CEN/WS rPVB

Date: 2024-09

prCWA XXXX:2024

Secretariat: DIN

Plastics — Recycled plastics — Characterization of polyvinyl butyral (PVB) recyclates

Kunststoffe — Kunststoff-Rezyklate — Charakterisierung von Polyvinylbutyral (PVB) Rezyklaten

Plastiques — Plastiques recyclés — Caractérisation des recyclats de butyral de polyvinyle (PVB)

CCMC will prepare and attach the official title page.

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European foreword

Draft CWA xxxx:2024 has been developed in accordance with the CEN-CENELEC Guide 29 "CEN/CENELEC Workshop Agreements – A rapid way to standardization" and with the relevant provisions of CEN/CENELEC Internal Regulations – Part 2. The proposal was approved and supported by CEN following a public call for participation made on 2024-03-04. The Kick-off Meeting took place on 2024-04-12, the draft CWA was approved on 2024-09-30 and the final CWA is intended to be approved by representatives of interested parties in a Workshop on 2024-11-26. It does not necessarily reflect the views of all stakeholders who may have an interest in its subject matter.

Results incorporated in this CEN Workshop Agreement received funding from the European Union's Horizon 2020 research and innovation programme under the grant agreement number 958243 (SUNRISE).

The following organisations and individuals developed this draft CEN Workshop Agreement:

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Introduction

Recycling plastic waste, by mechanical recycling, is one type of material recovery process intended to save resources (virgin raw materials, water, and energy), while minimizing harmful emissions into air, water and soil as well as any impacts on human health. The environmental impact of recycling has to be assessed over the whole life cycle of the recycling system (from the waste generation point to the disposal of final residues). To ensure that recycling constitutes the best environmental option for treating the available waste, some prerequisites should preferably be met:

- recycling scheme being contemplated should generate lower environmental impacts than alternative recovery options;
- existing or potential market outlets should be identified that will secure a sustainable industrial recycling operation;
- collection and sorting schemes should be properly designed to deliver recyclable plastics waste fractions fitting reasonably well with the available recycling technologies and with the (changing) needs of the identified market outlets, preferably at minimum costs to society.

This document has been produced in accordance with the guidance produced by CEN on Environmental Aspects and in accordance with CEN/TR 15353.

NOTE CEN/TR 15353 considers the general environmental aspects, which are specific to the recycling process.

It is often impossible to trace back each individual product at the end user stage and to check whether the product has been used correctly through its life. Consequently, products are out of industrial control for a period of time. It is possible that during this period contamination with other materials might occur that could affect the product's suitability for recycling into the intended application.

1 Scope

This document defines a method of specifying delivery conditions for polyvinyl butyral (PVB) recyclates.

It gives the most important characteristics and associated test methods for assessing PVB recyclates intended for use in the production of semi-finished/finished products.

It is intended to support parties involved in the use of PVB obtained by mechanical and/or mechanochemical recycling to agree on specifications for specific and generic applications.

This document is applicable without prejudice to any existing legislation.

This document does not cover the characterization of plastic waste, which is covered by EN 15347, neither traceability topics which are covered by EN 15343.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 12099, Plastics piping systems — Polyethylene piping materials and components — Determination of volatile content

EN 15343, Plastics — Recycled plastics — Plastics recycling traceability and assessment of conformity and recycled content

EN ISO 105-A03, Textiles — Tests for colour fastness — Part A03: Grey scale for assessing staining

EN ISO 178, Plastics — Determination of flexural properties

EN ISO 489, *Plastics* — *Determination of refractive index*

EN ISO 527-1, Plastics — Determination of tensile properties — Part 1: General principles

EN ISO 527-2, *Plastics* — *Determination of tensile properties* — *Part 2: Test conditions for moulding and extrusion plastics*

EN ISO 527-3, Plastics — Determination of tensile properties — Part 3: Test conditions for films and sheets

EN ISO 1133-1:2022, *Plastics — Determination of the melt mass-flow rate (MFR) and melt volume-flow rate (MVR) of thermoplastics — Part 1: Standard method (ISO 1133-1:2022)*

EN ISO 3451-1:2019, Plastics — Determination of ash — Part 1: General methods (ISO 3451-1:2019)

EN ISO 11357-2, Plastics — Differential scanning calorimetry (DSC) — Part 2: Determination of glass transition temperature and step height

EN ISO 11358-1, Plastics — Thermogravimetry (TG) of polymers — Part 1: General principles

EN ISO/CIE 11664-4, Colorimetry — Part 4: CIE 1976 L*a*b* colour space

ISO 105-A02, Textiles — Tests for colour fastness — Part A02: Grey scale for assessing change in colour

ISO 3534-2, Statistics — Vocabulary and symbols — Part 2: Applied statistics

ISO/CD 13659, Chain of Custody — Book and Claim — Requirements and guidelines

ISO 14782:2021, Plastics — Determination of haze for transparent materials

ISO 17223:2014, Plastics — Determination of yellowness index and change in yellowness index

ASTM D1003-21, Standard Test Method for Haze and Luminous Transmittance of Transparent Plastics

ASTM E313, Standard Practice for Calculating Yellowness and Whiteness Indices from Instrumentally Measured Color Coordinates

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN ISO 472 and CEN/TR 15353 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <u>https://www.iso.org/obp/</u>
- IEC Electropedia: available at <u>https://www.electropedia.org/</u>

3.1

post-consumer

descriptive term covering material, generated by the end-users of products, that has fulfilled its intended purpose or can no longer be used (including material returned from within the distribution chain)

Note 1 to entry: The term "post-use" is sometimes used synonymously.

[SOURCE: EN ISO 472:2013, definition 2.1700]

3.2

pre-consumer

descriptive term covering material diverted during a manufacturing process

Note 1 to entry: This term exludes re-utilized material, such as rework, regrind or scrap that has been generated in a given process and is capable or being reclaimed within that same process.

Note 2 to entry: The term "post-industrial material" is sometime used synonymously.

[SOURCE: EN ISO 472:2013, definition 2.1701]

4 Symbols and abbreviations

For the purposes of this document, the symbols and abbreviations related to recyclates are given in EN ISO 1043-1.

5 Characterization of PVB recyclates

A single batch is the quantity of recyclate that has homogenous characteristics within the specified tolerances.

The characteristics of PVB recyclates, which shall be met for every batch according to ISO 3534-2 are given in Table 1, and are divided into two types:

- a) mandatory characteristics (M) required to characterize PVB recyclates in general and required for all applications;
- b) optional characteristics (0) needed to characterize PVB recyclates according to customer specifications and applications.

NOTE PVB or PVB-containing plastic wastes for recycling may contain different PVB types such as acoustic and non-acoustic PVB. Further variations are seen in the form of the added plasticizer (type and amount). The properties

and performance of recyclates derived from such wastes will depend on the relative proportions of the PVB types and the plasticizers.

These characteristics shall be assessed by using the test methods given in Table 1. Where possible, the supplier should provide information on the original applications.

When the PVB recyclate is intended for the use in laminated glass applications, the following optional characteristics are mandatory and shall be reported: colour, glass transition temperature, haze, light transmittance, yellowness index.

Other tests may be carried out by agreement between the purchaser and the supplier and results shall be reported.

A certificate of analysis giving the test results for each batch of recyclate shall be provided by the supplier to the purchaser upon request.

Where several methods are listed in Table 1, the certificate of analysis shall specify the method used.

The purchaser may require some additional information on recyclate composition from the recycler in order to facilitate the legal use of the recyclate.

In addition to the physical and chemical characteristics specified in Table 1, each batch shall be accompanied by an information stating:

- product name;
- intended (suitable) application(s).

Characteristic	Unit	Test method	PVB recyclate	Comment
Ash content	%	EN ISO 3451- 1:2019, Method A	М	The amount of PVB (in g) used to determine the ash content shall be stated in the report.
				Ash content can be used to indirectly determine the glass and metal content of the PVB recyclate.
Colour		Visual inspection	М	ISO 105-A02 and EN ISO 105-A03
Impurities		based on ISO 12418-2:2012, Annex A	М	Depending on the original use and the recycling process different impurities can be present (e.g. glass, metals, calcium carbonate). Additional tests may be agreed between the interested parties depending on the impurities present in the recyclate. Report the size range of the visually detected impurities using a ruler.

Table 1 — Characterization of PVB recyclates

Characteristic	Unit	Test method	PVB recyclate	Comment
Original application (i.e. which product was the PVB in before being processed to a recyclate) and material history		Supplier to declare	М	e.g. pre-consumer, post- consumer, automotive windshields, buildings, acoustic PVB, non-acoustic PVB
Plasticizer content	%	ISO 11358-1	М	
Residual humidity	%	EN 12099	М	Although the scope of EN 12099 is limited, it is considered relevant.
Yellowness index		ASTM E313, ISO 17223	М	YI can be used to determine indirectly degradation extent (i) For measurement on a foil: The results should be reported with the PVB-foil thickness. (ii) For measurement on a laminate of glass/PVB/glass: Results should be reported together with lamination conditions, PVB foil thickness, glass thickness and utilized glass type. If PVB flakes were processed to a foil, then the foil processing method should be reported by the supplier. Do be measured on foil and/or laminated foil.
Adhesion to glass	N/cm	To be agreed between the interested parties.	0	

Characteristic	Unit	Test method	PVB	Comment
	<u> </u>		recyclate	
Colour	L, a, b values	EN ISO/CIE 11664-4		Observer: 10°, Illuminant: D65, measured in transmission mode (i) For measurement on a foil: The results should be reported with the PVB-foil thickness. (ii) For measurement on a
			0	laminate of glass/PVB/glass: Results should be reported together with lamination conditions, PVB foil thickness, glass thickness and utilized glass type. If PVB flakes were processed to a foil, then the foil processing method should be reported by the supplier.
				Do be measured on foil and/or laminated foil.
Elasticity modulus		EN ISO 178	0	
Filtration level	μm	Mesh size	0	for granules produced by extrusion
Flexural modulus	МРа	EN ISO 178	0	
Flowability		1	0	e.g. loose, sticky
Glass transition point	°C	EN ISO 11357- 2	0	Indirect measurement for plasticizer content which lowers the glass transition temperature of PVB. The scanned temperature range shall start by at least -20 °C.
Haze		ASTM D1003 and/or ISO 14782	0	For laminated glass application: Measured on a laminate of glass/PVB/glass. Results should be reported together with lamination conditions, PVB foil thickness, glass thickness and utilized glass type. If PVB flakes were processed to a foil, then the foil processing method should be

Characteristic	Unit	Test method	PVB	Commont
			recyclate	comment
Light transmittance	%	ASTM D1003 with illuminant A/2° or C/2°	0	For laminated glass: (i) Relevant for laminated glass; optional characteristic only for this application Measured in a laminate of glass/PVB/glass. Results should be reported together with lamination conditions, PVB foil thickness, glass thickness and utilized glass type. If the supplier processed the PVB flakes to a foil, then the foil processing method should be reported by the supplier. (ii) Measured on the PVB foil: The results should be reported together with the PVB-foil thickness.
Melt mass-flow rate	g/10 min	EN ISO 1133-1	0	Optional because only relevant for extrusion process
Refractive index		EN ISO 489	0	Preferably, the refractive index should be measured at 20°C or 25°C.
Tensile stress at yield	МРа	EN ISO 527-1 EN ISO 527-2 EN ISO 527-3	0	
Tensile strain at break	%	EN ISO 527-1 EN ISO 527-2 EN ISO 527-3	0	
Thermal conductivity			0	relevant for PV applications

6 Quality assurance

In order that the purchaser of the recyclate can have confidence in the quality of the product, the supplier/recycler shall maintain records of the quality control carried out by the supplier/recycler.

NOTE A quality management system certified to EN ISO 9001 can be a suitable guarantee of consistent recyclate quality but not the recycled content.

The specification and the standard deviation or range of values within and between batches of material shall be agreed between the supplier and the purchaser.

Where a statement of recycled content, or the previous history of the material, is requested, documentary evidence shall be provided. These records should be available to the purchaser on request.

Where a recyclate has been produced via a melt process, the supplier may choose to state the level of filtration applied during that process. This will determine the maximum size of any non-melting contaminants present in the recyclate. The statement of filtration level shall include details of the filter.

Recyclates that have not passed through a melt process cannot be quantified in the same way, and the supplier may state this.

Traceability shall be ensured according to EN 15343, that describes a qualified recycling process and gives details of traceability and the assessment of recycled content. The calculation of the recycled content shall follow ISO/CD 13659.

Bibliography

- [1] CEN/TR 15353, Plastics Recycled plastics Guidelines for the development of standards for recycled plastics
- [2] EN 15347, Plastics Recycled plastics Characterisation of sorted plastics wastes
- [3] EN ISO 472, Plastics Vocabulary
- [4] EN ISO 1043-1, Plastics Symbols and abbreviated terms Part 1: Basic polymers and their special characteristics
- [4] EN ISO 9001, Quality management systems Requirements