

COTREP

Technical Committee for the Recycling of Plastic Packaging

TEST PROCEDURE TO DETERMINE THE INFLUENCE OF A NEW PACKAGING OR NEW MATERIALS FOR PACKAGING ON THE PRODUCTION OF FIBERS FROM R-PET

INTRODUCTION

These test procedures, drafted by the Elipso, Eco-Emballages and Valorplast, in collaboration with the EPBP (European PET Bottle Platform), take account of the specific features of the European market for packaging made out of PET. They are intended to detail the tests to be carried out at the request of the Technical Committee for the Recycling of Plastic Packaging (COTREP) in order to assess a new packaging or new materials for packaging.

OBJECTIVE

To determine whether, at concentrations thresholds calculated as representative for future PET flows, a new material contained in PET bottles obtained from French selective collection and likely to be found in R-PET are of a nature to affect the extrusion process or the properties of the fiber to an unacceptable degree.

TESTS TO BE CARRIED OUT

A. Sampling:

Samples to be tested: pellets coming from PTI in Switzerland:

- Sample A = pellets made from 100% standard flakes
- Sample B = pellets made from X% standard flakes, Y% new packaging

Quantities to be used: about 5 to 10 kg of pellets for each sample.

B. Implementation

During implementation, the measurements and/or observations obtained, in accordance with the analysis methods currently used by the Recycler, on all or part of the following points considered necessary, will be recorded compared with those obtained for the test specimen (sample A) tested under the same conditions.

- drying :(final humidity < 50 ppm)

⇒ color of pellets after drying, => observations + photo (*100 g of the material need to be put aside for further testing*)

⇒ adhesive effect => observations

⇒ emission of smoke or smells, => observations

- Pellets properties:

- ⇒ IV value for the three samples (the test material should have an IV between 0.60 and 0.65)
- ⇒ DSC evaluation (graphs and table) for the three samples
- ⇒ Filterability: the test material must have a good filterability in the spinning pack. The pressure increase in a filtration pack during the production of the fibers, required for testing, will be measured with the standard filtration test (SFT) procedure. Significant deviations with respect to the standard (sample A) are not acceptable.

- Fiber spinning:

The polymer melt temperature is set for 290° and could require optimization per sample. Allowed difference in setting between control and innovative sample is +/- 5°C for the purpose of optimizing the spinning behaviour. If the difference is higher, this will be reported and studied. The filter on the extruder is 325 mesh.

In the first step a **25 dtex** fibre will be produced, the minimum run time is 2 hours; the required amount of material for the second step is around 1Kg.

The stability of the spinning process is an indicator of the rheological properties of the samples and the absence of major disrupting gels and contaminants.

A criterion for a good performance is a stable process for 2 hours.

In the second step the fibres are stretched at 5 stretch ratios: 5, 5.25, 5.5, 5.75 and 6.

This stretching is carried out in 2 steps, the first step on a Pin at temperatures of 90°C and the second step in the hot air oven at 210°C.

A stable stretching operation is an indicator of the stretch-ability of the samples and the absence of gels and contaminants. Anything strange in the melt or in the fibre leading to break or other problems will be monitored and reported.

One pellet sample will lead to 6 final counts.

Properties to check:

- ⇒ drop in intrinsic viscosity, => ΔIV (pellet to fiber)
- ⇒ increase in pressure during extrusion, => time measurement where $\Delta P = 2P$
- ⇒ fiber coloration after extrusion => observations + photo
- ⇒ number of breaks during spinning and stretching, => value

C. Fiber properties

According to the use of the fibers, the values and observations measured on all or part of the following points considered necessary will be recorded compared with the values obtained on a specimen fiber made out of R-PET flakes used for dilution:

- Mechanical properties in traction (tenacity) :

- ⇒ Tensile strength (ASTM 885), => **values**
- ⇒ Elongation (ASTM 885), => **values**

⇒ Modulus (TASE 5%), => **values**

• Thermal stability :

The shrinkage measurement is carried out (without pre-tension) in boiling water for 30 min or in hot air at 100°C. The percentage difference in the length before and after the heat treatment is evaluated and reported.

Acceptable deviation in shrink value from the control is 25%.

⇒ Shrinkage => **values**

In order to compare samples together:

1. At least 5 different values (each corresponding to a different drawing ratio) are needed for each sample and each set of properties.

2. For each test, a table and a graph are to be provided, showing:

- the different values obtained for each sample
- a graph showing those different values

=> Values for each sample are to be compared together in order to assess whether or not samples B and C are within the +/- 25% tolerance compare to sample A.

For example:

The test should verify whether or not the samples B and C are within tolerances concerning tenacity:

$$0.75 \times \text{Tenacity (A)} < \text{Tenacity (B)} < 1.25 \times \text{Tenacity (A)}$$
$$0.75 \times \text{Tenacity (A)} < \text{Tenacity (C)} < 1.25 \times \text{Tenacity (A)}$$

COST OF TESTS

The Recycler will send C.O.T.R.E.P. a detailed estimate intended for the Applicant for the implementation of the tests described above.

RECYCLER'S REPORT

The Recycler will draft a report in which it will indicate:

- the operating conditions and the equipment used for each test,
- the results, photos and observations compared with the test specimen,
- the positive or negative impact that can be envisaged on the recycling of the material tested in the application concerned,
- all of the samples subjected to analyses must be tested in accordance with a rigorously identical methodology.

CONFIDENTIALITY

The Recycler undertakes vis a vis third parties that it will keep confidential any element relating to the application, the content of the report and the results and observations in particular.