	A.50 lower than 1 wt% (A.100 lower than 2 wt%)
Moisture content (wt%)	Moisture analyser or EN ISO 15512 or equivalent	< 0,1 wt%
Melt Temperatures (°C)	ISO 11357-3 (Heat-cool-heat cycle under N_2 at 10 °C/min from 0 °C to 250 °C with 1 minute of isotherm between each ramp)	Melt temperature second heating <170 °C
Impurities (unmolten particles)	Visual inspection	Record
Surface appearance	Visual inspection	Record
PE (%), PE-Comonomers in PP are not counted	Differential Scanning Calorimetry or Spectroscopic measurement via FTIR (method under development)	No more than 2.5 % for A.50 (and eventually no more than 5 % for A.100)
Reflection Colour	(L*, a*, b*) + ΔE Reflectance mode, D65, 8-10°	For natural stream: ΔE < 5

5.3 CONVERSION

Prior the recyclability assessment, the RecyClass PO Films TC will decide the process to be used for conversion according to the highest value recyclate application for the innovation. In the present case, all innovative PP Films recyclate will be converted via cast film extrusion.

For cast films production, three blends of innovation and control pellets will be produced aiming to assess different innovation concentration in the recycling stream, as described below.

5.3.1 PELLET BLENDS PREPARATION

Once PP pellets have been produced and tested, three additional blends of at 50 % virgin – 50 % blend "A.X" (X being 0, 25,50 or 100) shall be produced for the converting tests. Keep separated the pellet samples previously produced and dry them to a residual moisture level inferior to 0.1 wt%. Then according to the values reported in Table 4 prepare three different blends with 0% innovation (50 % virgin and 50 % A.0 pellets), 12.5% innovation (50 % virgin and 50 % A.25 pellets), and 25 % innovation (50 % virgin and 50 % A.50 pellets), and tag them as samples B.0, B.25 and B.50 respectively.

Depending on the application and its market penetration, the TC can ask the Applicant also to perform the tests with a sample of 50 % virgin and 50 % innovation (i.e. B.100, by replacing the test with B.25).

For the purpose of the tests the Applicant should provide enough virgin materials which allows for the blend preparations. The laboratory carrying out the Protocol testing can define the amounts according to their best knowledge.

5.3.2 PELLET BLENDS COMPOSITION

Three different blends of cast film at 50 % virgin pellet – 50 % Blend A shall be produced as described in Table 4. Blends will be composed of 0 %, 12.5 % and 25 % content (eventually ending at 50 %) based on the weight of the initial innovation film.

Table 4: Pellet blends composition for the application tests

BLEND	COMPOSITION	% VIRGIN RESIN	EFFECTIVE % CONTROL FILM	EFFECTIVE % INNOVATION FILM
B.0	50 % Virgin Pellet 50 % A.0	50	50	0
B.25	50 % Virgin Pellet 50 % A.25	50	37.5	12.5
B.50	50 % Virgin Pellet 50 % A.50	50	25	25
OPTIONAL B.100	50 % Virgin Pellet 50 % A.100	50	0	50

5.3.3 CAST FILM PRODUCTION

Procedure:

- Report the equipment and the die used.
- Produce cast film at a melt temperature of 240 ± 10°C, a die gap of 300 ± 100 µm, chill roll temperature of 50 ± 10 °C and a thickness of 35 ± 5 µm
- Report the melt temperature and the chill roll temperature profiles during the 30-min run time.
- Film samples must be used as produced and stored at least 16 hours at (23 ± 2) °C and (50 ± 10) %rH before performing characterisation.

- Record cast film properties in Table 5, including information regarding structure, holes, and stability of the cast film.
- For “Gels & Specks” evaluation, refer to the Annex 3 “PP Film Surface Impression Evaluation and Impurity Visual Inspection”. If gels are large enough, an IR analysis is requested on these gels.
- Report the morphology of the gels by reporting microscopy pictures of some of the most represented defects.

5.3.3.1 CAST FILM PROPERTIES CHARACTERISATION

Table 5: Cast film properties characterisation

ASSESSMENT	STANDARDS	BENCHMARK RECOMMENDATION	
Thickness (µm)	ISO 4593; DIN 53370	35 µm	
Tear Strength (TD**) (N)	DIN EN ISO 6383-1 (trouser tear method)	No more than a 25 % decrease to B.0	
Tear Strength (MD***) (N)			
Tensile Stress at Yield (TD) (MPa) (if observed and if not take it at 10 % elongation)	DIN EN ISO 527-3 (Type 2 samples, L0 = 50 mm, test speed = 100 mm.min^{-1})		
Tensile Stress at Yield (MD) (MPa) (if observed and if not take it at 10 % elongation)			
Elongation at Yield (TD) (%)			
Elongation at Yield (MD) (%)			
Tensile Stress at Break (TD) (MPa)			
Tensile Stress at Break (MD) (MPa)			
Elongation at Break (TD) (%)			
Elongation at Break (MD) (%)			
Dart Impact (g)	ISO 7765-1 (report used mass)		
Puncture Resistance (N)	DIN EN 14477		
Haze (%)	DIN EN ISO 14782	Record. Increase of haze will lower the visual aspects	
Gels and Specks (Annex 3)	Annex 3 procedures	Record the amount and aspect of each gels, specks, fisheyes and holes separately. Add microscope pictures of them to show size, shape, distribution.	< 100 Gels & Specks
Surface Appearance		Record. Limit the end use application.	

**Film testing results are minimum conditions. Historical data over time may require adjustments for specification change and requirements for specific applications. **TD: transverse direction ***MD: machine direction*

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DOCUMENT VERSION HISTORY

VERSION	PUBLICATION DATE	REVISION NOTES
1.0	January 2021	Recyclability Evaluation Protocol for PP Films release
1.1	May 2021	Major modifications about procedure, wording & template
2.0	January 2022	Revised wording and removal of some testing
2.1	August 2022	Mandatory washing and floatation step for control sample removed Wording for sample quantity requested for testing Temperature recommendations for extrusion are now melt temperatures
3.0	January 2023	Addition minimum requirements for control material Addition pre-treatment yield and Annex 2 Addition of densification step prior extrusion, and addition of extrusion procedures Removal of TGA analysis for ash content Modification of the gels & specks evaluation with addition of Annex 3
4.0	January 2024	Modification of conditions for virgin material selection Modification grinding and drying conditions Removal of washing as mandatory for control material Addition of reference to APR densification procedure Removal of bulk and pellet size characterisation Clarification on colour, volatiles, and gas content characterisations Decrease of extrusion and cast film melt temperatures Harmonisation mechanical characterisation parameters Microscopy mandatory for gels/specks analysis
5.0	January 2025	Revised wording Addition of video recording during extrusion Clarification on odours, fumes, dye build ups characterisations Addition of bulk density / Removal gas content measurement Modification volatiles characterisation method Clarification tensile stress at yield Peer reviewed version
5.1	January 2026	Template modification Addition detailed procedure cast film production Addition of guidance on blends production Modifications of standard method for volatiles, moisture content and tear strength Update of Annex 3

Identification of versions

v.X - Structural modification impacting the protocol.

v.X.Y – Updates to testing conditions. integration of new tests, technologies or analytical methods.

v.X.Y.Z - Editorial modifications not impacting the content of the Protocol.

ANNEX I – CONTROL SAMPLES SELECTION

Application	Copolymer structures	Grade	MFR at 230 °C, g/10 min	Melting temperature, °C	Flexural Modulus ISO178 (MPa), measured on IM specimen 23 °C / 50 %RH
Cast film	Homopolymer	HD204CF	8	164	1350
Cast film	Homopolymer	HD601CF	8	164	1400
Cast film	Heterophasic copolymer	BC918CF	3	168	1400
Cast Film	Random Copolymer	RD204CF	8	150	1100
BOPP	Terpolymer	TD310BF	6	130	n.a.
BOPP	Homopolymer	HC101BF	3.2	161	1350

For control material, a mono-material (mono-grade) approach remains mandatory, selecting a grade with properties as close as possible from the innovation. In the case the control material selected has an MFI out of 3 to 8 g/10min, the virgin material should be selected as homopolymer : copolymer blend that can lead to an MFI for B.25 and B.50 blends in the range of 3 to 8 g/10min (230°C, 2.16 kg).

ANNEX II – MASS BALANCE PRE-TREATMENT

MASS (g)	CONTROL SAMPLE	INNOVATION SAMPLE
Before grinding: m_0		
After grinding: m_1		
After drying: m_4		
Pre-treatment yield: η_{PT}		

ANNEX III – PP FILM SURFACE IMPRESSION EVALUATION AND IMPURITY VISUAL INSPECTION

The film visual characterisation must be done according to the following procedures:

- Use the 35 µm films B.0, B.25 and B.50 (optionally B.100) for the visual characterisation.
- A first count must be done by human naked eyes, under the following conditions:
    - 5 squares of 100 cm^2 must be defined for each film.
    - Only defects (gels, contamination, structural defects, holes) bigger than 200 µm should be counted, which corresponds to any defect visible by a naked human eye at 30 cm distance from the sample.
    - Before counting the gels, use a brush to remove any surface impurities and dust.
    - This first count of defects should take between 5 and 7 minutes per 100 cm^2 surfaces.
    - The samples should be taped on a clean window to receive light by transmission with a neutral background. No additional tools or contrast enhancers are required.
    - Report the number of each category of defect including standard deviations based on the 5 squares of 100 cm^2. The reported number must be for an average 100 cm^2 surface.
    - Comment on the nature or appearance of the defects if possible.
- If the number of defects (gels, contamination, structural defects or holes) are not within the preferred range (see below) and if less than 200 defects were identified per 100 cm^2, a microscopy check must be done to ensure that only defects bigger than 200 µm are counted.
- Select the 3 most representatives 100 cm^2 squares of each film for microscopy screening. Each square must be divided into 9 smaller squares (3.3x3.3 cm^2) by drawing 2 horizontal and 2 vertical lines.
- The following conditions for the microscopy screening are recommended: Magnification of 20x (recommended to be able to see about 100 to 150 mm^2 per picture), transmitted light, use of a software to analyse microscope pictures on a wide screen.
- One picture must be analysed per each smaller square (meaning 9 pictures per 100 cm^2) and the number of defects counted. This must be repeated for the 3 selected 100 cm^2 square.
- Use an image software to evaluate if defects are bigger than 200 µm (at least one dimension superior to 200 µm).
- Take few representative microscopy images of the observed defects and add them to the report, including the scale on the images.
- The average number of defects counted as well as the standard deviation must then be re-scaled to give a number for a 10*10 cm^2 (10 000 mm^2).
- Report the number of each category of defect including standard deviations based on the 3 squares of 100 cm^2. The reported number must be for an average 100 cm^2 surface.

8.

PP Film Surface Impression Evaluation and Impurity Visual Inspection

Defect description	Rating			
Texture	Very Smooth	Smooth	Rough	Very rough
Gels (>200 µm)	: Defined as particle in the film matrix not blended with the matrix and often acting as a miniature lens.			
	< 50	50-100	> 100	for 100 cm^2

Contamination (>200 µm)	: Defined as any particle in or on the film matrix affecting irradiated light differently than the matrix (dirt, oxidized additives or material, catalyst residues, solid particles, metallic particles, undispersed pigments or additives, etc.)			
	< 10	10-15	> 15	for 100 cm^2
Structural defects	: Defined as visual deviation not caused by gels or contaminations, for example, air bubbles, wrinkles, die lines, sharkskin, arrowheads.			
	≤ 3	4-6	> 6	for 500 cm^2
Holes	: Defined as tears in the blown film bubble starting at, or caused by, gels, specks or structural defect.			
	0	1 or more		

Preferred range	Limited	Non-acceptable

With naked eyes				
Defect description	B.0	B.25	B.50	B.100
Texture				
Gels (>200 µm)				
Contamination (>200 µm)				
Structural defects				
Holes				

Report here the exact amount of defects observed, as well as standard deviations.

With microscopy (if applicable)				
Defect description	B.0	B.25	B.50	B.100
Texture				
Gels (>200 µm)				
Contamination (>200 µm)				
Structural defects				
Holes				

Report here the exact amount of defects observed, as well as standard deviations.

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