RecyClass

RECYCLABILITY EVALUATION PROTOCOL

FOR PP FILMS

STANDARD LABORATORY PRACTICE

VERSION 2.0

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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 B.0 pellets
B.25	Film made of 50% of virgin pellets and 50% of B.25 pellets
B.50	Film made of 50% of virgin pellets and 50% of B.50 pellets
B.100	Film made of 50% of virgin pellets and 50% of B.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	Thermo Gravimetrical 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 an initiative aiming at enhancing and evaluating the recyclability of plastic packaging through a technical perspective. The Recyclability Evaluation Protocols will promote recyclability by encouraging industry to test new plastic technologies, materials or product before market launch and giving advice and recommendations to the companies.

The Recyclability Evaluation Protocols are available for download in the RecyClass websites. Companies providing plastic packaging concepts are encouraged to use them to self-assess the impact of their solutions on recyclability and highlight potential issues. **However, compliance to a Recyclability Evaluation Protocol is not a replacement for an official assessment and may not be used as a marketing tool.**

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

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

RecyClass

1. INTRODUCTION AND PURPOSE OF THE PROTOCOL

The "Recyclability Evaluation Protocol for PP Films" referred to in this document as "The Protocol" describes the methodology that must be followed by the applicant at a laboratory scale in order 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 into the market. The applicant shall proceed with the Protocol as established in the Assessment Process for Applicants of Recyclability Evaluation in the "RecyClass[1] Internal Procedures".

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. It aims to guarantee recyclability[2] 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.

This document provides guidance on the tests 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 85% of its weight is plastic, with up to 15% 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.

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 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.

2. SCOPE OF THE PROTOCOL

The scope of the Protocol covers any innovation introduced to the existing packaging solutions for PP films. Prior to initiating the evaluation, the applicant shall review the Design for Recycling Guidelines for clear PP films or coloured PP films[1] in order to confirm that the PP innovation film is compatible with these requirements.

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 the PP film.
4. Paper and PP labels
5. Inks and pigments, including direct, reverse, laminated, and other printing technologies.
6. Compatibilizers and other additives otherwise not specified.
7. Adhesives in laminated mono-PP-structures

Following RecyClass recyclability methodology[2], packaging containing aluminium, metal, degradable plastics, black carbon surface, as well as PVC and PVDC and PET layers are considered disqualified for PP films recyclability. By consequence, packaging containing one of these features do 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 an additional opinion on the recyclability of the tested packaging.

Within RecyClass, “easy-to-empty” and “easy-to-access” indexes are important factors when considering the recyclability of a package. Washing operation at a recycling facility uses mild conditions, no detergents nor strong chemicals. Consequently, any food 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 leftover material at the end of its useful life. Nonetheless, this factor is beyond the scope of this Protocol.

4. LABORATORY TEST METHODOLOGY

This methodology aims to reproduce the recycling process at 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 must be noted during the testing phase. A Lab Evaluation Report compiling all the results obtained shall be prepared to report to the RecyClass PO films Technical Committee which will interpret the final results. Any remarks during the laboratory tests described in the Protocol shall be also noted down.

See below in Figure 1 a diagram where the flow of the methodology is described.

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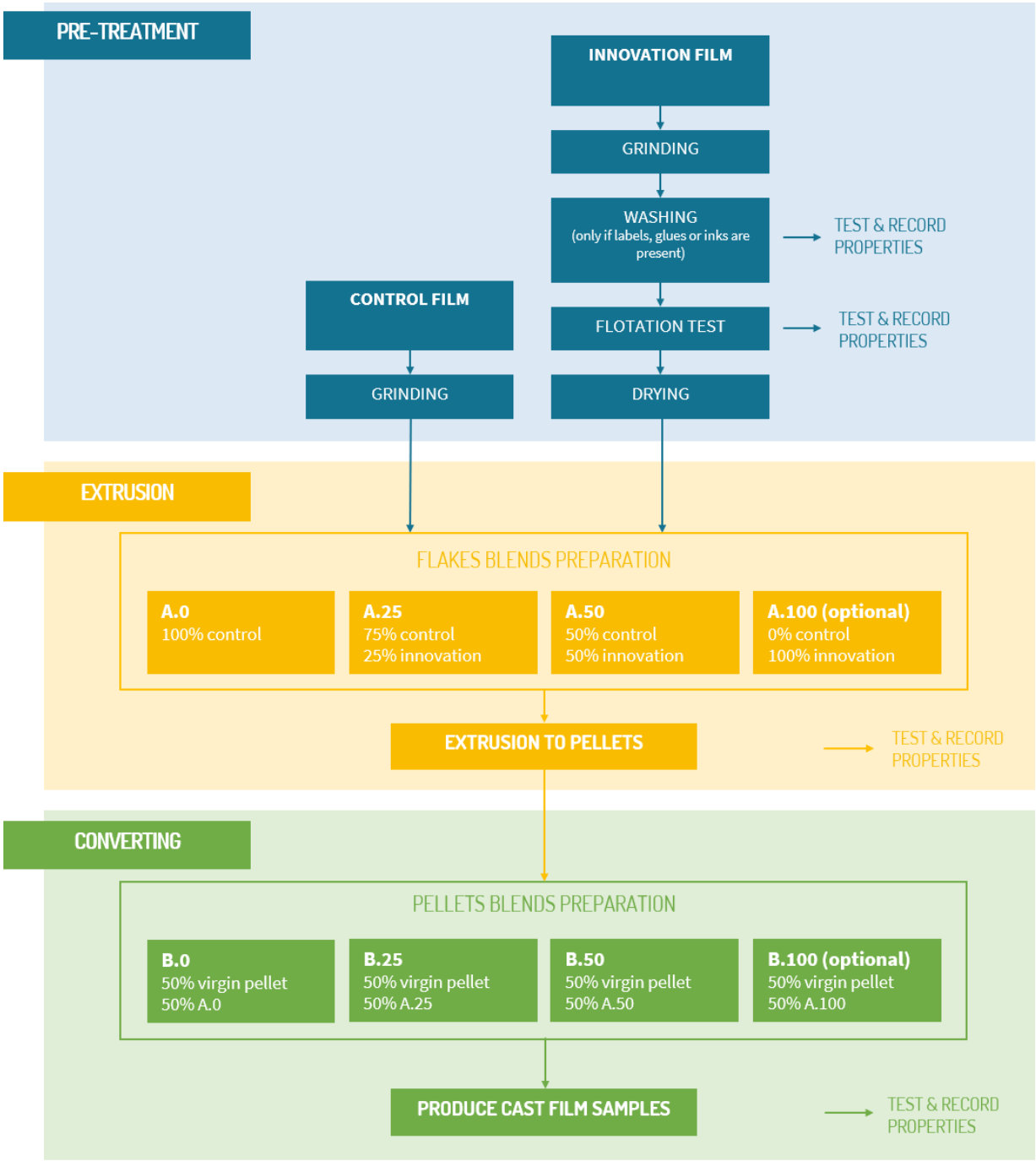
1 Design for Recycling Guidelines

[2] RecyClass Methodology

Figure 1: Methodology Diagram



4.1 CONTROL SAMPLE SELECTION

The control sample for use following the Protocol can be selected by:

- **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, with/upon the approval of the RecyClass PO films Technical Committee (see Annex 1). The selected PP resin must be extruded at 220°C to obtain the control sample.

These options are to 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 …) for the recyclability study.

For the purpose of the tests the Applicant should provide at least 10 kg amount of the innovation sample (as film) and 25 kg amount of control material which allows for blend preparations of at least 5 kg each. More innovation sample could be requested in case optional tests are required by the RecyClass PO films Technical Committee. It is worth pointing out that the innovation to be tested is not limited to the film but to all its parts. Therefore, the innovation has to be submitted to the laboratory procedures with labels, adhesives, attachments, prints, seals (if any). If it can be correctly argued that labels and adhesives have no impact on the innovation, the innovation samples can be processed without the presence of labels and adhesives.

4.2 VIRGIN SAMPLE SELECTION

The virgin PP sample for use following the Protocol can be selected by the PP resins listed in the Annex and used as it is (i.e. without applying any thermal pre-treatment).

5. LABORATORY TEST PROCEDURES

5.1 PRE-TREATMENT STEPS

5.1.1 GRINDING

Control (if provided as film) and innovation samples are separately grinded in order to fit the throat of a standard laboratory extruder. In case the control is submitted under the shape of pellets, only the innovation sample has to be grinded.

Procedure:

- Grind separately control and innovation sample to flakes of 10 to 20 mm.

- Store in separate containers.

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 the innovation film. If none of those are present, go directly to step 5.1.3.

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.

- Take photos at each step.

Save the wash and rinse water separately for visual observation. Record the presence of suspended particles or fibers within the water as well as any water coloration. Check and record if the glue has been diluted after the rinsing or 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 has to be utilized for both control and innovation samples, separately.

Procedure:

- 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 tank from the magnetic stirrer.

- Collect all particles that float on the surface with a sieve.

- Collect separately the particles that sink.

Record the amount of material that float and the amount that sink in grams and %.

The efficiency of the sink/float separation should be measured using 50 g of washed flakes of innovative samples and a graduated beaker filled with tap water, as described by the following procedure. Repeat the procedure for washed and dried innovation flakes, with and without caps and labels.

Procedure:

- Fill a 1 l graduated beaker with 700 ml of tap water (pH between 7 and 8).

- Boil the water for 10 minutes, and then cool at room temperature.

- Transfer 300 ml of water in a graduated beaker.

- Put the innovative sample in the water and stir at 500 rpm for 2 minutes.

- Stop the magnetic stirrer and allow the water to rest for 2 minutes.

- Take photo of the beaker.

- Remove all particles that float at the surface with a sieve.

- Take photos of the floating and sinking fractions separately.

- Save the wash for visual evaluation.

- Dry the floating fraction for 1 hours 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.

- Repeat the procedure with 50 g of the innovation flakes without labels (if any).

- Calculate the test efficiency as (weight of sinking fraction) / (weight of innovative sample) x 100 (in %), separately for the innovation samples with and without labels.

5.1.4 DRYING

Reduce the flake moisture according to the following procedure. A minimum of 2 kg of material are necessary to proceed with the moisture content determination.

Procedure:

- Heat the oven to 60°C.

- Divide the flakes evenly between the 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 for 24 hours.

- Weight the material after 6 hours in the oven. Weight the material at the end, after 24 hours in the oven.

- Record the moisture content.

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 h at ambient air. Then, according to the values reported in Table 1 prepare three different 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.

Eventually, depending on the application and its market penetration, the TC can 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).

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.

Eventually, depending on the application and its market penetration, the TC can 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

5.2.3 PELLET PRODUCTION

Both control and innovation flakes can be mixed manually before extrusion for blends preparation. The flakes will be dried at the same conditions with a desiccant bed drying unit or with hot air and extruded using co-rotative twin-screw extrusion at temperatures of 250 °C. The extrudate will be melt filtered at 110 microns.

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. See additional information in Table 2.

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 has to be also mechanically cleaned with round brass brushes from the mandrel to the run-out zone.

Table 2: Pellet production purpose & overview

FLAKE COMPOSITIONS	KG OF BLEND REQUIRED	PURPOSE OF BLEND
A.0 100% Control flake	Per lab requirement for a 30-minute run time	All tests compared to control values
A.25 75% control with 25% innovation	Per lab requirement for a 30-minute run time	Required for comparison to control values
A.50 50% control with 50% innovation	Per lab requirement for a 30-minute run time	Required for comparison to control values
OPTIONAL **A.100** 100% innovation	Per lab requirement for a 30-minute run time	Optional, to evaluate the impact of higher concentration of innovation on recycling.

Procedure:

- Dry samples A.0, A.25 and A.50 (optionally A.100) with a bed desiccant for 1 hours at 90 °C or with hot air at 90 °C for 1 hours.

- Extrude at a preferred melt temperature from 250ºC with a suggested filtration screen at 110 µm. If the range is not optimal, record temperature and state reasons for alteration.

- Extrusion run time per variable, no less than 30 minutes.

- Extrusion load > 60%

- Maintain pressure increase to less than 25% from the control over a stable 15 minutes run time.

Record properties' results in Table 3. The processing conditions used for all the samples must be identical. If some operating conditions have to be modified for A.25 and A.50 (optionally A.100) samples, this information must be documented in the report. A small amount of each sample (50 g) will be retained for RecyClass PO films Technical Committee and the Applicant. The extruded pellets will be tested for pellet properties evaluation (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 the Table 3.

5.2.3.1 PELLET PROPERTIES EVALUATION

Table 3: Pellet properties evaluation

ASSESSMENT	RESULT	STANDARD	BENCHMARK RECOMMENDATION
Bulk Density (kg/m^3)		ISO 60 or EN 15345	Minimum 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/2,16kg)	A.25 and A.50 (and eventually A.100) less than15% deviation in respect to A.0
Ash content (wt%)		ISO 3451-1 (muffle) or ISO 11358 (TGA)	A.50 lower than 1wt% (A.100 lower than 2wt%)
Filtration (110 µm)		Visual inspection	No build-up on screen
Pellets size (average)		EN 15344:2008	Record
Pellets distribution (min – max)		EN 15344:2008	Record
Gas content (wt%)		Weight loss at 120°C	Record
Moisture content (wt%)		Moisture analyser	< 0,1 wt%
Melt Temperatures (°C)		ISO 11357-3 (Heat-cool-heat cycle at 10°/min from 0°C to 250°C with 5 minutes of isotherm between each ramp)	Melt temperature second heating <170°C
Impurities		Visual inspection	Record
Surface appearance		Visual inspection	Record
Volatiles (wt%)		10 g air-dried pellets exposed to 180°C for 10 minutes	±0,1% for A.25 and A.50 (and eventually A.100) respect to A.0
PE (%), PE-Comonomers in PP are not counted		Differential Scanning Calorimetry or Spectroscopic measurement via FTIR microscope in case of doubt	No more than 2,5% for A.50 (and eventually no more than 5% for A.100)
Reflection Colour		(L*, a*, b*)	Record
Delta Pressure (MPa)		Average Pressure after extruding through 110 microns for the stable 15 minutes run time	No more than 25% higher pressure respect to the control sample

Variation Delta Pressure (MPa)		($\Delta P_{5\ last\ minutes}$ - $\Delta P_{5\ first\ minutes}$)	No increase higher than 25% compared to start
Extrusion process		Unusual sticking, fumes, odour, and any build-up	Record

5.3 CONVERSION

Based on the obtained results, the RecyClass PO films Technical Committee will decide if the innovation presents some critical properties. On that basis, the Technical Committee reserves the right to further test the innovation. Otherwise, if the results are aligned with PP film recyclate specimens the Technical Committee and the Applicant will define the way to further test the innovation on the base of the main applications available on the market.

The Protocol aims to assess the highest value recyclate application, i.e. cast film production.

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 reported in the following paragraph.

However, in case of failed tests the RecyClass PO films Technical Committee according with the Applicant could decide to test the innovation for injection moulding. In this case, the lab will refer to the Recyclability Protocol for PP containers[1].

5.3.1 PELLET BLENDS PREPARATION

Once PP pellets have been produced and tested, three additional blends of at 50% virgin – 50% blend A shall be produced for converting tests. Keep separated the pellet samples previously produced and dry them for 10 minutes at 60°C. Then according to the values reported in following 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.

Eventually, 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 (end eventually 50%) of the initial innovation film.

[1] RecyClass Recyclability Evaluation Protocol for PP containers

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

Prepare blends for cast film extrusion as described in table 5.

Table 5: Cast film production purpose & overview

PELLET COMPOSITIONS	KG OF BLEND REQUIRED	PURPOSE OF BLEND
B.0 50% A.0 pellet and 50% Virgin pellet	Per lab requirement for a 30-minute run time	All tests compared to control values
B.25 50% A.25 pellet and 50% Virgin pellet	Per lab requirement for a 30-minute run time	Required for comparison to control values
B.50 50% A.50 pellet and 50% Virgin pellet	Per lab requirement for a 30-minute run time	Required for comparison to control values
OPTIONAL B.100 50% A.100 pellet and 50% Virgin pellet	Per lab requirement for a 30-minute run time	Optional, to evaluate the impact of higher concentration of innovation on recycling.

Procedure:

-Produce cast film at a melt temperature of 250 – 270°C, chill roll temperature of 50-60 °C and a thickness of 35 µm.

-Report the melt temperature and the chill roll temperature profiles during the 30-min run time.

-Record production properties in table 6, including information regarding structure, holes, and stability of the cast film.

5.3.3.1 CAST FILM PROPERTIES EVALUATION

Table 6: Cast film properties evaluation

ASSESSMENT	RESULTS	STANDARDS	BENCHMARK RECOMMENDATION
Thickness (µm)		ISO 4593; DIN 53370	35 µm
Tear Strength (TD**) (N)		DIN EN ISO 6383-2	
Tear Strength (MD***) (N)			
Tensile Stress at Yield (TD) (MPa) (if observed)		DIN EN ISO 527-3 (Type 2 samples, strain-rate closed to 2 min^{-1})	No more than a 25% delta decrease to B.0
Tensile Stress at Yield (MD) (MPa) (if observed)			
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	
Puncture Resistance (N)		DIN EN 14477	
Haze (%)		DIN EN ISO 14782	Record. Increase of haze will lower the visual aspects
Gels and Specks (amount)		5 samples of 100 cm^2 for a gel and specks count greater than 200 µm seen by the naked eye at 30 cm from sample. The number will be recorded but no standard required.	Record the amount and aspect. All gels & specs will weaken the film quality
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*

RecyClass

DOCUMENT VERSION HISTORY

VERSION	PUBLICATION DATE	REVISION NOTES
1.0	January 2021	Recyclability Evaluation Protocol for PP Films release
2.0	May 2021	Major modifications about procedure, wording & template
3.0	January 2022	Revised wording and removal of some testing

ANNEX I – CONTROL SAMPLES SELECTION

Application	Copolymer structures	MFR at 230°C, g/10min	Melting temperature, ° C	Flexural Modulus ISO178 (MPa), measured on IM specimen 23 °C / 50%RH
Cast film	Homopolymer	MFR 2=8	162	1400
Cast Film	Random Copolymer	MFR 2=8	150	1000
BOPP	Terpolymer	MFR 2=6	130	n.a.
BOPP	Homopolymer	MFR 2=3.2	161	n.a.

The reference granules are typically for cast PP films 70% Homopolymer and 30% Random-Copolymer and for BOPP films 90% Homopolymer and 10% Terpolymer.

RecyClass

c/o Plastic Recyclers Europe
Avenue de Broqueville 12
1150 Brussels – Belgium

Phone : +32 2 786 39 08
info@recyclass.eu

www.recyclass.eu