

POLYMATERIAL LTD.
LMPERIAL COLLEGE LONDON I-HUB WHITE CITY CAMPUS, 84 WOOD LANE, LONDON W12
0BZ UNITED KINGDOM

Sample Description : BPE (BIODEGRADABLE PE)
 Polymateria Sample ID : JJ1200(+)
 Material Type : BPE
 Material Composition : BPE
 Supplier : SHENZHEN HENGCHANGTAI TECHNOLOGY CO., LTD.

Sample Receiving Date : JUL. 05, 2023
 Testing Period : JUL. 05, 2023 TO AUG. 28, 2023

Test Items	PAS 9017 Requirement	Sample	Conclusion
Polyolefin Product Category	Annex B	BPE	/
Carbonyl Index	>1	1.54	PASS
Number Average Molecular Weight (Mn)	<5000 Da	1560 Da	PASS
Higher Weight Average Molecular Weight (Mz)	<30000 Da	9009 Da	PASS
% Loss of Weight Average Molecular Weight (Mw)	>90%	95.0%	PASS
Seedling Emergence and Seedling Growth Test	OECD 208	-	VALID
Daphnia magna Reproduction Test	OECD 211	-	VALID
Earthworm Reproduction Test	OECD 222	-	VALID
Soil Biodegradation Testing	>90%	94.28%	PASS

Remark:

1. Results are in accordance with the eco-toxicity requirements of OECD 208, 222, and 211 standards and were carried out at the Research Centre for Toxic Compounds in the Environment (RECETOX) which can be found in the Ecotoxicity Statement provided and in line with the requirements specifies in PAS9017.
2. Results are in accordance with the requirements in PAS9017 and were carried out at AIMPLAS in line with ISO 17556, to which the test sample achieved 94.28% biodegradation in 278 days. The data can be found in "AIMPLAS report AST-21-097-06"
3. GB/T 22047-2008 is equivalent to ISO 17556-2003 "Plastics--Determination of the ultimate aerobic biodegradability in soil by measuring the oxygen demand in a respirometer or the amount of carbon dioxide evolved", the technical content is exactly the same.

Signed for and on behalf of
 SGS-CSTC Standards Technical Services (Shanghai) Co., Ltd.

Melody Zhang
 Authorized Signatory



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Check details as below:

TEST ITEM	REPORT NO.	ISSUED DATE	LAB	STANDARD	RESULT
Soil Biodegradation Testing	AST-21-097-06	07/02/2023	AIMPLAS	ISO17556	VALID

Testing Procedure:

The UV weathering parameters followed should be in accordance with the specifications stated in PAS 9017:2020 accordance with ASTM D4329 / ISO 4892-3.

Test Condition:

Lamp type: UVA-340

UV Irradiance: 1h UV at (60±2)°C BPT, 23h condensation at (60±2)°C BPT

Irradiance: (0.80±0.02) W/(m²•nm) @340nm

Total test time of Exposure duration: 336h

Sample Frequency:

The sample frequency has been stated in hours which relate to the elapsed test time of the UV apparatus. See table below.

At each sample interval, one sample (2.5" by 5.5" holder) of each material needs to be removed from the UV apparatus and placed in a bag, analysis is carried out where stated. The bags containing the samples should be labelled with the material ID and time interval removed ready to send to Polymateria.

Frequency (time = hours)	Material
0	IR & GPC
72	IR
168	IR
240	IR
336	IR & GPC

Sample Analysis:

1.FTIR

A Thermo Nicolet IS10 FTIR, equipped with an Attenuated Total Reflectance (ATR) attachment was used for the infrared analysis of the specimens.

ATR is a sampling technique used in conjunction with infrared spectroscopy which enables samples to be examined directly in the solid or liquid state without further preparation. Specimens were tested without any previous preparation straight after conditioning. The FTIR is annually calibrated, serviced and maintained by Thermo fisher engineers. In total three specimens per material per conditioning time were tested using FTIR. The FTIR testing parameters used were as follows:

- Wavenumber range: 550 cm⁻¹ to 4000 cm⁻¹
- Scanning resolution: 4 cm⁻¹
- Number of scans: 32 scans
- Number of background scans: 32 scans, before every sample.

From the spectra collected in FTIR tests, the carbonyl peaks were identified and the carbonyl index, CI was calculated as follows:



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$$CI = \frac{\text{Peak area of the carbonyl peak around } 1714\text{cm}^{-1}}{\text{Peak area of the } CH_2 \text{ scissoring peak around } 1470\text{cm}^{-1}}$$

When calculating the carbonyl index the following peak regions were used:

- For the carbonyl peak = 1650 cm^{-1} to 1850 cm^{-1}
- For the CH_2 peak = 1420 cm^{-1} to 1500 cm^{-1}

2.GPC

A single solution of each sample was prepared by adding 10 mL of solvent to 20 mg of sample. The sample solutions were heated and shaken at 160°C for 4 hours, or until dissolved. The vials were placed in an autosampler where injection of part of the contents of each vial was carried out automatically. All masses, volumes, times, and temperatures in this section are only approximate.

- Instrument: Agilent Technologies PL GPC220,
- Columns: PLgel Olexis
- μm , Solvent: 1, 2, 4-trichlorobenzene with anti-oxidant,
- Flow-rate: 1.0 mL/minute (nominal),
- Temperature: 160°C (nominal),
- Detector: refractive index with differential pressure and light scattering.

Data capture and subsequent data handling was carried out using Agilent Technologies 'Cirrus' software.

The GPC system used for this work was calibrated using a series of Agilent/Polymer Laboratories EasiVial PS-H polystyrene calibrants with known molecular weights but a mathematical correction, making use of Mark-Houwink parameters has been applied to express the results as for linear polyethylene or polypropylene homopolymer (the Mark Houwink parameters used are identified in table below).

Mark Houwink Parameters	Polystyrene (calibrants)	PP
Alpha	0.65	0.725
$K \times 10^{-5} \text{ dL/g}$	19	19

The samples appeared to be completely soluble. There were no apparent problems with either the filtration or the chromatography of any of the solutions.



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Test Result:
1. Average Carbonyl Index (CI)

Sample	Average Carbonyl Index (CI) after Light Ageing Test-UV Exposure				
	0h	72h	168h	240h	336h
Sample	0.05	0.04	0.78	0.97	1.54
STDVA	0.01	0.01	0.02	0.01	0.03

 Note: Carbonyl Index (CI)= (Area under band 1850-1650 cm⁻¹)/(Area under band 1500-1420 cm⁻¹)

Appendix – FTIR data

	Run	Sample		Carbonyl Index (CI)
		Area under band 1850-1650 cm ⁻¹	Area under band 1500-1420 cm ⁻¹	
Original sample	1	0.18	3.02	0.06
	2	0.12	3.01	0.04
	3	0.13	3.08	0.04
	AVERAGE	0.14	3.04	0.05
	STDVA	0.03	0.04	0.01
UV Exposure 72h	1	0.10	3.21	0.03
	2	0.14	3.24	0.04
	3	0.16	3.20	0.05
	AVERAGE	0.13	3.22	0.04
	STDVA	0.03	0.02	0.01
UV Exposure 168h	1	2.23	2.81	0.79
	2	2.19	2.87	0.76
	3	2.37	2.98	0.80
	AVERAGE	2.26	2.89	0.78
	STDVA	0.09	0.09	0.02
UV Exposure 240h	1	2.70	2.79	0.97
	2	2.40	2.47	0.97
	3	2.90	2.95	0.98
	AVERAGE	2.67	2.74	0.97
	STDVA	0.25	0.24	0.01
UV Exposure 336h	1	4.76	3.02	1.58
	2	4.45	2.93	1.52
	3	4.71	3.07	1.53
	AVERAGE	4.64	3.01	1.54
	STDVA	0.17	0.07	0.03

2. Molecular weight analysis

	Mw original	Mw 336h	Mn original	Mn 336h	Mz original	Mz 336h	Mw loss (%)	Mw/Mn(P D) original	Mw/Mn(P D) 336h
Sample	89995	4476	26372	1560	218627	9009	95.0%	3.41	2.87
STDVA	1473	143	4282	277	16919	600	/	/	/



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Appendix – GPC data

Condition	Run	Sample
Mw, No conditioning	1	90137
	2	88456
	3	91392
	AVERAGE	89995
	STDVA	1473
Mw,336h UV weathering	1	4552
	2	4311
	3	4564
	AVERAGE	4476
	STDVA	143
Mn, No conditioning	1	21578
	2	29816
	3	27721
	AVERAGE	26372
	STDVA	4282
Mn,336h UV weathering	1	1241
	2	1697
	3	1741
	AVERAGE	1560
	STDVA	277
Mz, No Conditioning	1	231793
	2	199544
	3	224543
	AVERAGE	218627
	STDVA	16919
Mz,336h UV weathering	1	9521
	2	8349
	3	9156
	AVERAGE	9009
	STDVA	600

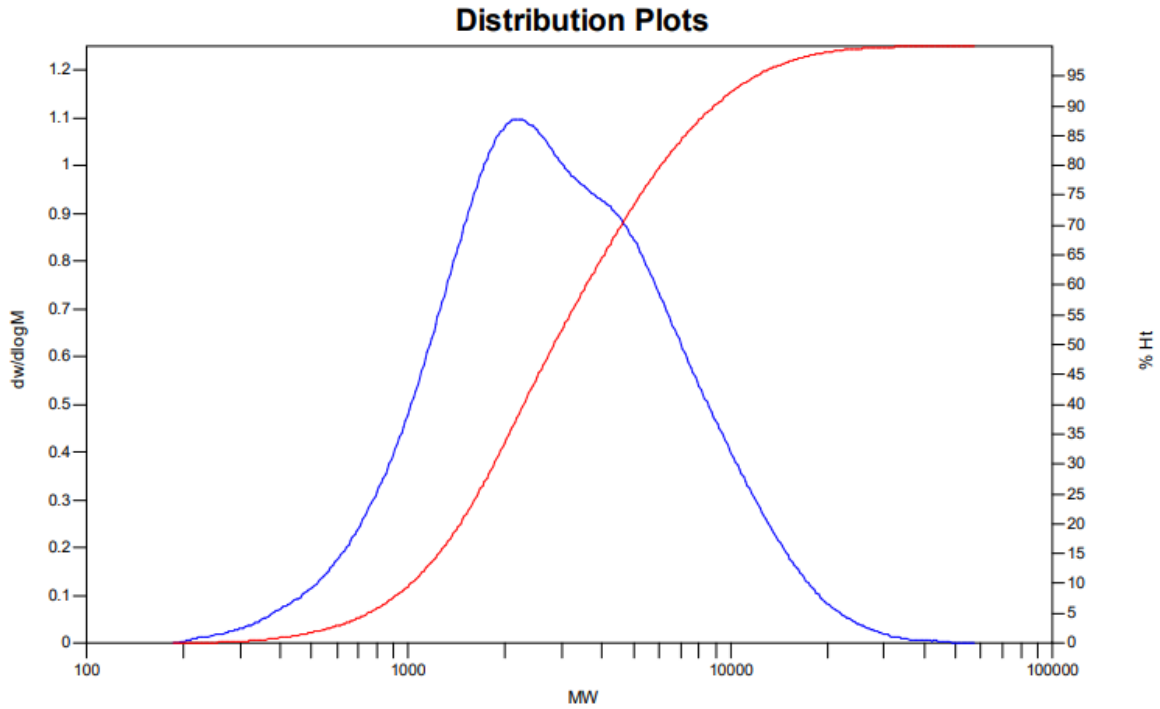
Note: the GPC test was subcontracted to GZ SPECIAL PRESSURE EQUIPMENT INSPECTION AND RESEARCH INSTITUTS



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GPC distributions:

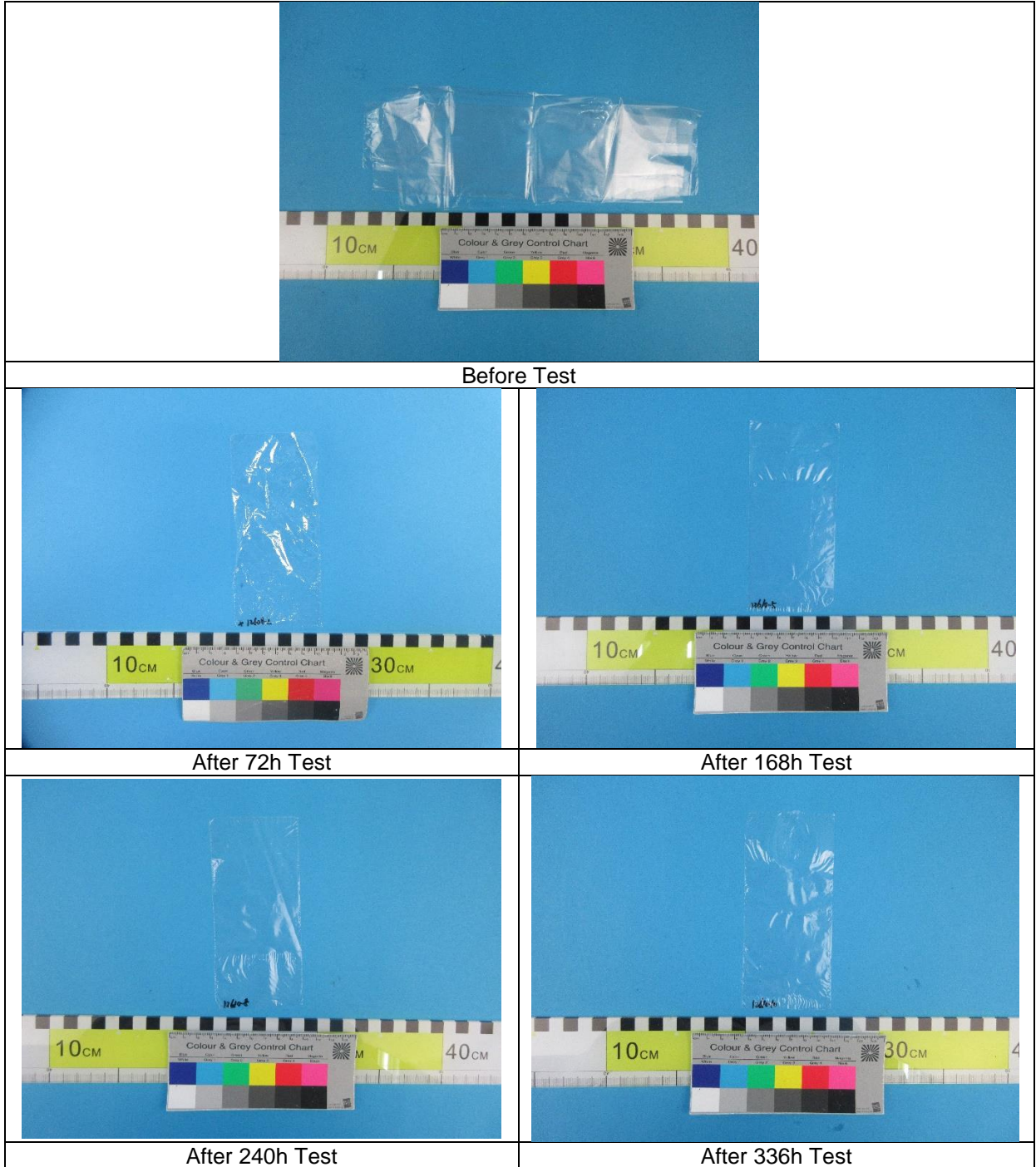


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Research Centre
for Toxic Compounds
in the Environment

1

Ecotoxicity Statement

This statement confirms that plastic products biotransformed by Polymateria's additive technologies, specified hereunder, have met all ecotoxicity criteria set out by BS PAS 9017:2020 for:

- **Freshwater aquatic and;**
- **Soil invertebrates and;**
- **Plant eco-toxicity**

And can thus be considered safe for contact in these environments.

The testing and reporting were conducted at RECETOX, a third-party laboratory ISO17025:2005 accredited for chemical trace analysis and conducting the research, development and utilization of environmental eco-toxicity testing.

Persons responsible for the ecotoxicological testing and reporting – Mgr. Jana Vašíčková, Ph.D. & Mgr. Marek Šudoma, Ph.D.

Study dates: 22nd December 2020 - 20th February 2021




Place of study - Research Centre for Toxic Compound in the Environment (RECETOX), Faculty of Science, Masaryk University, Brno, Czech Republic.

Biotransformed products in study:

Sample ID	Description of the material tested	PAS 9017 Annex B category
Product 8	Plastic bottle (PP) containing Masterbatch formulation No. 1 of Cycle+	Polypropylene (+ BA)
Product 9	Plastic film (PE) containing Masterbatch formulation No. 1 of Cycle+	Polyethylene (+ BA)

Biotransformed products met criteria for:

- Chronic toxicity:

OECD 208	OECD 211	OECD 222
		
Terrestrial plants	<i>Daphnia</i> sp.	Earthworm

OECD 208: Terrestrial plant test (*Sinapis alba* and *Hordeum vulgare*): Terrestrial Plant Test: Seedling, Emergence and Seedling Growth Test

OECD 211: *Daphnia magna* Reproduction Test (freshwater aquatic eco-toxicity test).

OECD 222: Earthworm reproduction test (*Eisenia fetida*).



Research Centre
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in the Environment

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Conclusion: The trial concluded that the biotransformed plastic products (PP and PE) containing Polymateria's technologies (Cycle+) have met the criteria for all ecotoxicity tests required to reach BS PAS 9017 specification and can thus be considered safe for contact in soil, plant and water environments.

Professor Luděk Bláha
Deputy Director of RECETOX

Signature..... 

In Brno, CZ, on date 30th April, 2021

Stamp:

MASARYKOVA UNIVERZITA
Přírodovědecká fakulta
Centrum pro výzkum
toxických látek v prostředí
Kotlářská 2, 611 37 Brno



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* The marked activities are not covered by the ENAC accreditation.

TEST CERTIFICATE		Number: AST-21-097-06 Date of tests: 03/02/2022-09/11/2022 Certificate issue: 07/02/2023
COMPANY	POLYMATERIA LTD Imperial College i-Hub White City Campus 80 Wood Lane W120BZ LONDON UNITED KINGDOM	
PETITIONER	Florence Huynh	
PRODUCT NAME	PP_PVM_25072021_559_W - Description: Polypropylene sample from a polypropylene product, containing Polymateria Technology at 2% loading rate (wt%), manufactured by [REDACTED] - Code: AST-21-097-06	
TESTS PERFORMED ACCORDING TO STANDARDS OR INTERNAL PROCEDURES	Determination of the ultimate aerobic biodegradability of plastic materials in soil under controlled soil conditions according to the UNE-EN ISO 17556:2019 standard.	
RESULTS	According to reports: AST-21-097-6-EN/1	

SAMPLE	
	Customer's reference PP_PVM_25072021_559_W Polypropylene sample from a polypropylene product, containing Polymateria Technology at 2% loading rate (wt%), manufactured by [REDACTED]
	AIMPLAS code AST-21-097-06
	Description Yellowish crushed
	Reception date 19/01/2022



TEST CERTIFICATE	Number: AST-21-097-06 Date of tests: 03/02/2022-09/11/2022 Certificate issue: 07/02/2023
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Test method

Standard: Determination of the ultimate aerobic biodegradability of plastic materials in soil under controlled soil conditions according to the UNE-EN ISO 17556:2019 standard.

Method: The determination of the aerobic biodegradability of the plastic material samples has been carried out under controlled soil conditions.

The test method used is based on the method reflected in the UNE-EN ISO 17556:2019 standard and is based on the measurement of carbon dioxide generated by gravimetry.

In the present test, the soil used is a standardized soil and the material to be analyzed is tested in a size of 20x20 mm.

2500 mg (dry solids) of sample are mixed with 300 g (dry solids) in each 3-liter capacity reactor and intensively maintained under optimum oxygen conditions, temperature 25 ± 2 ° C and humidity (optimum water content between 40% and 60% of the total water holding capacity) for a test period of six months, extendable to 2 years under specific circumstances.

Air was supplied into the vessels to ensure aerobic conditions throughout the test. Furthermore, the test vessels were subjected to stir twice a week in order that the air distribution was homogeneous throughout the vessel.

The test vessels were distributed as follows:

- Three reactors for the blank (compost).
- Three reactors for reference material (cellulose).
- Three reactors for each test material.

During the aerobic biodegradation of the material, the amount of carbon dioxide generated in each of the reactor is continuously recorded and analyzed by an IR detector and the flow rate is determined by an on-line flowmeter. The percentage of biodegradation is calculated as the ratio of the carbon dioxide generated from the test material to the theoretical maximum amount of carbon dioxide in the test material (ThCO₂).

The amount of carbon dioxide generated is absorbed in a column and measured at intervals of time by weighing the grams of CO₂ retained using a precision electronic scale. The percentage of biodegradation is calculated by the relationship between the carbon dioxide generated from the test material and the maximum theoretical amount of carbon dioxide (ThCO₂) that can be produced from the test material.

Standard: Determination of Total Nitrogen (Nt)^o

- Method:**
- Compost matrix: Internal procedure PI-LTL-6.038.
 - Plastic matrix: Internal procedures B-N211 and B-N214 according to ASTM D5291



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Test method D for matrices: Biomass (wood, pruning residues, sludge, manure, residues from the agricultural, wood and paper industries), solid biofuels and for Petroleum Products

Standard: Determination of Total Organic Carbon (COT)^d
 Method: - Compost matrix: Internal procedure PI-LTL-6.081 (Oxid. Dichromate)
 - Plastic matrix: Internal procedures B-N211 and B-N214 according to ASTM D5291

Test method D for matrices: Biomass (wood, pruning residues, sludge, manure, residues from the agricultural, wood and paper industries), solid biofuels and for Petroleum Products

^c Subcontracted test covered by ENAC accreditation N° 121/LE1782 in compost matrix.
^d Subcontracted test covered by accreditation RvA N° 135 in plastic matrix.

Results

Start date: 03/02/2022
 Finish date: 09/11/2022

1) IDENTIFICATION AND CHARACTERIZATION OF PARAMETERS

- Normalised soil composition

Normalised soil	Theoretical amount (g dry mass/reactor)
Amount of normalized soil/ reactor	200,000
Quartz sand	140,000
Clay	20,000
Natural soil	32,000
Compost	8,000
KH ₂ PO ₄	0,040
MgSO ₄	0,020
NaNO ₃	0,024
Urea	0,012
NH ₄ Cl	0,024



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2) TEST QUANTITIES

QUANTITY OF CARBON DIOXIDE AND CALCULATION OF THE PERCENT OF BIODEGRADABILITY

The following tables and figures show the evolution of CO₂ production and degree of biodegradation of the vessels containing the blank, the reference material and the test sample, respectively.

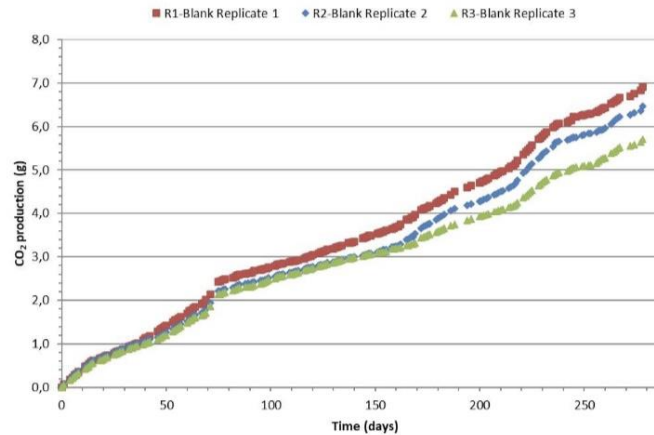


Figure 1. Evolution of CO₂ production (for each replica) of the blank

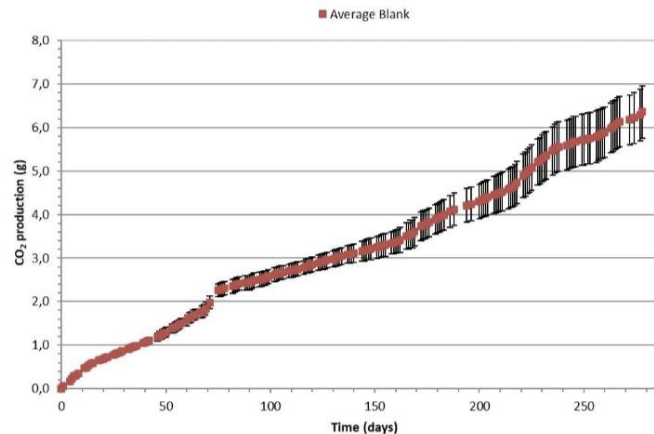


Figure 2. Evolution of CO₂ production (average value) of the blank



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- Reference (cellulose) results

The graphical evolution of CO₂ production and the percentage of biodegradation (for each replica) and the average percentage of biodegradation of the reference material are shown below:

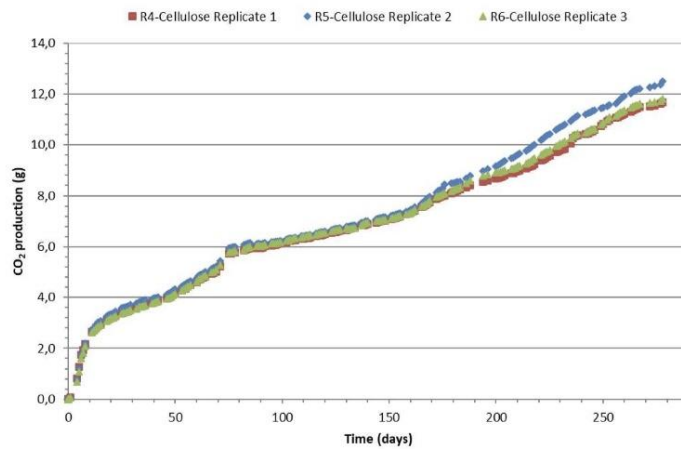


Figure 3. Evolution of CO₂ production (for each replica) of the reference material (cellulose)

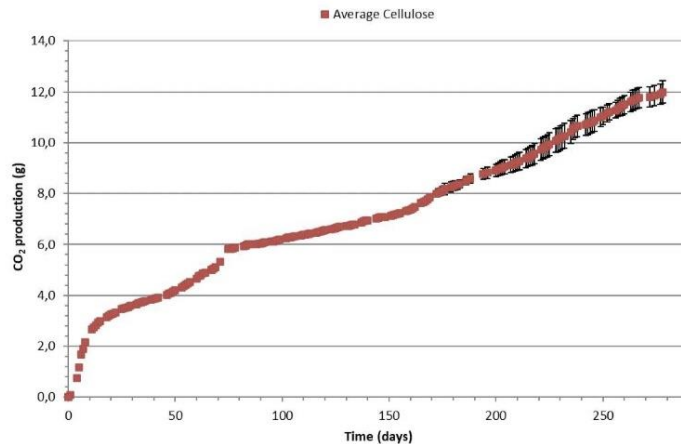


Figure 4. Evolution of CO₂ production (average value) of the reference material (cellulose)



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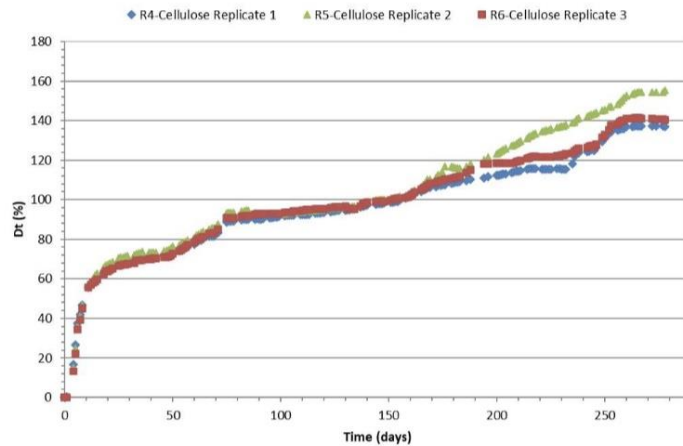


Figure 5. Evolution of the percentage of biodegradation (for each replica) of the reference material (cellulose)

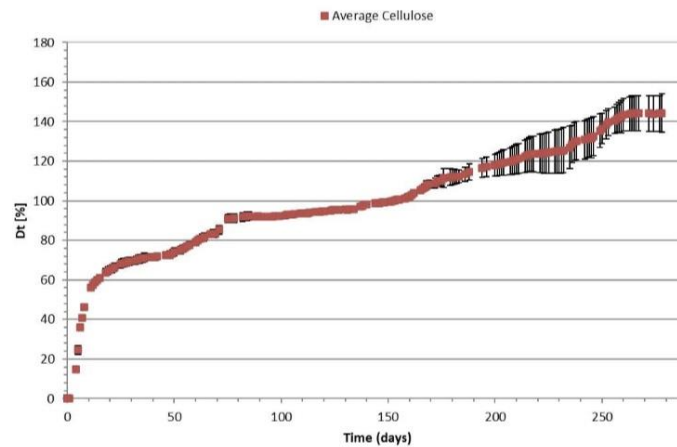


Figure 6. Evolution of the percentage of biodegradation (average value) of the reference material (cellulose)



TEST CERTIFICATE Number: AST-21-097-06
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• **Results sample AST-21-097-06**

The graphical evolution of CO₂ production and the percentage of biodegradation (for each replica) and the average percentage of biodegradation of the reference material are shown below:

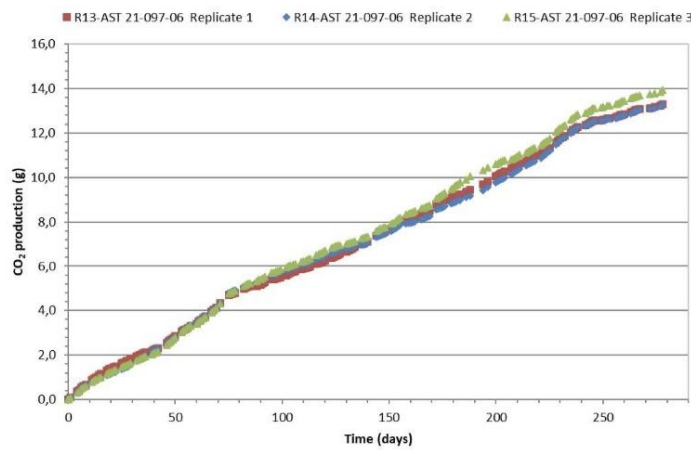


Figure 7. Evolution of CO₂ production (for each replica) of sample AST-21-097-06

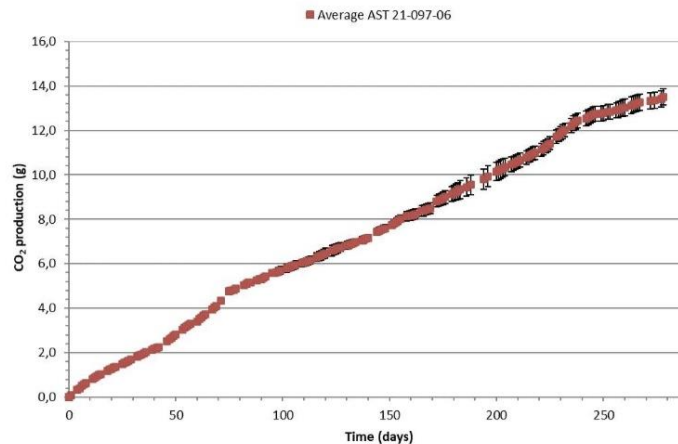


Figure 8. Evolution of CO₂ production (average value) of sample AST-21-097-06



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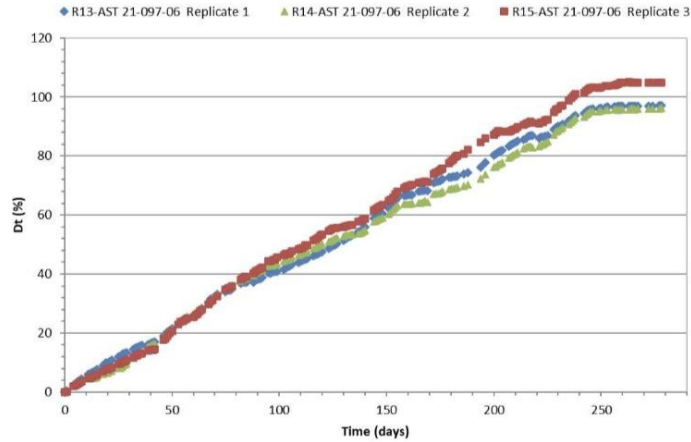


Figure 9. Evolution of the percentage of biodegradation (for each replica) of sample AST-21-097-06

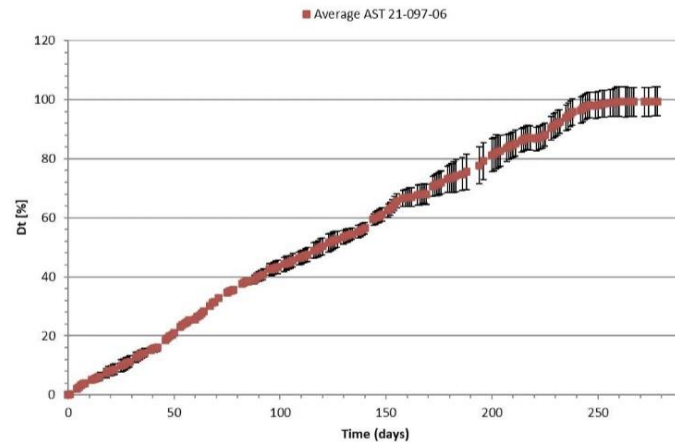


Figure 10. Evolution of the percentage of biodegradation (average value) of sample AST-21-097-06



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3) MAINTENANCE OPERATIONS CARRIED OUT DURING THE TEST AND AT THE END OF THE TEST

- Temperature control during the test
- Humidity control and other maintenance operations during the test

Water is added to each reactor during the stirring process to maintain the total weight of the reactors, and thus, to maintain the optimum humidity in the mixture.

- pH control

4) VALIDITY OF THE TEST

In accordance with the UNE-EN ISO 17556:2019 standard, the test is considered valid if the following criteria are met:

- (a) The degree of biodegradation of the reference material is greater than 60% in the plateau phase or at the end of the test.
- (b) The DBO or carbon dioxide in three blanks are at 20% in the plateau phase or at the end of the test.



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***CONCLUSIONS**

Determination of the ultimate aerobic biodegradability of plastic materials in soil under controlled soil conditions according to the UNE-EN ISO 17556:2019 standard.

The percentage of biodegradation average value of the samples after 278 days of testing, is shown in the following table:

Sample	D _{tm} (%) 278 days
AST-21-097-06	99,40
Standard requirement	>90%

To demonstrate that a product is biodegradable under controlled soil conditions according to this standard, 90% biodegradation has to achieved within a minimum of 6 months and a maximum of 24 months.

Electronic signature of the authorized signatories:

NEUS(SORIANO)MARCÓ
 Material Characterization & Testing Group Leader
 2023.02.08 12:04:02 +01'00'



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