COTREP

Comité Technique pour le Recyclage des Emballages Plastiques

Protocol
Flexible PE-2

TEST PROTOCOL FLEXIBLE PE-2

PRODUCTION OF RECYCLED PE FILMS BY BLOW EXTRUSION

COTREP

The mission of the Technical Committee for the Recycling of Plastic Packaging (COTREP) is to help designers and decision-makers develop recyclable plastic packaging while also providing scope for innovation. The committee includes various stakeholders in the plastic household packaging chain (Citeo, Elipso, SRP and Valorplast) and works on all types of plastic packaging (bottles, dispenser bottles, pots and trays, films and flexible packaging). Protocols for tests performed by COTREP are devised based on work with stakeholders in household plastic packaging end-of-life.

VERSION NO.	DATE	DESCRIPTION
1	September 2021	Initial version

1. CONTEXT

COTREP has drawn up this protocol in collaboration with French manufacturers involved in regenerating flexible PE household packaging. It is representative of the most commonly used processes in Europe. Its purpose is to specify tests to be performed for assessing the suitability of recycled flexible PE granulate produced during regeneration tests in accordance with protocol Flexible PE-1 for transformation into films by blow extrusion (currently one of the two most common outlets for recycled flexible PE).

Results obtained from tests described below may be submitted to COTREP for analysis and potentially included in French recommendations on eco-design aimed at improving recyclability.

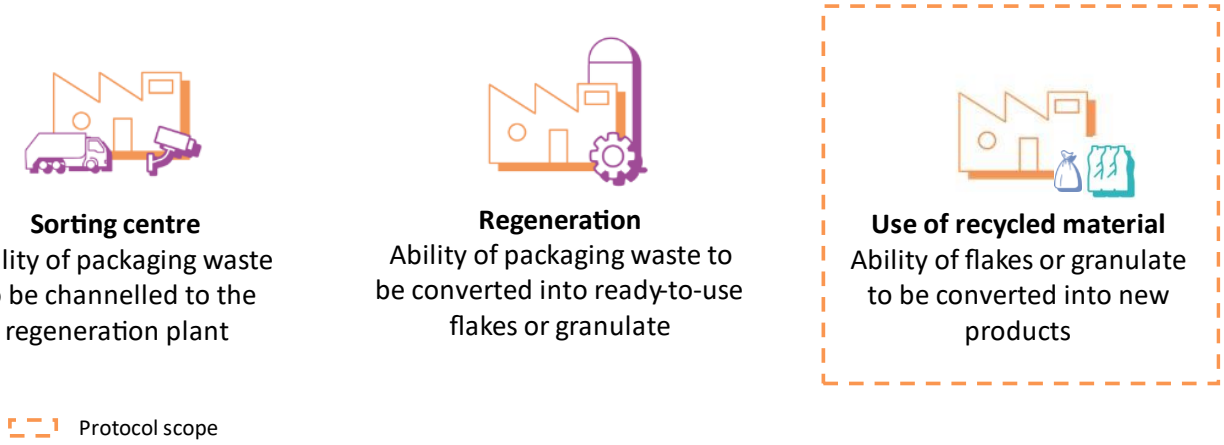

Figure 1: Scope of the COTREP protocol

This protocol takes account of current technical knowledge and processes for transforming rPE granulate into film.

Results obtained from tests performed based on this protocol are insufficient for determining packaging recyclability. This protocol merely reflects the process of transforming regenerated granulate into film and provides no basis for judging the suitability of packaging for sorting.

Please note: This protocol is not appropriate for assessing the suitability of packaging for regeneration in a "mixed plastics", "mixed films" or "mixed PO" stream since such a stream does not exist for French packaging waste.

2. AIMS

This test protocol should be implemented after and in addition to the flexible PE packaging regeneration protocol (PE-1). Its aim is to allow companies to test the production of recycled PE films including regenerated granulate produced from test packaging in semi-industrial conditions. It covers:

- Technical feasibility of transforming granulate into new film,
- An analysis of the quality of the film produced.

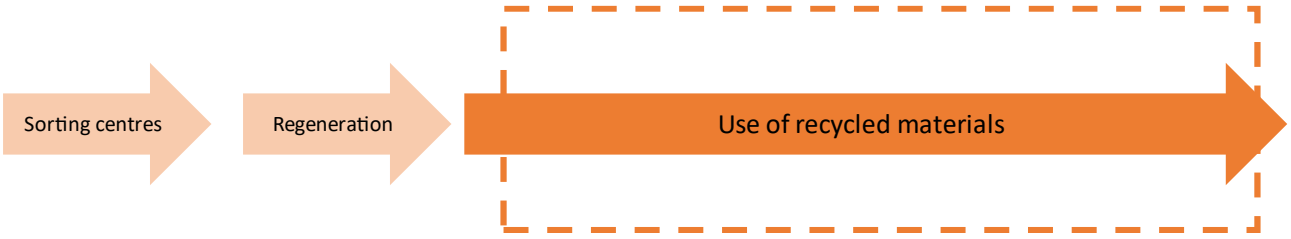

Figure 2: Analytical scope of the blow extrusion protocol

3. TERMS OF REFERENCE

Any company (packaging manufacturer, marketer, resin manufacturer, distributor, etc.) seeking to determine how granulate produced from a specific packaging item according to protocol PE-1 impacts the process of forming by blow extrusion can use this protocol to perform testing.

Companies wishing to perform tests shall be referred to hereafter as **"Requesters"**. COTREP-certified test laboratories able to comply with this test protocol shall be referred to hereafter as "**Laboratories**". A list of certified laboratories is provided in the "Practical information" section.

4. PREPARATION FOR TESTING

Étape 1 : After validating the success criteria for Protocol Flexible PE-1

Once success criteria for Protocol Flexible PE-1 have been validated, the **Requester** should confirm that it wishes the **Laboratory** to perform tests in accordance with Protocol PE-2.

Étape 2 : Prepare for application of Protocol Flexible PE-2.

The **Requester** should provide the selected Laboratory with GM1, GM2 and GT granulate prepared in accordance with the COTREP test protocol on PE regeneration. The test comprises the following steps:

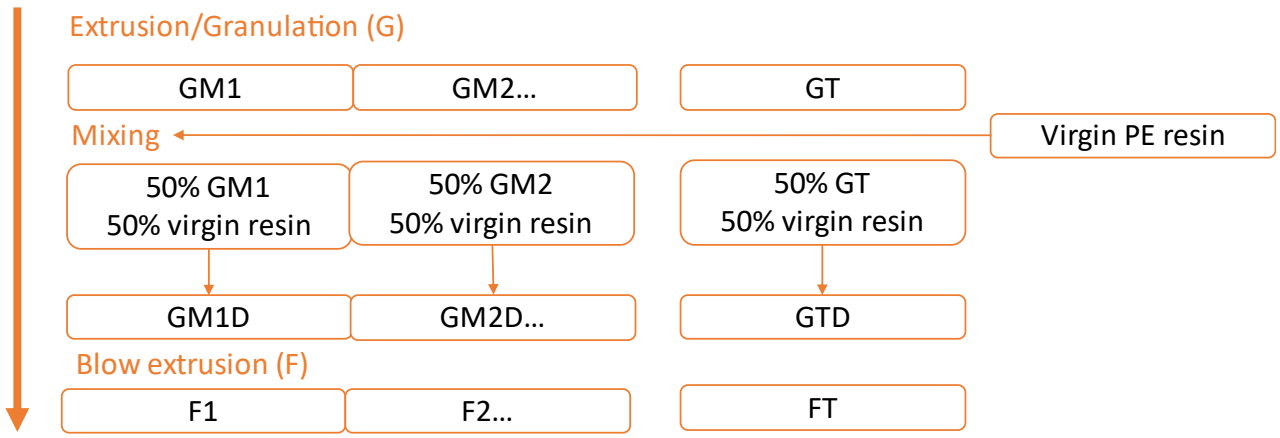

Figure 3: Description of blow extrusion protocol steps

A sufficient quantity of granulate should be supplied to manufacture **25kg** of each blend (GTD, GM1D, GM2D, etc.).

5. METHODOLOGY

The protocol set out below is intended for COTREP-certified **Laboratories** with equipment representative of current transformation processes applied in industrial units. The following steps should be performed:

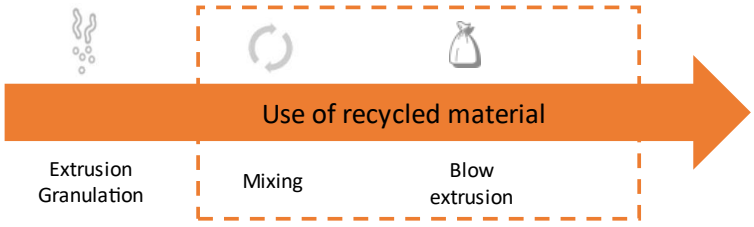

Figure 4: Detailed description of blow extrusion steps

Étape 1 : Preparing mixtures

Granulate GT, GM1, GM2, etc. prepared in advance according to the COTREP PE regeneration protocol (Flexible PE-1) should be mixed with a virgin LDPE resin at a rate of 50% by weight to produce mixtures GTD, GM1D, GM2D, etc. The mixtures should ideally be produced mechanically.

The virgin LDPE granulate used for these tests should be free-radical LDPE granulate of a density of 0.92 kg/m3 and a grade close to 0.5. The following products have been identified as appropriate:

- EXXON 171BA
- SABIC 2801TH00W
- DOW 535E

The **Laboratory** should order the materials required for testing and specify the product used in its final report.

Étape 2 : - Blow extrusion

The prepared mixtures should be extruded to produce a film using the blow extrusion method. The following implementing conditions are recommended for testing:

- Blow-up ratio – between 2.5 and 3
- Filter mesh size 150µm
- Film thickness 50 microns - Tolerance: +/- 5% on average and occasionally +/- 20%

A filter change should be performed for each new mixture. The equipment used and the extrusion conditions applied should be recorded in the final report including the following information:

- Typical extruder type: (screw diameter, L/D ratio)
- Blow film die diameter
- Temperatures of the different zones
- Pressures/amperage
- Air gap
- Extrusion speed
- Blow pressure
- Cooling type (internal/external, air/water)
- Bubble width
- Extrusion time

Blow extrusion: success criteria

- No faults or damage to the line due to the nature of samples (clogging, etc.)
- No bubble deterioration during implementation

The final report should include the following observations:

PROPERTIES EXAMINED	ANTICIPATED RESULTS
IMPLEMENTATION	Observations
EMISSIONS OF FUMES OR ODOURS	Observations
EXTRUSION PARAMETERS	Variation compared to the control sample
FILTRATION	For each filter, provide the reference, visual assessment and photographs
BUBBLE SIZE	Bubble stability (video)
FILM APPEARANCE	Observations (bubbles, holes, etc.)
DIE DEPOSIT	Observations

Étape 3 : Characterisation of films produced

All films produced should be characterised based on the tests set out below. The results should be included in the report.

➔ **Dimensional properties**

PROPERTIES EXAMINED	STANDARDS	ANTICIPATED RESULTS
FLAT WIDTH OF THE EXTRUDED BUBBLE	ISO 4592	*Values*
SHRINKAGE RATE (LONGITUDINAL/TRANSVERSE)	NFT 54-115	*Values*
THICKNESS PROFILE	ISO 4593	*Values*
MEAN THICKNESS	ISO 4591	*Values*

➔ **Mechanical properties**

PROPERTIES EXAMINED	STANDARDS	ANTICIPATED RESULTS
ELONGATION AT BREAK	NF EN ISO 527-3	*Values*
BREAKING STRESS	NF EN ISO 527-3	*Values*
BREAKING STRENGTH	NF EN ISO 527-3	*Values*
COEFFICIENT OF FRICTION (STATIC/DTN)	NF EN ISO 8295	*Value*
SUITABILITY FOR SEALING	/	*Measurable with KOPP equipment*

➔ **Optical and visual properties**

PROPERTIES EXAMINED	STANDARDS	ANTICIPATED RESULTS
COLORIMETRIC TESTING	/	*Testing of Delta E versus a standard sample*
FILM APPEARANCE AFTER ROLLING	/	*Observations regarding gels, surface defects, etc.*
GELS	/	*Visual observation*
SURFACE DEFECTS	/	*Observations*

Characterisation of films: success criteria

- Less than X surface defects observed versus control sample
- Variation < 10% for dimensional properties versus control sample
- Variation < 10% for mechanical properties versus control sample

6. TEST REPORT

The commissioned **Laboratory** should draw up a test report including the following details:

- The report concerning regeneration protocol Flexible PE-1
- A description of samples received including photographs
- APPENDIX 1 completed and appended to the report
- The operating conditions and equipment used for each test
- Results for each step and observations versus the control sample including photographs for each step

Important:

The methodology used for testing all samples submitted for analysis should be strictly identical. The **Laboratory** undertakes to follow the entire protocol, record any deviations in the test report, and send the test reports to COTREP.

The report should include the following declaration:

"*The tests were performed according to COTREP protocol Flexible PE-2 for flexible PE packaging (Reference no./Version/Date). These results do not constitute a full packaging recyclability assessment and are not valid as a recyclability certificate."*

Any deviations should be clarified and will be examined by COTREP to determine whether the results are valid.

7. CONFIDENTIALITY

By signing a confidentiality agreement to be observed with respect to all third parties except COTREP, the **Laboratory** undertakes to maintain the confidentiality of any information concerning the request, the content of the report, and in particular, any results and observations.

8. PRACTICAL INFORMATION

COTREP contacts

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Laboratory contacts

IPC

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Cost of tests

For information: the approximate cost of performing tests in accordance with protocol Flexible PE-2 is €10,000 excl. VAT for the standard and two concentrations of a product.

The **Requester** should also budget for the cost of shipping samples to the **Laboratory**.

ANNEXE 1 : COTREP test request form

REQUESTER:

COMPANY:	*To be completed*
FIRST NAME/LAST NAME:	*To be completed*
POSITION:	*To be completed*
EMAIL:	*To be completed*
TELEPHONE:	*To be completed*

IMAGE OF THE PACKAGING

DESCRIPTION OF THE TEST PACKAGING

PACKAGING TYPE:	*E.g. bottle, dispenser bottle, pot, tray, tube, etc.*
MAJORITY RESIN:	*To be completed*
PACKAGING STRUCTURE:	*If multilayer, describe the layers.* *Specify the % by mass of each component (barrier, additives, adhesive, tie layer, etc.)*
FORMING METHOD:	
COLOUR/PRINTING:	*Specify if on surface or blended*
ASSOCIATED ELEMENTS:	*Labels, tap, zip, tie, etc.* *Specify the composition of each associated element*
VOLUME MARKETED:	*Tonnes per year* *If not yet marketed, provide projections*
COMMENTS:	*Any other potentially useful information for the test*

Company stamp:	**Date:**	**Last name, first name and signature**