

RecyClass

RECYCLABILITY EVALUATION PROTOCOL

FOR LAMINATING
ADHESIVES APPLIED
ON PE FILMS

STANDARD LABORATORY PRACTICE

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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 PE film (or PE resin that has already been thermally processed once) used as benchmark
EN	European Standard
Innovation Sample	Film containing the innovative laminating adhesive
ISO	International Organization for Standardization
MFI	Melt Flow Index
PE	Polyethylene
TC	Technical Committee
Virgin Material	PE resin that will for the first time be converted to a plastic product (no thermal pre-treatment)
wt%	Weight Percentage

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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 website. 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.** The RecyClass Steering Board, following the recommendations of the Technical Committees, is the decision-maker about the compatibility of the innovation with recycling according to the results of the evaluation, granting a technology or product approval letter to the Applicant.

All tests must follow the Evaluation Protocols recommended by the RecyClass Technical Committees and must 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 in the [RecyClass website](#).”

1. INTRODUCTION AND PURPOSE OF THE PROTOCOL

The “RecyClass¹ Recyclability Evaluation Protocol for Laminating Adhesives Applied on PE 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 laminating adhesive innovation is compatible with the post-consumer PE film recycling stream. The Protocol targets companies responsible for introducing a packaging product or a laminating adhesive into the market. The applicant shall proceed with the Protocol as established in the Assessment Process for Applicants of Recyclability Evaluation in the RecyClass Internal Procedures² and RecyClass Technology & Product Approval Quality Management & Procedures document³

The Protocol analyses whether a PE laminate 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 recyclability⁴ of plastics packaging while encouraging innovation in the PE 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 PE film recycling process. This protocol is a simplified version of the Recyclability Evaluation Protocol for PE Films, with a focus on laminating adhesive, and was designed with the support of RecyClass Laminating Adhesive Working Group.

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

PE 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.

Please note that all units in this protocol are expressed following The International System of Units⁵, from the Bureau International des Poids et Mesures.

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 Technology & Product 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 laminating adhesive introduced to the existing packaging solutions for PE films. Prior to initiating the evaluation, the applicant shall review the Design for Recycling Guidelines for clear or coloured PE films¹ in order to confirm that the PE laminate is compatible with these requirements.

This Protocol only covers the recyclability evaluation of PE laminates with laminating adhesive considered as the Innovation. The addition of any other packaging component such as inks, barrier layer or coating will lead to a recyclability assessment that must be performed according to the general Recyclability Evaluation Protocol for PE films.

3. DISCLAIMER

The Protocol is created to represent a simplified version of the actual PE recycling works at an industrial scale. Washing and separation by density steps are skipped in this Recyclability Evaluation Protocol, as they will not affect the rest of the evaluation. RecyClass Technical Committee reserves the right for further testing if necessary, to issue a final opinion on the recyclability of the tested laminate. Note that, the Recyclability Evaluation Protocol establishes some benchmark recommendations to guide the decision-making process. However, not all the properties listed in the protocol are provided with a benchmark recommendation, being the evaluation also based on the technical expertise of the Technical Committee.

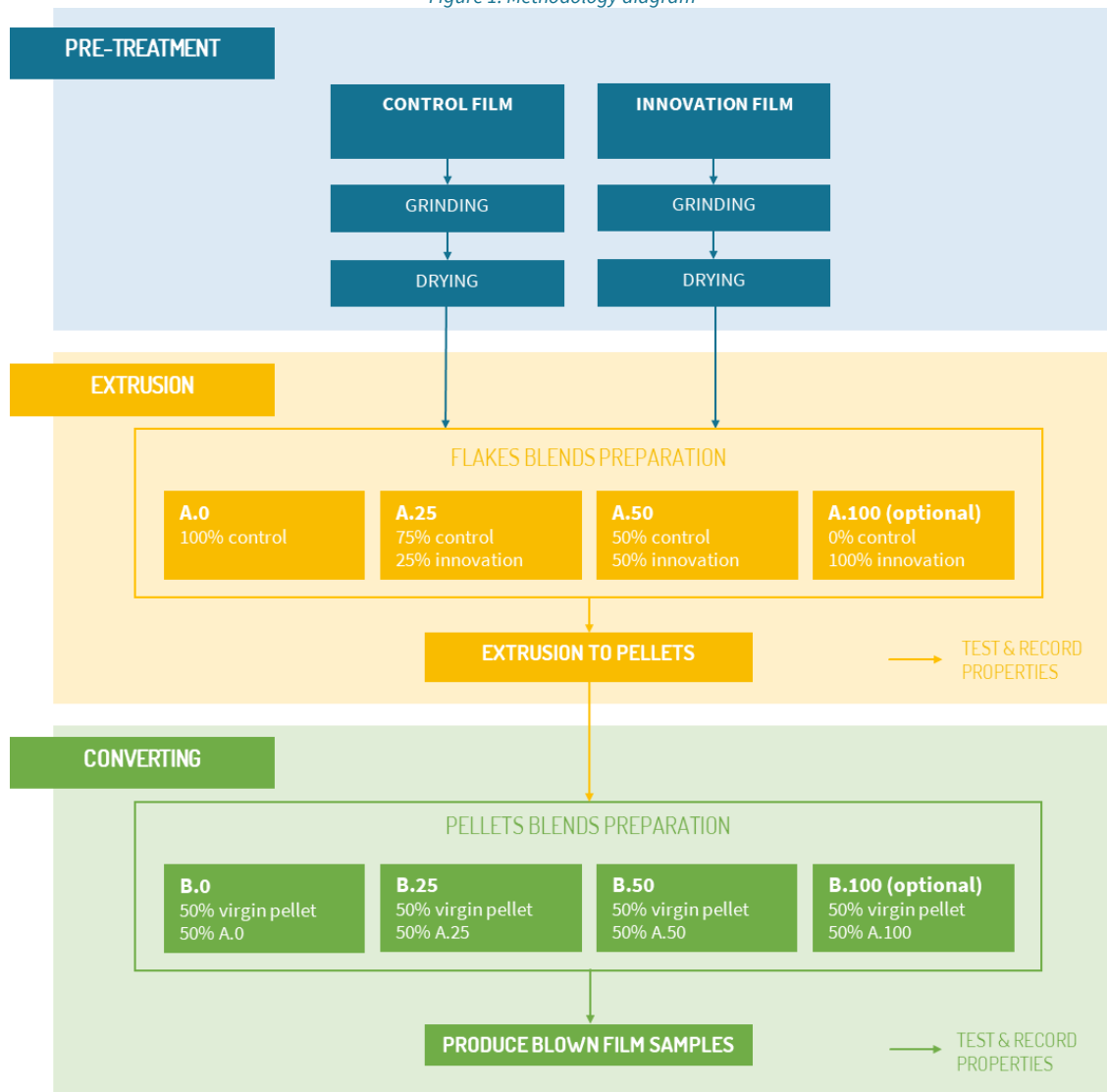
4. LABORATORY TEST METHODOLOGY

This methodology aims to reproduce the recycling process at laboratory scale to determine the suitability of a laminating adhesives with the PE film recycling stream. The methodology described below shall be followed precisely and any modifications or problems must be noted during the testing phase. A Laboratory Evaluation Report compiling objectively 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.

¹ [Design for Recycling Guidelines](#)

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 PE film known to be recyclable, consisting of the same base PE resin as the Innovation, except/apart from the laminating adhesive being evaluated, it can be selected as the control for this Protocol, with the approval of the RecyClass PO films TC. This option should be the one preferred to limit the number of variables in the test.

- **Option 2:** The applicant can select a PE resin listed in the Annex (or another grade with similar MFI and density from alternative suppliers) to be used as control for this Protocol, with/upon the approval of the PO films RecyClass Technical Committee. To obtain the control, the selected PE resin must be extruded once, following the recommendations for extrusion present in this protocol, in order to simulate the same thermal history that an actual packaging may have. The same physical form as the innovative material should be preferred.

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, 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) need 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.

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 5 kg each.

4.2 VIRGIN SAMPLE SELECTION

The virgin PE sample to be used in this Protocol can be selected from the PE 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 ground 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 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 DRYING

Reduce the flake moisture at 60°C without application of 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 flakes before introducing them in the oven.
- As soon as the oven has reached 60 °C, the flakes are added to the oven without the application of 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 following:

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

Where:

Γ : 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 PE 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 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, 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 under the same conditions with hot air and extruded using a co-rotative twin-screw extruder at a melt temperature of 230 °C. The extrudate will be melt 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 APR¹.

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 has to be also 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.
- Dry samples A.0, A.25 and A.50 (optionally A.100) with hot air at 90 °C for 1 hour maximum to decrease surface moisture below 1%. Any agglomeration of flakes must be reported.
- Extrude at a melt temperature of 230 ± 5 °C with a suggested filtration screen of 110 µm. If the range is not optimal, record temperature and state reasons for alteration. Melt residence time should be less than 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%
- Verify that the average pressure is less than 25 % superior to the control over a stable 15 minutes run time.

Record properties' results in Table 2. 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 2). 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 2.

¹ [APR PE Film Practices FPE-P-00](#)

5.2.3.1 PELLET PROPERTIES CHARACTERISATION

Table 2: Pellet properties characterisation

ASSESSMENT	STANDARD	BENCHMARK RECOMMENDATION
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 screen
Impurities (unmolten particles)	Visual inspection	Record
Surface appearance	Visual inspection	Record
Reflection Colour	(L*, a*, b*) + ΔE Reflectance mode, D65, 8-10°	For full compatibility: Δb* < 5 For limited compatibility: Δb* < 8
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 % delta increase to A.0
Pressure Variation (MPa)	(ΔP _{25-30minutes} - ΔP _{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

Prior the recyclability assessment, the RecyClass PO Films Technical Committee 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 PE Films recyclate will be converted via blown film extrusion. For blown 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.

5.3.1 PELLET BLENDS PREPARATION

Once PE pellets have been produced and tested, three additional blends of 50 % virgin – 50 % blend “A.X” (X being 0, 25, 50 or 100) 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 3 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 blown film at 50 % virgin pellet – 50 % Blend A shall be produced as described in Table 3. Blends will be composed of 0 %, 12.5 % and 25 % content (end eventually 50 %) of the initial innovation film.

Table 3: 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 BLOWN FILM PRODUCTION

The following procedure must be followed for the blown film production for all blends.

Procedure:

- Produce blown film with a fixed blow-up ratio (BUR) between 2.5 and 3, at a melt temperature of 200 – 230 °C. A film thickness of $25 \pm 5 \mu\text{m}$ should be targeted for at least 30 minutes. Exact similar conditions must be used for all samples.
- Report the melt temperature during the 30-minutes run time of the $25 \mu\text{m}$ film production.
- Record production properties in Table 4, including information regarding structure, holes, and stability of the $25 \mu\text{m}$ blown film.
- For “Gels & Specks” evaluation, refer to the Annex 3 “PE Film Surface Impression Evaluation and Impurity Visual Inspection”.
- Report the morphology of the gels by performing microscopy or any other optical measurement allowing it.

5.3.3.1 BLOWN FILM PROPERTIES CHARACTERISATION

Table 4: Blown film properties characterisation.

ASSESSMENT	STANDARDS	BENCHMARK RECOMMENDATIONS	
Thickness (μm)	ISO 4593; DIN 53370	25 \pm 5 μm	
Haze (%)	DIN EN ISO 14782	Record. Increase of haze will lower the visual aspects.	
Gels and Specks (Annex 3)	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 of each gels, specks, fisheyes and holes separately. Add microscope pictures of them to show size, shape, distribution.	Δ gels compared to B.0: <25 for full compatibility <50 for limited compatibility
Surface Appearance		Record. Limit the end use application.	

DOCUMENT VERSION HISTORY

VERSION	PUBLICATION DATE	REVISION NOTES
1.0	January 2024	Recyclability Evaluation Protocol for Laminating Adhesives applied On PE Films release

ANNEX I – CONTROL & VIRGIN SAMPLES SELECTION

TYPE OF RESIN	LDPE & LLDPE RESINS*	DENSITY, g/cm ³	MFI at 190 °C / 2.16 kg, g/10 min
LDPE	BOREALIS FT5230	0.923	0.75
LDPE	DOW 310E	0.923	0.75
LDPE	ENI Versalis Riblene FF33	0.923	0.80
LDPE	ExxonMobil LD150BW	0.923	0.75
LDPE	LyondellBasell Lupolen 2420F	0.923	0.75
LLDPE	Dowlex 2045G	0.920	1.0
LLDPE	Dowlex 2740G	0.940	1.0
LLDPE	Dowlex 2750ST	0.950	1.5
LLDPE	Dowlex 2107GC	0.917	2.3

*Other LDPE or LLDPE grades with similar MFI and density from alternative suppliers can also be accepted.

ANNEX II – MASS BALANCE FOR PRE-TREATMENT STEPS

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 – PE FILM SURFACE IMPRESSION EVALUATION AND IMPURITY VISUAL INSPECTION

PE Film Surface Impression Evaluation and Impurity Visual Inspection

Defect description	Rating			
	Very Smooth	Smooth	Rough	Very rough
Texture				
Gels larger than 0.2 mm	: Defined as visually uniform small clumps of crosslinked polymers			
Carbon black particles/Specks	: Defined as very small contaminants, possibly darker in colour			
Fisheyes	: Defined as small round or oblong shapes with darker/more concentrated centre			
Holes	: Defined as tears in the blown film bubble starting at, or caused by, gels, specks or fisheyes			

Preferred range	Limited	Non-acceptable
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For evaluation procedures, RecyClass recommends following the Appendix A “Evaluation of Blown Film Appearance Rating Calculation Totals for Control and Test” present in the APR PE Film critical guidance FPE-CG-01¹

Defect description	B.0	B.25	B.50	B.100
Texture				
Gels larger than 0.2 mm				
Carbon black particles/specks				
Fisheyes				
Holes				

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

¹ APR PE Film critical guidance FPE-CG-01

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