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Deliverable report

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CREATOR CONSORTIUM

PARTICIPANT NUMBER	ABBREVIATION	ORGANISATION
1	ICT	Fraunhofer-Gesellschaft zur Förderung der angewandten Forschung – Institut für Chemische Technologie
2	VLB	Volbas S.A.
3	MOS	Machinefabriek Otto Schouten BV
4	CLR	Coolrec BV
5	REL	Treee SRL
6	GKR	Fundacion Gaiker
7	TCK	Transfercenter fur Kunststofftechnik GmbH
8	RMA	Erema Engineering Recycling Maschinen und Anlagen Ges.m.b.H
9	CTB	Centre Scientifique & Technique De L'industrie Textile Belge
10	MAI	Maier S. Coop.
11	DAW	DAW SE
12	CYC	Cyclefibre S.L.
13	CID	Fundacion Cidaut
14	KLU	Kuhne Logistics University GmbH
15	OVM	Openbare Vlaamse Afvalstoffenmaatschappij
16	RWE	RWEnergia Robert Wudarczyk
17	ITB	ITRB Group LTD

DOCUMENT HISTORY AND CONTRIBUTION OF THE PARTNERS

VERSION NR	REVISER	CONTENT
V0	GKR	Document edition (first draft)
V1	ICT	Revision
V3	GKR	Document edition (final version)
V4	ICT	Submission

PARTNER	SHORT NAME	ROLE IN THE WP	CONTRIBUTION TO THE DELIVERABLE
Gaiker	GKR	Leader of WP1 and Task 1.5, and participant in Tasks 1.1, 1.3, 1.4 and 1.6	Characterisation of purified PS, preparation of the report and lead partner
Fraunhofer Institute for Chemical Technology	ICT	Participant in Tasks 1.2, 1.3, 1.4, 1.5 and 1.6	Supply and characterisation of purified PS, and report revision

LIST OF ABBREVIATIONS

Br	Bromine
PS	Polystyrene
UL 94	Underwriters Laboratories test standard UL 94
WP	Work package
XRF	X-ray fluorescence

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1 INTRODUCTION

This report summarises a basic proposal of properties to be included in the technical data sheet of recycled plastics and, therefore, in the CREAToR smart labelling system. The labelling system was reported previously in the deliverable D1.6 Smart labels for the materials used within CREAToR[1]. The properties or characteristics for the labelling of recycled plastics have been selected and agreed by the project consortium, with particular consideration for the requirements of recyclers and material end-users.

The abovementioned properties have been analysed for the case study of purified polystyrene (PS) obtained in the scope of work package 3 *Purification of the stream and removing the flame retardants*. The characterisation tests were carried out by GKR, and some of the analytics were also performed in parallel by ICT (round robin tests). These analytics were carried out within Task 1.5 *Data generation for the creation of smart labels for the materials* within work package 1 *Material requirements, labelling & standardisation* in the CREAToR project.

[1] *https://creatorproject.eu/publications/*

2 MATERIAL CHARACTERISTICS FOR LABELLING

The basic material characteristics to be considered for the CREAToR smart label have been selected by project partners (ITB, GKR, CLR, DAW and MAI). This selection was carried out after several discussions and was also based on the information derived from the workshop for recyclers held at the Kühne Logistics University (KLU) in March 2020. The identified characteristics will provide key information to end-users (plastic compounders and convertors) about the quality of recycled plastics, thus helping to overcome market barriers to their use as secondary raw materials.

The physical, rheological, and mechanical properties, flammability ratings and average bromine content have been determined in the PS derived from the purification process developed in WP3 (Figure 1). The target material characteristics and the corresponding test methods are listed in Table 1.

Figure 1. Purified PS (microspheres) from ICT

Table 1. Specifications for labelling

TECHNICAL SPECIFICATION	TEST METHOD	DENOMINATION
Density	UNE-EN ISO 1183	Plastics – Methods to determine the density of non-cellular plastics – Part 1: inmersion method, liquid pycnometer method and triation method[2]
Melt-flow rate	UNE-EN ISO 1133	Plastics – Determination of the melt mass-flow rate (MFR) and melt volume-flow rate (MVR) of thermoplastics – Part 1: standard method[3]
Flexural modulus **Flexural strength**	UNE-EN ISO 178:2020	Plastics – Determination of flexural properties[4]
Tensile modulus of elasticity **Tensile strength**	UNE-EN ISO 527-2/1A/1:2012	Plastics – Determination of tensile properties – Part 2: test conditions for moulding and extrusion plastics[5]
Charpy impact	UNE-EN ISO 179-1/1eU:2011	Plastics – Determination of Charpy impact properties – Part 1: non-instrumented impact test[6]
Flammability rating	UL94 standard	UL94 vertical burning test (V-0, V-1 or V-2) UL94 horizontal burning test (HB)
Bromine concentration	X-ray fluorescence	--

Regarding the flammability rating, UL 94 (Underwriters Laboratories test standard UL 94) is a widely used flammability test to determine the relative flammability of plastics. It measures the ability of a plastic part to

[2] *UNE-EN ISO 1183. Plastics – Methods to determine the density of non-celular plastics – Part 1: inmersion method, liquid pycnometer method and triation metod*
[3] *UNE-EN ISO 1133. Plastics – Determination of the melt mass-flow rate (MFR) and melt volume-flow rate (MVR) of thermoplastics – Part 1: standard method.*
[4] *UNE-EN ISO 178:2020. Plastics – Determination of flexural propeties*
[5] *UNE-EN ISO 527-2/1A/1:2012. Plastics – Determination of tensile properties – Part 2: test conditions for moulding and extrusion plastics.*
[6] *UNE-EN ISO 179-1/1eU:2011. Plastics – Determination of Charpy impact properties – Part 1: non-instrumented impact test*

extinguish the flame after ignition and its dripping behaviour in response to a small open flame or radiant heat source under controlled laboratory conditions[7]. The tests results are intended to serve as a preliminary indication of the material sustainability with respect to flammability for a specific application.

The UL standard classifies a plastic according to the minimum thickness at which it stops burning when tested in a horizontal or vertical orientation. The flammability tests covered by the UL94 standard include vertical and horizontal burning tests. The UL test methods are aligned with the flowing standards used to determine fire properties: IEC 60707 *(Flammability of solid non-metallic materials when exposed to flame sources - list of test methods)*, IEC 60695-11- 10 (*Fire hazard testing - Part 11-10: Test flames - 50 W horizontal and vertical flame test methods)*, IEC 60695-11-20 *(Fire hazard testing - Part 11-20: Test flames - 500 W flame test methods)*, ISO 9772 *(Cellular plastics — Determination of horizontal burning characteristics of small specimens subjected to a small flame)* and ISO 9773 *(Plastics - Determination of burning behaviour of thin flexible vertical specimens in contact with a small-flame ignition source)*.

The UL94 vertical rating measures the self-extinguishing time of the vertically oriented polymer specimen. The top of the test specimen is clamped to a stand and the burner is placed directly below the specimen. The distance between the top of the burner and the impingement zone is 10 ± mm. A 50 W calibrated flame is applied on the edge of the specimen and every specimen receives two successive 10 s flame impingements. In this test two sets of specimens (5 units each) are tested: one set is quick-aged at 70 ± 2ºC for 168 ± 2h and the other is not quick-aged (original specimens). The parameters checked during the test are: i) afterflame and afterglow times, both obtained once the burner is removed, ii) flame front propagation and iii) dripping or material detachment from the specimen. Considering the test results, the material is rated into one of three classes: V-0, V-1 or V-2. Table 2 shows the classification criteria for V-0, V-1, and V-2.

Table 2. Flammability rating UL94 vertical burning test

CRITERIA CONDITIONS	V-0	V-1	V-2
Afterflame time for each individual specimen (t1 or t2)	≤ 10 s	≤ 30 s	≤ 30 s
Total afterflame time for any condition set (t1 plus t2 for the 5 specimens)	≤ 50 s	≤ 250 s	≤ 250 s
Afterflame plus afterglow time for each individual specimen after the second flame application (t1 + t2)	≤ 30 s	≤ 60 s	≤ 60 s
Afterflame or afterglow of any specimen up to the holding clamp	No	No	No
Cotton indicator ignited by flaming particles or drops	No	No	Yes

The UL94 horizontal rating (HB) requires plastic specimens to be horizontally orientated, and then they are subjected to a defined flame ignition source for a specified period:

- A 50 W calibrated flame is applied to the edge of the specimen.
- Every specimen receives one 30 s impingement.
- One set of specimens (3 units) is tested.
- The parameters checked during the test are: i) burned length, and ii) elapsed time to travel the burned length.

A material with a thickness between 3 and 13 mm is classified as a HB material if it does not have a burning rate exceeding 40 mm per minute. For materials with a thickness less than 3 mm the burning rate should not

*7 https://omnexus.specialchem.com/polymer-*properties*/properties/flammability-ul94#horizonatal-burning*

exceed 75 mm per minute. The material will also be rated as HB if it ceases to burn before the 100 mm mark, independent of thickness (Table 3).

Table 3. Flammability rating UL94 horizontal burning test (HB)

TEST CRITERIA	BURNING RATE	FLAMMABILITY RATING
Test specimen thickness 3-13 mm	≤ 40 mm/min	HB
Test specimen thickness < 3 mm	≤ 75 mm/min	HB
Flame is extinguished before first mark	= 0 mm/min	HB

An X-ray fluorescence (XRF) analyser, *Vanta C Series*, with a dedicated fully shielded workstation was used to determine the average concentration of bromine in the purified PS. Several specimens moulded from the material were analysed.

3 MATERIAL CHARACTERISATION

3.1 CHARACTERISATION RESULTS (GAIKER)

The purified PS was characterisided by GKR according to the specifications and test methods listed in Table 1. Previously, the material was processed by injection moulding at 170 °C to obtain "dog bone" specimens that are required for testing (Figure 2).

Figure 2. PS "dog bone" specimens

The technical specifications of the purified PS are shown in Table 4.

Table 4. Characterisation results (GKR)

TECHNICAL SPECIFICATION	TEST METHOD	AVERAGE VALUE / CLASS	STANDARD DEVIATION
Density	UNE-EN ISO 1183	1.053 g/cm^3	0.0004 g/cm^3
Melt mass-flow rate*	UNE-EN ISO 1133	14.9 g/10 min	0.056 g/10 min
Melt volume-flow rate*		15.15 cm^3/10 min	0.286 cm^3/10 min
Flexural modulus**	UNE-EN ISO 178:2020	2680 MPa	20.74 MPa
Flexural strength**		48.1 MPa	1.215 MPa
Tensile modulus of elasticity**	UNE-EN ISO 527-2/1A/1:2012	3168 MPa	8.94 MPa
Tensile strength**		34.3 MPa	0.50 MPa
Charpy impact	UNE-EN ISO 179-1/1eU:2011	4820 J/m^2	128 J/m^2

Technical specification	Test method	Average value / class	Standard deviation
Flammability rating	UL94 vertical burning test (V-0, V-1 or V-2): not quick-aged set	V-2	Not applicable
	UL94 vertical burning test (V-0, V-1 or V-2): quick-aged set	V-2	Not applicable
	UL94 horizontal burning test (HB): not quick-aged set	HB	Not applicable
Br concentration* **	X-ray fluorescence	0.57 %	0.0012 %

(*) Test conditions: Temperature = 160 °C, Mass = 5 kg; (**) Five specimens tested; (***) Eight specimens analysed

The results of the flammability tests are shown in detail below (Table 5, Table 6 and Table 7):

UL94 vertical burning test (V-0, V-1 or V-2):

As mentioned above, two sets of specimens were tested. The first set was not subjected to a quick-aging process (at 70 ± 2ºC for 168 ± 2h) and the second one was.

Table 5. UL 94 vertical burning test – not quick-aged set

Specimen	Width (mm)	Thickness (mm)	Afterflame time after first flame application, t1 (s)	Afterflame time after second flame application, t2 (s)	Afterglow time after second flame application, t3 (s)
1	13.25	4.18	-	1	-
2	13.18	4.56	-	-	-
3	13.06	4.12	2	-	-
4	13.15	4.05	2	-	-
5	13.04	4.30	2	-	-
Total afterflame time = 7 s					
(-) No flame was observed					
Cotton indicator burns in every specimen due to flammable drops					
The specimens do not burn up to the holding clamp					
Classification: V-2					

Table 6. UL 94 vertical burning test – quick-aged set

Specimen	Width (mm)	Thickness (mm)	Afterflame time after first flame application, t1 (s)	Afterflame time after second flame application, t2 (s)	Afterglow time after second flame application, t3 (s)
1	13.20	4.15	2	-	-
2	13.13	4.25	-	-	-
3	13.20	4.15	-	-	-
4	13.04	4.26	-	-	-
5	13.18	4.34	-	-	-
Total afterflame time = 2 s					
(-) No flame was observed					
Cotton indicator burns in every specimen due to flammable drops					
The specimens do not burn up to the holding clamp					
Classification: V-2					

UL94 horizontal burning test (HB):

A not quick-aged set of 3 specimens was used to determine the flammability rating of the material according to the UL 94 horizontal burning test.

Table 7. UL 94 horizontal burning test – not quick-aged set

Specimen	Width (mm)	Thickness (mm)	Burned length (mm)	Elapsed time (mm)
1	12.96	4.22	-	-
2	13.20	4.12	-	-
3	13.12	4.24	-	-
Burning rate = 0 mm/min				
(-) No flame was observed				
All the specimens shed flammable drops				
Classification: HB				

3.2 CHARACTERISATION RESULTS (ICT)

The purified PS was also characterised by ICT, using the same test standards as at GKR. In particular, ICT determined the density, melt volume-flow rate and mechanical properties (flexural, tensile and Charpy impact properties) of the material. The test results are shown in Table 8.

Table 8. Characterisation results (ICT)

TECHNICAL SPECIFICATION	TEST METHOD	AVERAGE VALUE	STANDARD DEVIATION
Density	UNE-EN ISO 1183	1.053 g/cm^3	---
Melt mass-flow rate*	UNE-EN ISO 1133	4.97 g/10 min	---
Melt volume-flow rate*		5.11 cm^3/10 min	---
Flexural modulus	UNE-EN ISO 178:2020	3066 MPa	21 MPa
Flexural strength		59.3 MPa	0.7 MPa
Tensile modulus of elasticity	UNE-EN ISO 527-2/1A/1:2012	2992 MPa	45 MPa
Tensile strength		30.48 MPa	0.32 MPa
Charpy impact	UNE-EN ISO 179-1/1eU:2011	6.02 kJ/m^2	0.12 kJ/m^2

(*) Test conditions: Temperature = 160 °C, Mass = 2.16 kg

4 CONCLUSIONS

The characteristics to be considered for labelling recycled plastics (Table 1) have been analysed for purified PS. The analytics (density, melt volume-flow rate and mechanical properties) have been performed in parallel by GKR and ICT following the same standards. The datasheet of this material is summarised in Table 9.

Table 9. Characterisation results (comparison GKR - ICT)

TECHNICAL SPECIFICATION	TEST METHOD	AVERAGE VALUE / CLASS (GKR)	AVERAGE VALUE (ICT)
Density	UNE-EN ISO 1183	1.053 g/cm^3	1.053 g/cm^3
Melt mass-flow rate	UNE-EN ISO 1133	14.9 g/10 min*	4.97 g/10 min**
Melt volume-flow rate		15.15 cm^3/10 min*	5.11 cm^3/10 min**
Flexural modulus	UNE-EN ISO 178:2020	2680 MPa	3066 MPa
Flexural strength		48.1 MPa	59.3 MPa
Tensile modulus of elasticity	UNE-EN ISO 527-2/1A/1:2012	3168 MPa	2992 MPa
Tensile strength		34.3 MPa	30.48 MPa
Charpy impact	UNE-EN ISO 179-1/1eU:2011	4.82 kJ/m^2	6.02 kJ/m^2
Flammabiltiy rating	UL 94 vertical burning test	V-2	---
	UL 94 horizontal burning test	HB	---
Br concentration	XRF	0.57 %	---

(*) Temperature = 160 °C, Mass = 5 kg; (**) Temperature = 160 °C, Mass = 2.16 kg

Comparing the characterisation results between GKR and ICT, the following aspects can be summarised:

- The measured density of the samples is nearly identical.
- The measured melt flow rate is different. This is due to the different test conditions between the laboratories. The higher melt flow rate values measured by GKR are due to the higher load used during the measurement.
- The mechanical properties of the samples show a slight difference; however, the deviation is less than 20 %. This difference may result from different characterisation conditions (speed, mounting, clamping, etc.) and is uncritical.

Generally, the results are in a good accordance and the values comprehensible.