

CHI-C8-PEF-1 Recycling Application Compatibility Test for PE-based Flexible Packaging

Version 4.1

This CHI test method was developed by cyclos-HTP for LDPE-based flexible packaging samples with at least one material component or layer in the packaging structure with unknown or theoretically critical properties for the mechanical recycling to produce post-consumer recyclates based on PE-LD films.

This testing and evaluation method considers basic scientific principles, the state of the art and practical experiences during the industrial recycling process. The test program is oriented toward real-life practice in terms of the selection of reference material and takes into account relevant applications in connection with the technologies and procedures that are deployed as standard in the recycling industry.

For PE-based flexible packaging samples, this means that the material samples are evaluated regarding their suitability for both injection moulding applications (e.g., buckets) and typical blown film applications (e.g., garbage bags).

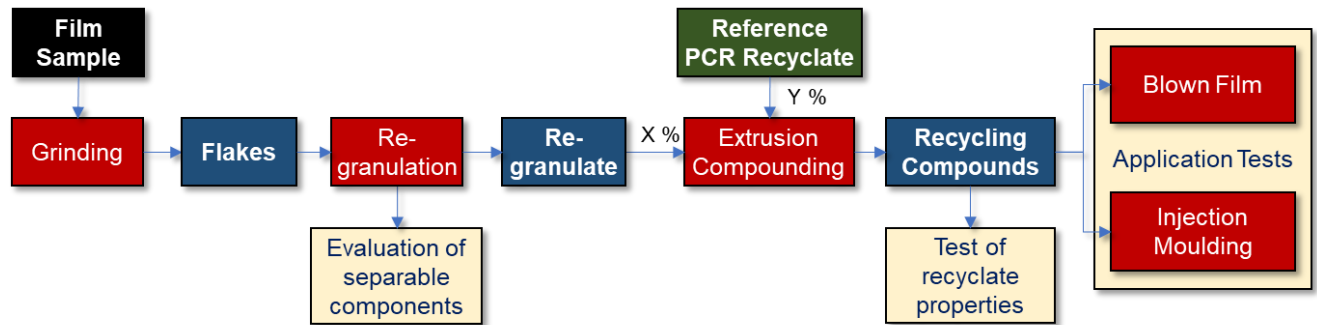
The compatibility test program includes the following steps. The principle of the test method is shown in the flow chart on page 3.

- Materials** to be provided:
 - Flexible PE-based packaging/material structure containing a component with an unknown or theoretically critical recycling compatibility, "**PCK-S1**"
 - A PE-based reference packaging structure with approved or recognized recyclability, made of the same base PE grades (PE-LD or PE-LLD) as "PCK-S1", but without the critical component, "**PCK-R0**"
 - Commercial PCR recyclate commonly used for the same target application in the recycling path, "**REF**"
 - Grinding** of the respective samples "PCK-S1" and "PCK-R0" separately to flakes on a granulator with 6-8 mm sieve.
 - Homogenization of grinded flakes** – Each of the grinded samples must be homogenized separately by manual mixing of the flakes required for the entire test program. This is to achieve a uniform quality of the sample flakes.
 - Homogenization of the reference recyclate** – Manual mixing of the PCR granules from all containers and batches required for the entire test program. This is to achieve a uniform quality of the recyclate so that all samples produced can be compared with each other. The reason is that the composition and quality of the PCR granulate can vary also within a production lot.
 - Regranulation of samples** – The sample flakes from step 3 are melted with a mono-screw or twin-screw extruder. The effective target melt temperature must be $225 \pm 5^\circ\text{C}$ (in a range of 220°C to 230°C). Therefore, suitable melt pressure settings and the right temperature settings of the extruder zones and the die must be chosen which are sometimes below the final melt temperature. This procedure is to ensure that the samples to be tested have a comparable history in terms of thermal and mechanical stress as the reference recyclate, before both components are combined to blends in the next step.
 - Melt Screening** (filter with 100 μm pore size) during regranulation of the sample flakes can be used by demand if relevant for the samples to separate heterogeneous components and to examine them qualitatively. The quantitative assessment of inhomogeneity is performed by documenting the melt pressure increase within 30 minutes after start of extrusion. Where indicated, the filtrate can be qualitatively examined to assign it to a component in the starting material.
- Regranulate samples to be produced: **RG-S1**, **RG-R0**.
- Characterisation of regranulates in comparison with the original film samples:**
 - Differential scanning calorimetry (DSC), to evaluate the melt temperatures and (by demand) the relative degree of crystallinity of the film samples (PCK-S1, PCK-R0) and regranulates (RG-S1, RG-R0).
 - Thermo-gravimetric analysis (TGA), to evaluate the ash content but also any volatile components in the film samples (PCK-S1, PCK-R0) and regranulates (RG-S1, RG-R0) that might be relevant for the applied temperatures in the recycling process. This analysis is optional only for relevant samples.

7. **Extrusion/compounding** of materials combinations on a lab-scale extruder with mono or double screw to form various recycling compounds and simulate different concentrations of the packaging samples in the recycling stream. The effective melt temperature must be $210 \pm 5^\circ\text{C}$ (in the range of 205°C to 215°C). Higher temperatures should be avoided to prevent further degradation or cross-linking of the PE. Therefore, suitable melt pressure settings and the right temperature settings of the extruder zones and the die must be chosen.
- The following compounds are produced:
 - **CHI05**: 5% **RG-S1** + 95% REF
 - **REF05**: 5% **RG-R0** + 95% REF
 - **CHI30**: 30% **RG-S1** + 70% REF
 - **REF30**: 30% **RG-R0** + 70% REF
 - The compounds REF05 and REF30 are produced as “zero samples” to prove whether a negative deviation of the test results with CHI05 or CHI30 originates from the PE base material in the sample or the critical component under investigation.
8. **Characterisation of recycling compounds:**
- Melt flow rate (MFR), to test if the compounds are in a typical viscosity range for the relevant applications as the PCR reference.
9. **Test specimens for Injection moulding** are prepared from recycling compounds CHI05, REF05, CHI30, REF30, according to DIN EN ISO 3167: M-REF05, M-CHI05, M-REF30, M-CHI30.
10. **Characterisation of Injection moulding specimens:** Mechanically by tensile testing (DIN EN ISO 527) and Charpy notched impact strength (DIN EN ISO 179-1), morphologically (microscopy) by demand.
11. **Blown Films** are produced containing 60% of the corresponding recycling compounds from step 6 and 40% virgin PE-LD/LLD according to the CHI test method *CHI-C8-BFPE*. The share of recycling compound in the film can be reduced to 50% or even to 40% by demand if no stable blown films can be produced with 60% REF05. Blown film samples to be produced: F-REF05, F-CHI05, F-REF30, F-CHI30.
12. **Testing of blown film samples** according to the CHI test method *CHI-C8-BFPE* regarding the mechanical and optical properties, as well as sealing properties and sealing seam tightness.
13. **Evaluation** of all test results is carried out according to the following criteria, whereby the evaluation refers to the tested applications. The results of samples CHI05 are compared with REF05. The results of samples CHI30 are compared with REF30. An evaluation for the applications in injection moulding and blown films is made in the following order:
- (A) A significant, negative deviation of the relevant test results between **CHI05** and **REF05** leads to a classification of the assessed packaging structure as "not recyclable", resp. the component as "not compatible for recycling" (CAT3 according to the CHI assessment and requirement catalogue). If the relevant results between CHI05 and REF05 are in a comparable range or CHI05 is even better, the assessed substance or packaging structure can be stated as “compatible for recycling” and the packaging structure is classified as “recyclable”.
 - (B) If the relevant results between **CHI30** and **REF30** are in a comparable range or CHI30 is even better, the assessed packaging structure or substance can be stated as “recycling compatible” and - under suitable conditions – completely or partially as “valuable material”. A definitive assessment of a single substance according to this criterion is only possible if the suspect material is present in the original packaging sample in a high concentration.
- The results of (A) and (B) are always compared to see any effects from the structure or its components with increased concentration in the recycling stream. Therefore, an assessment could be negative (“sample is not recyclable”) when certain parameters in (A) are acceptable but significant negative effects are obtained in (B) with the same parameters.
 - The evaluation is basically always considering the standard deviations of each measured parameter, if a parameter is calculated as the average of several measurements or specimen of the same sample material.

Appendix

A. Principle of the test method:



B. Overview of test samples and evaluation parameters:

	Trial #	REF05	CHI05	REF30	CHI30
Recyclate blends	PCK-S1 (sample with critical component)	0%	5%	0%	30%
	REF (reference PCR recyclate)	95%	95%	70%	70%
	PCK-R0 (reference packaging sample)	5%	0%	30%	0%
Recyclate blends characterisation	Density [g/cm³] (by demand)				
	MFR (190°C/2.16kg) [g/10min]				
Injection moulding application mechanical testing	Tensile Modulus (E _t) [Mpa]				
	Tensile Strength (σ _m) [Mpa]				
	Elongation at Break (ε _b) [%]				
	Charpy Impact strength (acU) [kJ/m²]				
Blown film application mechanical testing	Tensile strength (MD/CD)				
	Elongation at Break (MD/CD)				
	Puncture Resistance (static test)				
	Dart impact failure (dynamic test)				
Blown film application sealing properties	Sealing properties on lab sealing press (sealing temperature T _s vs. time t _s)				
	Tensile testing of sealing strength				
	CHI seam tightness test (see CHI-C8-BFPE)				

Version history:

Version No.	Date	Reason/Content of revision
1.0	Oct 2019	First version of test method
4.1	May 2023	Reworked test parameters and criteria, sample nomenclature