Phone : +32 2 742 96 82
Fax : +32 2 732 12 18
e-mail : recyclass@plasticsrecyclers.eu
website: www.recyclass.eu

RECYCLABILITY EVALUATION PROTOCOL FOR PE FILMS

Standard Laboratory Practice

Version 1.0

Published on 6th September 2018

CONTENTS

1 INTRODUCTION AND PURPOSE OF THE PROTOCOL

The "Recyclability Evaluation Protocol for 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 plastic packaging innovation is compatible with the post-consumer PE 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 analyzes 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 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 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 25 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.

2 SCOPE OF THE PROTOCOL

The scope of the Protocol covers any innovation 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 PE films or coloured PE films[3] in order to confirm that the PE innovation film is compatible with these requirements.

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

1. Non-PE layers and coatings, including PET, nylon, EVOH, and others not specified.
2. Rigid PE and Non-PE attachments to the PE film tested packaging.
3. Mineral fillers and other additives that alter the density of the PE film.
4. Paper and PE labels.
5. Inks and pigments, including direct, reverse, laminated, and other printing technologies.

1RecyClass 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 http://www.recyclass.eu/en/home/

[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.

[3] Design For Recycling Guidelines https://plasticsrecyclers.eu/downloads

6. Compatibilizers and other additives otherwise not specified.

Other packaging containing vacuum-deposited metalized layers, metal foil layers, degradable plastics, as well as PVDC layers or films shall be separately considered by the RecyClass Technical Committee in order to assess their suitability under the scope of this Protocol.

3 DISCLAIMER

The Protocol is created to represent as accurately as possible how the actual PE recycling works at an industrial scale. RecyClass Technical Committee reserves the right for further testing if necessary, to issue an additional opinion on the recyclability of the tested packaging.

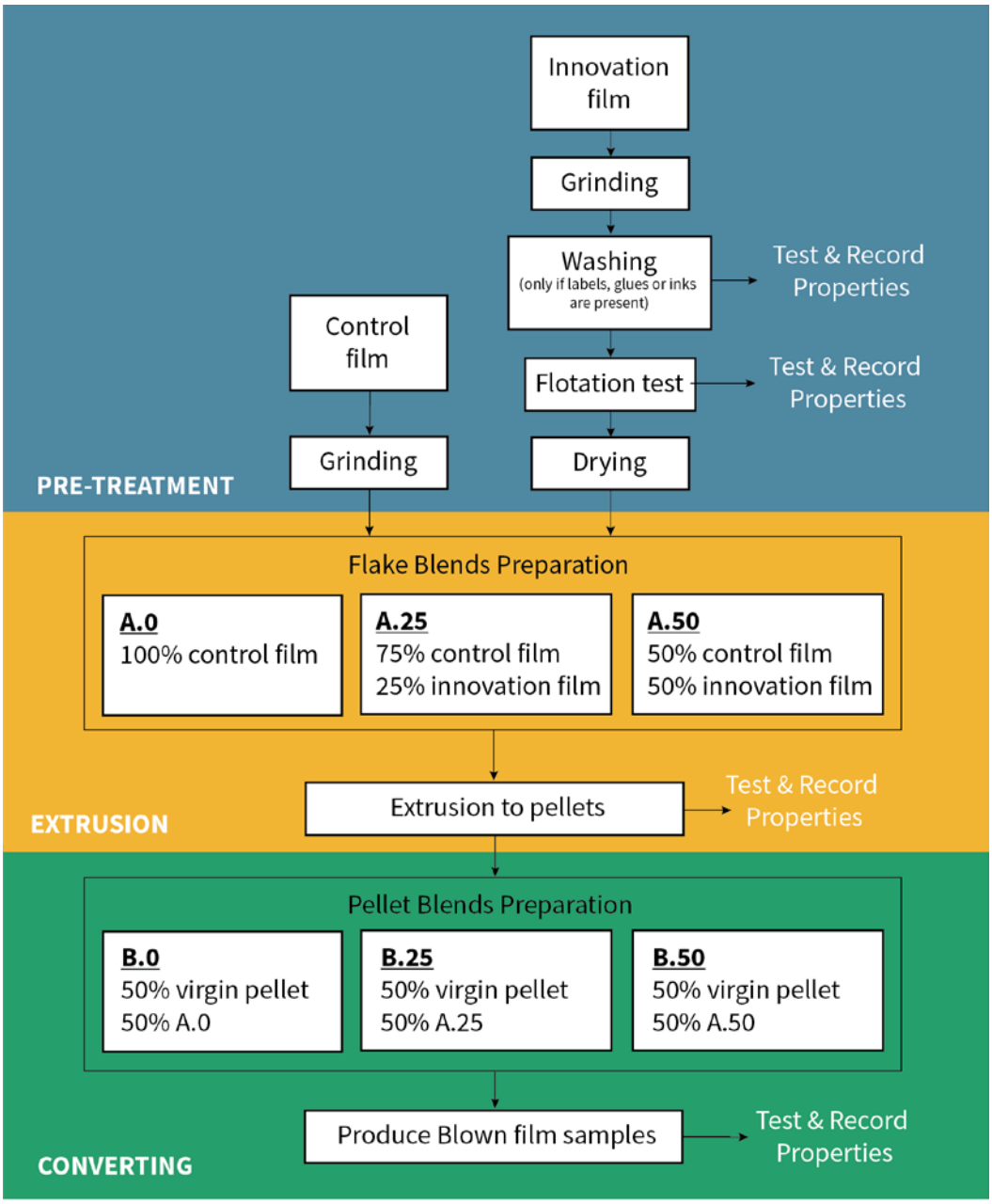
Within RecyClass, "easy-to-empty" and "easy-to-access" indexes are important factors when considering the recyclability of a package. Washing operation at a recycling facility uses mild conditions, no detergents nor strong chemicals. Consequently, any food residue constitutes an impurity for the recycling stream. Plastics Recyclers Europe 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 a small scale to determine the suitability of an innovation material for 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. An Evaluation Report compiling all the results obtained shall be prepared to report to the RecyClass Technical Committee which will interpret the final results. Any remarks during following the Protocol shall be also noted down.

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

Figure1: Methodology Diagram



4.1 CONTROL SELECTION

- **Option 1**: If there is a PE film on the market, similar to the Innovation and is known to be recyclable, it can be selected as the control for this Protocol, with/upon the approval of the RecyClass.

- **Option 2**: If there is a PE film article/virgin known to be recyclable, consisting of the same base PE virgin materials 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.

- **Option 3:** A PE virgin with the same critical technical specifications for MFI and density as the innovation article, ±0.02 MFI and ±0.005 density can be used as the control for this Protocol, with/upon the approval of RecyClass.

4.2 BLENDS PREPARATION

To evaluate and record the properties of innovation film against control as laid out in this Protocol, a set of pellet blends (A.0; A.25; A.50) and a set of blown film blends (B.0; B.25; B.50) are prepared as described in table 1 and table 2. Blends shall be produced once the control and innovation film have separately gone through all pre-treatment steps described below. Both control and innovation flakes can be mixed manually before extrusion. Further size reduction is acceptable if needed.

Blends B will be composed of 50% virgin pellet and 50% of blends A, to be transformed into blown film which will then be tested.

For the purpose of the tests the applicant should provide at least 5 kg amount of innovation material and 20 kg amount of control material which allows for blend preparations of 5 kg each. The laboratory carrying out the Protocol testing may modify these amounts according to their best knowledge. More information can be found in table 3 and 5.

4.3 FLAKE BLENDS COMPOSITION

Three different blends 0%, 25% and 50% of innovation film, will be prepared as described in table 1.

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

4.4 PELLET BLENDS COMPOSITION

Once new PE pellets have been produced (A.0; A.25; A.50) and tested, three additional blends of blown film at 50% virgin pellet – 50% Blend A shall be produced as described in table 2. Blends will be composed of 0%, 12.5% and 25% content of the initial innovation film.

Table 2: Pellet blends composition for the production of blown film

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

4.5 PRE-TREATMENT STEPS

4.5.1 GRINDING

Control and innovation film are separately grinded in order to fit the throat of a standard laboratory extruder.

Procedure:

- Grind separately control and innovation sample to flakes of 10 to 20 mm.
- Store in separate containers.

4.5.2 WASHING

Control and innovation film 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 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 – 25ºC). 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.

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 colouration. Check and record if the glue has been diluted after the rinsing or it remains attached to film flakes.

4.5.3 *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.

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.
- 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 %.

4.5.4 *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 granulates evenly between the 4 dishes. The dishes are sequentially numbered.
- Weigh the different dishes with the trial material before introducing them in the oven.
- As soon as the over has reached 60°C, the trials are added to the oven for 12 hours.
- Weight the material after 6 hours in the oven. Weight the material at the end, after 12 hours in the oven.
- Fill in the results in table 3.

Table 3. Moisture content determination

Trial number	Description	Temp.	Measurements					
			Time	Mass in g	Time	Mass in g	Time	Mass in g
			0 h		6 h		12 h	
			0 h		6 h		12 h	
			0 h		6 h		12 h	
			0 h		6 h		12 h	

4.6 EXTRUSION

4.6.1 PELLET PRODUCTION

Prepare blends for extrusion to pellets as specified in table 1. See additional information in table 4.

Table 4: 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	Optional sample innovators may run for information on the impact of concentration of the innovation on recycling
A.50 50% control with 50% innovation	Per lab requirement for a 30-minute run time	Required for comparison to control values

Procedure:

- Extrude at a preferred melt temperature from 200 – 230°C 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 5.

4.6.2 *PELLET PROPERTIES EVALUATION*

Table 5: Pellet properties evaluation

Assessment	**Result**	**Standard**	**Benchmark Recommendation**
Bulk Density (kg/m^3)		Annex B of EN 15344	No less than 500 kg/m^3
Melt Flow Rate (g/10 min)		ISO 1133	< 0.5 g/10min delta to control value
Ash content (%)		ISO 3451-1 by TGA	Record
Filtration (µm)			
Pellets size (average)		Annex A of EN 15348:2007	Record
Pellets distribution (min – max)		Annex A of EN 15348:2007	Record
Gas content (% weight)		TGA Weight loss at 120°C	Record
Differential Scanning Calorimetry (°C)		ISO 11357	Melt Temperature < 150 °C
Impurities content		Visual inspection	Record
Surface appearance		Visual inspection	Record
Volatiles (%)		Air dried pellets exposed to 160°C for 10 minutes	< 1.0%
Reflection Colour		(L*, b*, a*)	Record
Delta Pressure (MPa)		Less than 25% higher Δ pressure after extruding through 150 mesh for the stable 15 minutes run time, compared to 100% control. No build-up on screen.	No more than 25% delta to control

4.6.3 BLOWN FILM PRODUCTION

Prepare blends for blown film extrusion as described in table 2. See more information in table 6.

Table 6: Blown 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	Optional sample innovators may run for information on the impact of concentration of the innovation on recycling.

Procedure:

- Produce blown film with a blowup ratio > 2.5, at a melt temperature of 200 – 230°C and a thickness < 25 µm.

Record production properties in table 7, including information regarding structure, holes and stability of the blown film.

4.6.4 *BLOWN FILM PROPERTIES EVALUATION*

Table 7: Blown film properties evaluation

Assessment	**Results**	**Standards**	**Benchmark Recommendation**
Thickness (mm)		ISO 4593; DIN 53370	25 µm
Tear Strength (TD**) (g)		DIN EN ISO 6383; DIN EN ISO 1974	No more than 25% delta to control
Tear Strength (MD***) (g)		DIN EN ISO 6383; DIN EN ISO 1974	
Tensile Strength (TD) (MPa)		DIN EN ISO 527	
Tensile Strength (MD) (MPa)		DIN EN ISO 527	
Elongation at Yield (TD) (%)		DIN EN ISO 527	
Elongation at Yield (MD) (%)		DIN EN ISO 527	
Dart Impact (g)		ISO 7765	
Haze (%)		DIN EN ISO 13468	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. 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*