DigInTraCE

Fingerprints and markers for recycled material content v1

D5.6

DigInTraCE

A Digital value chain Integration Traceability framework for process industries for Circularity and low Emissions by waste reduction and use of secondary raw materials

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03	23/05/2024	Jorge Barrio Arbex & German Mancheno Sanz (CIRCE)	Input CIRCE on HSI
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06	25/06/2024	Ruben Geerinck (CTB) & Jorge Barrio Arbex (CIRCE)	Final version

Legal Disclaimer

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List of abbreviations and acronyms

Abbreviation	Meaning
DSC	Differential Scanning Calorimetry
GC	Gas chromatography
GCMS	Gas chromatography–mass spectrometry
IPA	Isophthalic Acid
NIAS	Non Intentionally Added Substance
PET	Polyethylene Terephthalate
HSI	Hyperspectral imaging
NIR	Near Infrared Range
ML	Machine Learning
SSP	Solid State Polymerization
TGA	Thermogravimetry
VOC	Volatile organic content

Executive Summary

This deliverable D5.6 is part of Work package 5 “Technologies for secondary materials upgrade and process optimization”, more specifically task T5.4 Fingerprints and markers for recycled material content. It is the first version of the work done in this task, and will be updated in a final version D5.7 to be submitted in M42. Within this task of DigInTraCE, the goal is to identify ‘fingerprints or markers’ that distinguishes recycled polyester from virgin polyester. Two pathways are being researched: (i) detailed chemical analysis (CTB) & (ii) use of hyperspectral imaging (CIRCE). CTB performed controlled aging to simulate lifetime of polyester products and trigger physical and chemical changes in the material. It was found that many physical properties change, however it remains difficult to associate (and quantify) these changes with recycled content. Secondly, CTB also tested various recycled grades, but no chemical marker was found. CIRCE used hyperspectral imaging, but the spectral fingerprint was found to be very similar. Next steps will focus on testing new samples with known recycled content, and further exploring the analytical techniques.

1. Introduction

1.1. Project intro

DigInTraCE project aims to develop a transparent and interoperable Decentralized Traceability platform using innovative tracking, sensing, and sorting techniques. The project also focuses on the dynamic updating of Digital Product Passport (DPP) schemes that support certification and quality validation. Furthermore, AI-based decision-making mechanisms are being developed to optimize processes and lifecycles. The project also aims to improve the utilization of secondary raw materials through up-cycling, reuse, and upgrade technologies. Additionally, DigInTraCE aims to contribute to standardization efforts and ensure open and easily accessible data. The project also explores the development of new business models that create economic opportunities and promote digital skills while addressing regional social needs.

The solutions developed within DigInTraCE will reach a Technology Readiness Level (TRL) of 6 by the project's conclusion. The project's solutions will be demonstrated in two sectors: Pulp & Paper, specifically in composite wood and furniture; and Chemicals, focusing on plastic parts for Information and Communication Technology (ICT) equipment and the automotive market, as well as polymers and textiles.

Overall, DigInTraCE is driven by six objectives that outline the project's trajectory. The first objective (O.1) entails designing, developing, and implementing solutions that promote the optimal utilization of secondary raw materials and minimize waste within circular value chains. The second objective (O.2) focuses on the development and demonstration of innovative concepts for material tracing, facilitated by a decentralized digital platform enabling the tracing and certification of secondary raw materials. Moving on, the third objective (O.3) involves the design and delivery of cutting-edge real-time sensing and sorting mechanisms, which enhance data exchange through a dynamic DPP. Additionally, the fourth objective (O.4) aims to enhance accessibility to crucial material data regarding composition and origin. This will be achieved through the utilization of smart tags, smart contracts, open software, and immersive technologies. The fifth objective (O.5) centers on validating the efficacy of DigInTraCE technologies across four distinct value chains. Lastly, the sixth objective (O.6) empowers local and regional entities by actively involving them in the development of educational resources for both workplaces and educational institutions. This collaborative effort not only fosters the adoption of DigInTraCE solutions within the broader community but also facilitates knowledge transfer to maximize the project's impact.

1.2. Deliverable introduction

The increasing emphasis on sustainability and circular economy principles has driven a surge in the use of recycled materials in general, as well as the use of recycled polyester (polyethylene terephthalate (PET) in textile industry in particular. Recycled polyester in textile industry is often sourced from post-consumer PET bottles or post-industrial textile waste, and this plays a crucial role in reducing dependency on virgin resources, minimizing waste, and lowering environmental footprints. However, ensuring the authenticity and quantification of recycled content in polyester products remains a significant challenge.

Quantifying the recycled content from post-consumer waste plastics through analytical methods remains a significant challenge due to the complexity and variability of the materials involved. As a result, the industry relies on third-party auditing organizations such as QA-CER[1] to certify the recycled content in products. These organizations perform detailed audits and assessments of recycling processes and supply chains to provide trusted certificates that verify recycled content. This certification process ensures transparency and reliability, helping manufacturers and consumers trust the sustainability claims associated with recycled plastic products. By using third-party verification, companies can confidently label their products as containing recycled materials, thus supporting the circular economy and promoting environmental sustainability. Big drawback however is the significant cost associated with these audits.

This necessitates the development of reliable analytical methods to identify and quantify recycled polyester content. Recycling PET into polyester fabric involves collecting and sorting waste materials, followed by processes like cleaning, shredding, re-polymerization and melt spinning. Despite these processes, recycled polyester can contain various degradation products, impurities, and residual monomers that potentially could serve as markers or fingerprints for recycled content. Within DigInTraCE, the goal is to identify ‘fingerprints’ that distinguishes recycled polyester from virgin polyester.

Two pathways are being researched:

- Detailed chemical analysis (CTB)
- Use of hyperspectral imaging (CIRCE)

1.3. Purpose of the deliverable

Attainment of the objectives and explanation of deviations

Table 1: Assignment of roles for D5.6.

Role:	Who:
Responsible	CTB
Drafting and input	CTB & ICCS
Internal review	CHIMAR, NTUA
Submission	ICCS

1.4. Intended audience

The intended audience for the first version of the “Fingerprints and markers for recycled material content” include:

- **Consortium partners**: The members of the DigInTraCE project consortium who have a vested interest in sustainability, recycled content and traceability.

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[1] https://www.bqa.be/en/bqa-qa-cer-recycled-content

- **Industry experts, stakeholders and end-users:** Individuals or organisations that are affiliated with the plastic and textile industry. They encompass manufacturers, suppliers, distributors, recyclers and consumers who have a direct or indirect involvement in the value chain.
- **Research and academic community**: Researchers, scientists, and scholars specialising in sustainability, circular economy, recycling, and related subjects.

1.5. Structure of the deliverable and its relation with other work packages/deliverables

This deliverable D5.6 is part of **Work package 5 "Technologies for secondary materials upgrade and process optimization"**, more specifically task T5.4 Fingerprints and markers for recycled material content. It is the first version of the work done in this task and will be updated in a final version D5.7 to be submitted in M42. Furthermore, it also links to **Work package 6 "Piloting and Demonstration"** where the obtained results will be specifically used in the Belgian case in T6.3 Establishment of the new solutions in the plastic based value chains.

The deliverable is structured to provide a thorough exploration of methods for identifying and quantifying recycled content in polyester textiles. The **introduction** section offers an overview of the project's aims, emphasizing the importance of sustainability and the role of recycled polyester in reducing environmental impact. It explains the specific objectives of this deliverable within the broader project, detailing the goals of identifying key markers for recycled polyester. The intended audience includes stakeholders, industry professionals, and researchers, ensuring the content is relevant and useful for a diverse readership. An overview of the document's layout and its connection to other project deliverables is also provided.

Following the introduction, the **literature overview** section reviews possible methods for detecting recycled content. It covers chemical analysis techniques such as Gas Chromatography-Mass Spectrometry (GC-MS) and High-Performance Liquid Chromatography (HPLC), which are used to identify degradation products and monomers in recycled polyester. The section also explores hyperspectral imaging as a non-destructive method for analyzing recycled content.

The **materials and methods** section details the materials used, including recycled polyester and reference materials, and describes the experimental procedures for both chemical analysis and hyperspectral imaging. The **results** section is divided into findings from controlled aging and chemical analysis, including key markers and degradation products, as well as rheological, mechanical, and thermal properties. It also presents results from hyperspectral imaging studies, demonstrating the technology's effectiveness in identifying recycled content.

The **conclusion and outlook** section summarizes the main findings, discusses their implications for the industry, and outlines potential future research directions. It emphasizes the importance of the developed methods for enhancing the traceability and reliability of recycled polyester content, supporting sustainability and the circular economy.

2. Literature overview markers and fingerprints in recycled PET

Markers or fingerprints in recycled plastics are specific chemical compounds or physical characteristics that can be used to identify and quantify the presence of recycled content. These markers could help differentiate recycled plastics from virgin materials and ensure the authenticity and quality of recycled products.

Different types of markers and fingerprints could be targeted:

- Chemical markers: these could be degradation products as the quality of PET can deteriorate during production, use, and recycling due to thermal degradation. Furthermore, the presence of solid contaminants and non-intentionally added substances (NIAS) could also be researched. Lastly, the presence of certain monomers or comonomers could also give information on the origin of the material.
- Physical markers: as certain physical properties (thermal, mechanical, rheological, etc.) will be shifted during the production, use and recycling phase of PET, these could potentially also help to assess recycled content and quality.

2.1. Markers and fingerprints

Chemical substances such as degradation products, NIAS, solid contaminants, monomers/comonomers will all provide information on the quality of recycled PET and could potentially also be used as marker or fingerprint to identify and quantify recycled content.

Various chemical analysis techniques to detect and monitor relevant substances in rPET are highlighted in a whitepaper from Slijkoord et al.[2]. For instance, Headspace Gas Chromatography (GC) is recommended for identifying NIAS that emerge during the melting steps of recycling. Furthermore, the whitepaper discusses the use of the GPE Partisol analysis to detect solid contaminants (gels) in rPET. It was found that more than 70% of solid particles in rPET are between 1-3 µm in size, which correlates with increased haze in PET packaging. Recommendations are provided to minimize these particles to improve transparency and reduce crystallization.

In the context of polyolefin analysis, Two-dimensional liquid chromatography (2D-LC) can be used to separate and identify the individual components of a complex mixture of polyolefins, which can be difficult to separate using traditional one-dimensional liquid chromatography (1D-LC) techniques. 2D-LC is a powerful analytical technique that combines two separate chromatographic separations to achieve high resolution and improved separation of complex mixtures. 2D-LC could also be used for tracing and qualifying of recycled materials.[3]

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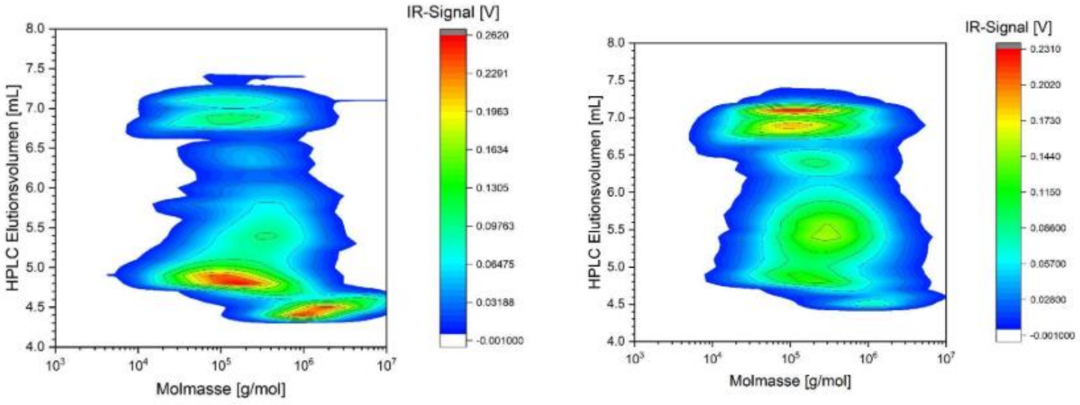
[2] How recycled PET product quality and product safety risks can be controlled, Slijkoord, J.W., Klootsema, E., Odolphy, F., Getec Park Emmen, 2020.

[3] https://www.lbf.fraunhofer.de/content/dam/lbf/de/documents/veranstaltungen/Robert%20Bruell_LBF.pdf



70 % rec

10 % rec

Figure 1: Tracing and qualifying recycled material using 2D-LC[3].

Monomers/comonomers also provide information on the origin of PET. The filed patent WO 2022/19440[4] introduces an innovative method to quantify recycled content in PET textiles by measuring the isophthalic acid (IPA) monomer content. The core innovation lies in the precise quantification of IPA, which is used as a marker to determine the amount of recycled PET in textile products. The process involves analyzing the PET material to detect and measure the presence of IPA, which in textile rPET mostly originates from bottle grade PET. The conclusions from this patent suggest that by accurately measuring the IPA content, manufacturers and researchers can reliably determine the proportion of recycled material in PET textiles. This method provides a standardized and quantifiable approach to assess recycled content, which can help in quality control, certification, and promoting transparency in the use of recycled materials in the textile industry. The approach seems promising, however two main issues are to be considered: (i) the approach relies on the presumption that IPA is solely used in virgin bottle grade PET, and not in textile grade PET and (ii) quantification of recycled content is only possible if the IPA is used in a fixed percentage in the virgin PET.

Overall, many analytical methods show promise for identifying chemical markers and fingerprints in recycled plastics. However, further research is required to analyze these techniques specifically for recycled PET from textiles, validate their accuracy, and ensure reliable and consistent identification and quantification of recycled content.

2.2. Hyperspectral imaging

The use of hyperspectral imaging (HSI) technology, particularly in the Near Infrared (NIR) spectrum, has gained significant attention in recent years due to its potential applications in various industries, including polymer identification and sorting. Polyethylene terephthalate (PET), a commonly used polymer in packaging, has been a focal point for research due to the environmental necessity of efficient recycling processes. Hyperspectral imaging combines imaging and spectroscopy to acquire both spatial and spectral information from an object. This technique captures a wide spectrum of light, enabling the detailed analysis of materials based on their spectral signatures. NIR hyperspectral

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[4] Muller de Morogues, G, Legoupil, S, Method for the determination of recycled polyethylene terephthalate, WO 2022/194408, Worms Safety Europe, 2022.

imaging, which typically covers wavelengths from 700 nm to 2500 nm, is particularly effective for identifying polymers due to the unique absorption features of different materials in this range.

Numerous studies have validated the efficacy of NIR hyperspectral imaging in polymer identification. For instance, research by Wu et al. (2020)[5] explored the separation of waste electrical and electronic equipment (WEEE) plastics from mixtures of PP, PS, ABS, and ABS/PC using NIR spectroscopy data (900-1700 nm). The study employed three classification methods: spectral angle mapper (SAM), partial least squares discriminant analysis (PLS-DA), and principal component analysis combined with linear discriminant analysis (PCA-LDA). Among these, PCA-LDA achieved the highest overall accuracy and was the least affected by preprocessing methods. SAM was more influenced by preprocessing, while PLS-DA was found unsuitable for classifying plastic waste. The study highlighted that NIR-based separation struggles with dark-colored plastics, indicating limitations that need addressing for broader applicability.

In another study by Henriksen et al. (2022)[6], the classification of plastics was investigated using short-wave infrared hyperspectral data (955-1700 nm) with unsupervised machine learning models. Techniques such as PCA and k-means clustering were applied, along with preprocessing steps like normalization and Savitzky-Golay filtering. The model successfully distinguished between twelve types of plastics, including PE, PP, PET, PS, PVC, PVDF, POM, PEEK, ABS, PMMA, PC, and PA12, demonstrating that this spectral range is sufficient for differentiating a wide array of plastics. This study underscores the potential of integrating machine learning with NIR HSI for efficient plastic sorting in recycling processes.

Further research by Peñalver et al. (2023)[7] focused on discriminating between recycled and virgin PET in water bottles using Raman spectroscopy combined with chemometrics. The study developed chemometric models using orthogonal partial least squares discriminant analysis (OPLS-DA) with Raman spectral data ranging from 600 to 1800 cm^{-1}. The resulting model achieved a success rate of 95%. Additionally, partial least squares regression (PLS) was employed to quantify the level of recycled material in PET bottles, yielding a good fit ($R^2 = 0.8920$). This research highlights the capability of Raman spectroscopy, alongside chemometric analysis, to effectively differentiate and quantify recycled content in PET products, which is crucial for ensuring material quality in recycling streams.

The existing literature highlights the increasing importance of NIR hyperspectral imaging in the identification and sorting of PET. However, this project aims to advance the current state of knowledge by investigating the feasibility of using this technology to detect specific features that enable the differentiation of various types of PET. This differentiation is crucial as it directly relates to the quality of the material, thereby enhancing the overall recycling process.

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[5] Auto-sorting commonly recovered plastics from waste household appliances and electronics using near-infrared spectroscopy, Xiaoyu Wu, Jia Li, Linpeng Yao, Zhenming Xu. Journal of Cleaner Production, Volume 246, 2020, 118732.

[6] Plastic classification via in-line hyperspectral camera analysis and unsupervised machine learning, Martin L. Henriksen, Celine B. Karlsen, Pernille Klarskov, Mogens Hinge. Vibrational Spectroscopy. Volume 118. 2022, 103329.

[7] Raman spectroscopic strategy for the discrimination of recycled polyethylene terephthalate in water bottles, Rosa Peñalver, Félix Zapata, Natalia Arroyo-Manzanares, Ignacio López García, Pilar Viñas. Journal of Raman Spectroscopy, 2023; 54: 107-112

3. Materials and methods

3.1. Materials

All virgin and recycled samples have been provided by partners SIOEN and CTB.
Table 2 gives an overview of the samples including supplier, description and performed analysis.

Table 2: Details of virgin and recycled samples

Sample name	Internal code	Description	Supplier	Analysis performed
PET 5530	T2303553	100% virgin PET	CTB	Ageing
PET 5140 nIV (vPET bssp)	T2312778	100% virgin PET before solid state polymerization	SIOEN	HSI, Ageing, thermal extraction, HR-GCMS
PET 5140 IV (vPET assp)	T2312779	100% virgin PET after solid state polymerization	SIOEN	HSI, Ageing, thermal extraction, HR-GCMS
rPET10 nIV	T2312780	100% recycled PET before solid state polymerization (10 -> waste before spinning)	SIOEN	Thermal extraction, HR-GCMS
rPET20 nIV	T2312781	100% recycled PET before solid state polymerization (10 -> waste before spinning)	SIOEN	Thermal extraction, HR-GCMS
rPET30 bssp	T2312782	100% recycled PET before solid state polymerization (30 -> waste from the weaving mills)	SIOEN	HSI, thermal extraction, HR-GCMS
rPET30 assp	T2312783	100% recycled PET after solid state polymerization (30 -> waste from the weaving mills)	SIOEN	HSI, thermal extraction, HR-GCMS
rPET30 assp bb	T2312784	100% recycled PET after solid state polymerization bad batch (30 -> waste from the weaving mills)	SIOEN	HSI, thermal extraction, HR-GCMS
rPET40 assp bb	T2313385	100% recycled PET after solid state polymerization bad batch (40 -> class that is no longer processable	SIOEN	HSI

		back into high tenacity PET fibers)		

Figure 2: Exemplary image of selected samples.

3.2. Methods

3.2.1. Controlled aging and chemical analysis

3.2.1.1. Controlled aging

In order to have samples for controlled aging, virgin materials (PET 5530, PET 5140 nIV and PET 5140 IV) were injection moulded at CTB into ISO B test specimens (rectangular shaped, 80x10x4 mm^3) using an Arburg Arburg Allrounder 320 S (50kN clamping force). Processing settings were kept constant between samples and are summarized below:

- Temperature profile: 287 - 275 - 270 - 260 – 250 °C (nozzle – inlet)
- Injection speed: 60 cm^3/s
- Holding pressure: 1300 bar
- Cooling time: 15 s
- Mould temperature: 50 °C

These injection moulded samples were controlled aged at CTB by using QUV aging according to ISO 4892-3 standard. 340 nm UV waves were used, at a temperature of 70°C, for a maximum of 800 hours. During the aging, the samples are subjected to water spray. Aged samples were collected after 50, 100, and 200 hours. At the time of writing, samples for 400 and 800 hours were just out of the equipment, and the results for 400 and 800 hours will be added to the next version of this deliverable.

3.2.1.2. Characterization of the controlled aged samples

Impact testing was conducted at CTB on the controlled aged samples using Zwick 5113 impact tester according to ISO 179-1. A 5J hammer was used, in a controlled testing environment on conditioned samples in a temperature of 23°C and relative humidity of 50±5%.

Tensile testing was done at CTB using a 20kN Zwick RetroLine dynamometer. Distance between the grips was set at 30 mm, using pneumatic clamps with clamping pressure of 2 bar on conditioned samples in a temperature of 23°C and relative humidity of 50±5%. A pre-load was set at 0.1 MPa, before determining the elastic modulus at 1 mm/min. After determination of the initial stress-strain behaviour, the test speed was increased to 50 mm/min until rupture. Three specimens were tested per sample.

The thermal properties were analyzed at CTB by Differential Scanning Calorimetry (DSC) and Thermogravimetric analysis (TGA). Regarding DSC, a TA instruments Discovery DSC2500 was used, according to ISO 11357-1. Approx. 5 mg of sample was weighted and analyzed using nitrogen flow. A heat-cool-heat programme was used between -50°C and 300°C, with heating/cooling flow of 10 °C/min. Regarding TGA, a TA instruments Discovery TGA5500 was used, according to ISO1158-1. Approx. 5 mg of samples was weighted and analyzed using nitrogen flow. Temperature was increased at 10 °C/min to 600 °C in nitrogen atmosphere, before increasing to 900 °C at 10°C/min in air atmosphere.

Rheological analysis was performed at CTB using an Anton paar MCR302 instrument. Samples were dried prior to analysis. A parallel plate (diameter 25 mm, die gap 1 mm) setup was used, with a shear rate between 0.1 and 3000 s-1 at a testing temperature of 285°C.

3.2.1.3. Chemical analysis

Selected samples (Table 2) were analyzed at CTB by thermal extraction to extract volatile components. One or more 1 cm diameter samples were heated in a glass tube at a defined temperature (120°C) and under an inert gas stream for 30 mins. The gas stream was then passed over a Tenax-filled tube where the volatile organic components are retained. The Tenax tube containing volatile organic components (VOC) was then thermally desorbed. The VOC are cryogenically trapped and injected into a GCMS to Agilent MSD detector.

Secondly, high-resolution GCMS was used. For this, the samples (Table 2) are extracted using ultrasonic solvent extraction using three different solvents (mixtures) being a methanol/acetone mixture, toluene and THF. For this a simple ultrasonic bath was used. The extraction was done with 1 gram sample for 60 min, with a temperature of 60 °C. The extracts were analyzed using a Thermo Scientific Trace 1300 gas chromatograph coupled with a Thermo Scientific Orbitrap Exploris GC mass spectrometer using electron ionization (EI). Orbitrap is an ion trap mass spectrometer making it possible to separate and detect ions with a mass accuracy of 5 digits after the decimal point.

3.2.2. Hyperspectral imaging

CIRCE is equipped with hyperspectral analysis tools that have been used so far for sample analysis (see section 3.1). The Specim FX17 hyperspectral equipment has been utilized to capture data from the samples (the characteristics of the equipment can be seen in Table 3). The samples have been scanned using the Specim LabScanner laboratory scanner (see Table 4) and processed with the dedicated SpecimCUBE computer (see Table 5). The data are analyzed using the SpecimInsight processing software. All this equipment can be seen in Figure 3.

During this task, the work carried out corresponds to an initial contact before defining the specific characteristics and requirements of the task. The samples processed in the first phase correspond to those detailed in section 3.1. These samples have been gathered at different stages of the process for virgin and recycled plastic. No specific criteria were established for sample selection; instead, available samples were chosen for an initial analysis.

During this period, it has been studied whether, in the NIR, with the FX17 camera, the samples can be distinguished. The methodology followed has been as follows: 1) the samples are placed in small boxes on the LabScanner tray; 2) the LabScanner is activated to move the samples and the FX17 hyperspectral camera captures data from the samples; 3) the data are loaded into SpecimInsight, where they are analyzed, processed, and ML models are trained to differentiate the samples; 4) the trained ML models are loaded into SpecimCUBE, allowing them to be executed in real-time on the samples to test their performance. The most critical part of this methodology and where most efforts are required, is the data analysis and ML model training.

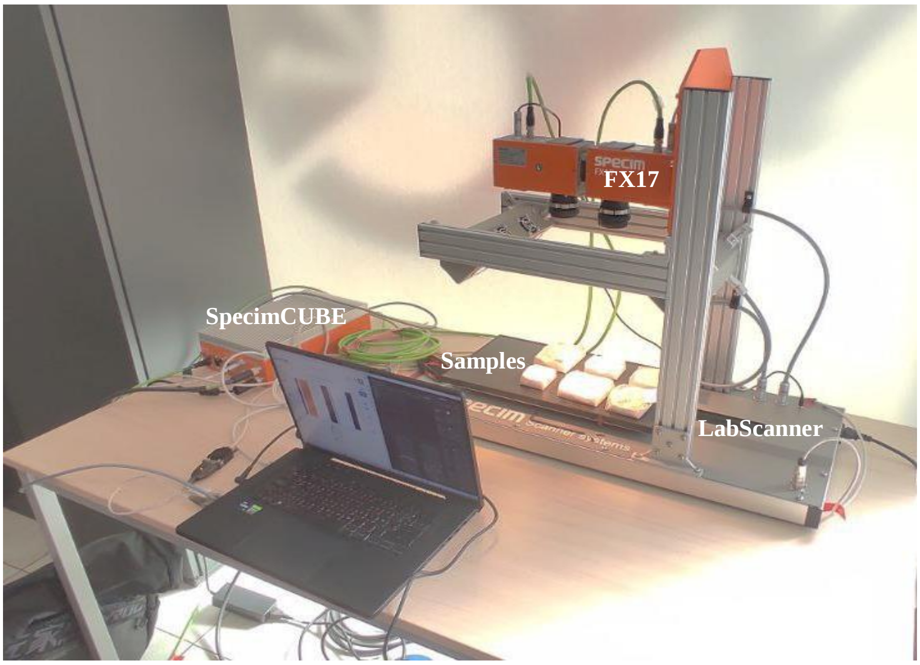


Figure 3: Hyperspectral equipment (Camera FX17, Scanner LabScanner and Dedicated PC SpecimCUBE).

Table 3: FX17 features.

Features	FX17
Type	Line-scan
Spectral range	900-1700 nm (NIR)
Spectral resolution	8 nm

Spectral sampling/pixel	3.5 nm
Spectral bands	224
Spatial samples	640
Bit depth	12
Maximum frame rate	527 FPS full range
FOV	38º
Camera digital data output/control interface	Gige VIsion
Power input	12 V DC
Power consumption	Max 24 W
IP	IP 52
Dimensions (LxWxH)	150x75x85 mm
Weight	1.56 kg
Operating temperature	+5 … +40ºC

Table 4: LabScanner features.

Features	LabScanner
Size (LxWxH)	699x335x710 mm
Sample tray dimensions	400x200 mm
Weight	13 kg
Scanning speed	0.1 – 99 mm/s
Max sample weight	5 kg
Illumination	DECOSTAR 51 ALU 20W 12V 36deg GU5.3 halogen
Input voltage	Nominal 100…240 VAC
Output voltage	12V DC
Output current	26.7 A
Max power consumption	Single illumination : 110W Dual illumination : 170W
Cameras supported	FX10, FX17, SWIR

Table 5: Dedicated PC SpecimCUBE features.

Features	SpecimCUBE
Size (LxWxH)	300x190x91 mm
Weight	4.3 kg
SoC	NVIDIA Jetson Xavier AGX
Storage	1 TB
Camera interface	GigE Vision

Input power	10-32 V DC

4. Results: controlled aging and chemical analysis

4.1. Controlled aging

4.1.1. QUV aging

Figure 4 shows images of 50, 100, 200, 400 and 800 hours QUV aged samples of (left) PET 5530, (middle) PET 5140 nIV and (right) PET 5140 IV. At the time of writing, samples 400 and 800 hours were just out of the equipment, and the results for 400 and 800 hours will be added to the next version of this deliverable.

Overall for all materials, a slight discoloration can be noticed upon progressive aging. The samples however remain physically intact.

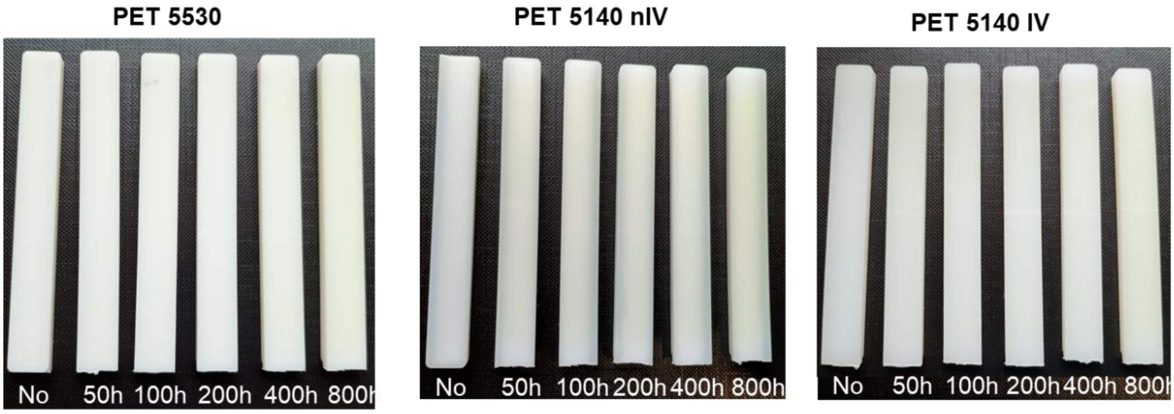


Figure 4: Images of 50, 100, 200, 400 and 800 hours QUV aged samples of (left) PET 5530, (middle) PET 5140 nIV and (right) PET 5140 IV.

4.1.2. Thermal extraction

Figure 5 shows an example of a GCMS chromatogram obtained after thermal extraction. The retention time (x-axis) is typically analyzed up to 60.00 seconds and is useful to determine both the presence of VOCs (<30 s) but also semi VOCs (>30 s). Each peak represents a chemical compound/component, and depending on the volatility semi-quantitative analysis is possible.

Several virgin and recycled samples were analyzed (Table 2). Table 6 gives an overview of all measured samples, and the most prominent chemical species identified. For all samples, only relevant chemical specie identified was benzyl benzoate. This compound however is regularly used as additive in textile processing and as such cannot be used as marker for recycled content. Also, the semi-quantitative results do not show meaningful differences between recycled and virgin PET.

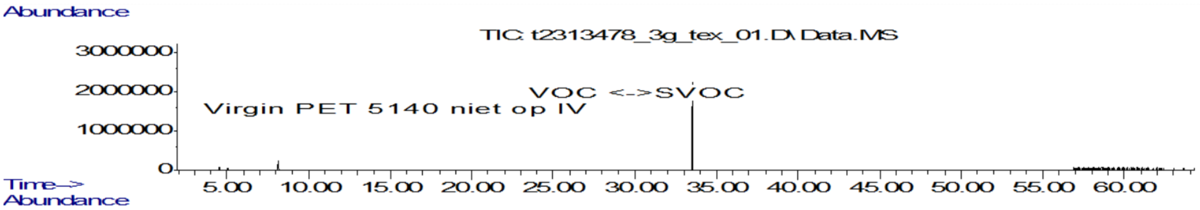
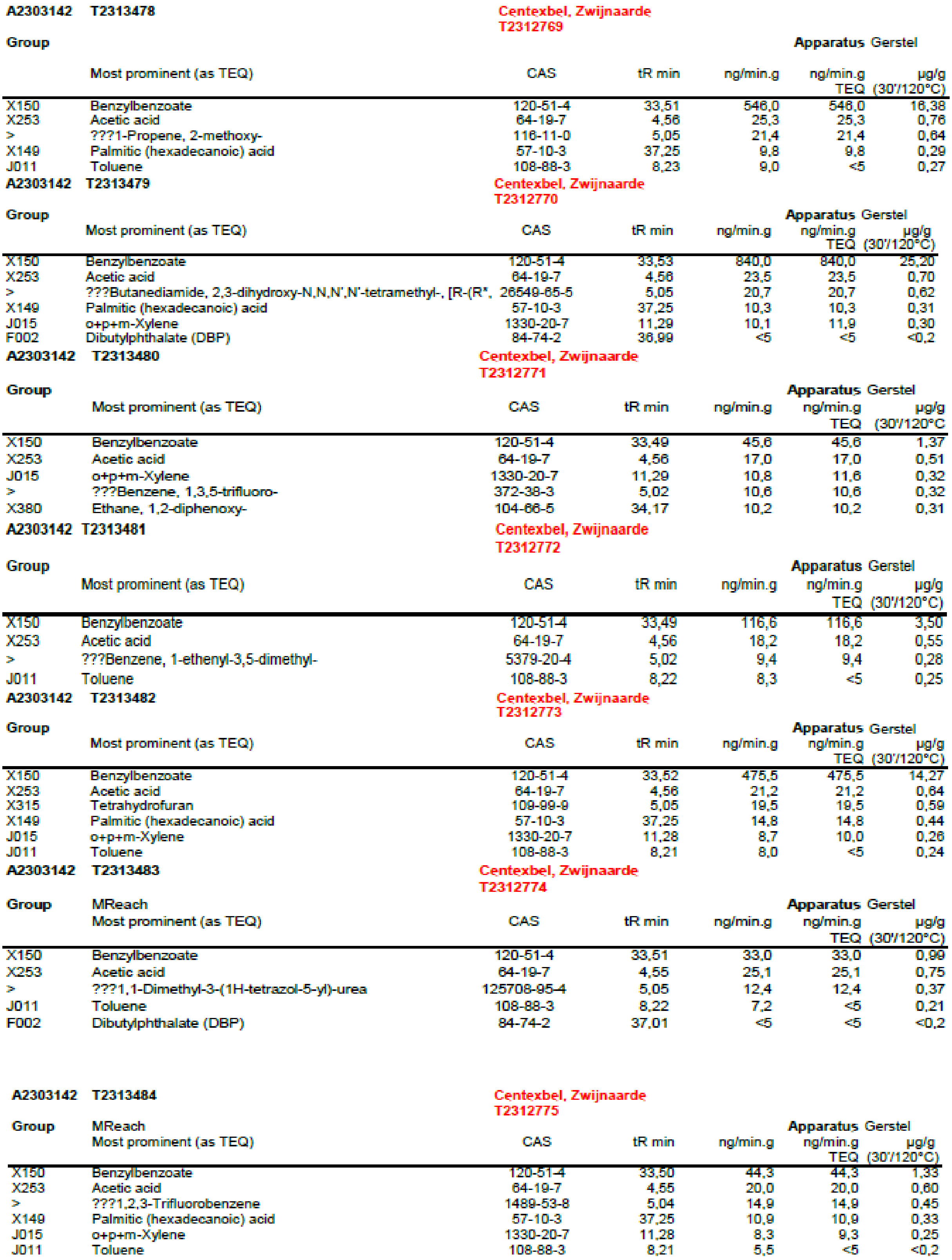


Figure 5: Example GCMS chromatogram for virgin PET obtained after thermal extraction.

Table 6: Identified volatiles using thermal extraction.



A2303142 T2313478 Centexbel, Zwijnaarde T2312769

Apparatus Gerstel

Group	Most prominent (as TEQ)	CAS	tR min	ng/min.g	ng/min.g TEQ	µg/g (30'/120°C)
X150	Benzylbenzoate	120-51-4	33,51	546,0	546,0	16,38
X253	Acetic acid	64-19-7	4,56	25,3	25,3	0,76
>	???1-Propene, 2-methoxy-	116-11-0	5,05	21,4	21,4	0,64
X149	Palmitic (hexadecanoic) acid	57-10-3	37,25	9,8	9,8	0,29
J011	Toluene	108-88-3	8,23	9,0	<5	0,27

A2303142 T2313479 Centexbel, Zwijnaarde T2312770

Apparatus Gerstel

Group	Most prominent (as TEQ)	CAS	tR min	ng/min.g	ng/min.g TEQ	µg/g (30'/120°C)
X150	Benzylbenzoate	120-51-4	33,53	840,0	840,0	25,20
X253	Acetic acid	64-19-7	4,56	23,5	23,5	0,70
>	???Butanediamide, 2,3-dihydroxy-N,N,N',N'-tetramethyl-, [R-(R*,	26549-65-5	5,05	20,7	20,7	0,62
X149	Palmitic (hexadecanoic) acid	57-10-3	37,25	10,3	10,3	0,31
J015	o+p+m-Xylene	1330-20-7	11,29	10,1	11,9	0,30
F002	Dibutylphthalate (DBP)	84-74-2	36,99	<5	<5	<0,2

A2303142 T2313480 Centexbel, Zwijnaarde T2312771

Apparatus Gerstel

Group	Most prominent (as TEQ)	CAS	tR min	ng/min.g	ng/min.g TEQ	µg/g (30'/120°C
X150	Benzylbenzoate	120-51-4	33,49	45,6	45,6	1,37
X253	Acetic acid	64-19-7	4,56	17,0	17,0	0,51
J015	o+p+m-Xylene	1330-20-7	11,29	10,8	11,6	0,32
>	???Benzene, 1,3,5-trifluoro-	372-38-3	5,02	10,6	10,6	0,32
X380	Ethane, 1,2-diphenoxy-	104-66-5	34,17	10,2	10,2	0,31

A2303142 T2313481 Centexbel, Zwijnaarde T2312772

Apparatus Gerstel

Group	Most prominent (as TEQ)	CAS	tR min	ng/min.g	ng/min.g TEQ	µg/g (30'/120°C)
X150	Benzylbenzoate	120-51-4	33,49	116,6	116,6	3,50
X253	Acetic acid	64-19-7	4,56	18,2	18,2	0,55
>	???Benzene, 1-ethenyl-3,5-dimethyl-	5379-20-4	5,02	9,4	9,4	0,28
J011	Toluene	108-88-3	8,22	8,3	<5	0,25

A2303142 T2313482 Centexbel, Zwijnaarde T2312773

Apparatus Gerstel

Group	Most prominent (as TEQ)	CAS	tR min	ng/min.g	ng/min.g TEQ	µg/g (30'/120°C)
X150	Benzylbenzoate	120-51-4	33,52	475,5	475,5	14,27
X253	Acetic acid	64-19-7	4,56	21,2	21,2	0,64
X315	Tetrahydrofuran	109-99-9	5,05	19,5	19,5	0,59
X149	Palmitic (hexadecanoic) acid	57-10-3	37,25	14,8	14,8	0,44
J015	o+p+m-Xylene	1330-20-7	11,28	8,7	10,0	0,26
J011	Toluene	108-88-3	8,21	8,0	<5	0,24

A2303142 T2313483 Centexbel, Zwijnaarde T2312774

Apparatus Gerstel

Group	MReach Most prominent (as TEQ)	CAS	tR min	ng/min.g	ng/min.g TEQ	µg/g (30'/120°C)
X150	Benzylbenzoate	120-51-4	33,51	33,0	33,0	0,99
X253	Acetic acid	64-19-7	4,55	25,1	25,1	0,75
>	???1,1-Dimethyl-3-(1H-tetrazol-5-yl)-urea	125708-95-4	5,05	12,4	12,4	0,37
J011	Toluene	108-88-3	8,22	7,2	<5	0,21
F002	Dibutylphthalate (DBP)	84-74-2	37,01	<5	<5	<0,2

A2303142 T2313484 Centexbel, Zwijnaarde T2312775

Apparatus Gerstel

Group	MReach Most prominent (as TEQ)	CAS	tR min	ng/min.g	ng/min.g TEQ	µg/g (30'/120°C)
X150	Benzylbenzoate	120-51-4	33,50	44,3	44,3	1,33
X253	Acetic acid	64-19-7	4,55	20,0	20,0	0,60
>	???1,2,3-Trifluorobenzene	1489-53-8	5,04	14,9	14,9	0,45
X149	Palmitic (hexadecanoic) acid	57-10-3	37,25	10,9	10,9	0,33
J015	o+p+m-Xylene	1330-20-7	11,28	8,3	9,3	0,25
J011	Toluene	108-88-3	8,21	5,5	<5	<0,2

4.1.3. HR-GCMS

From these chromatograms, not much difference can be seen. Besides the big difference in extraction efficiency using different solvents, where acetone/MeOH seems to give the most signals. From this we can see no consistent appearance or disappearing of signals (marker substances) that gives us the ability of telling virgin PET and recycled PET apart using this method.

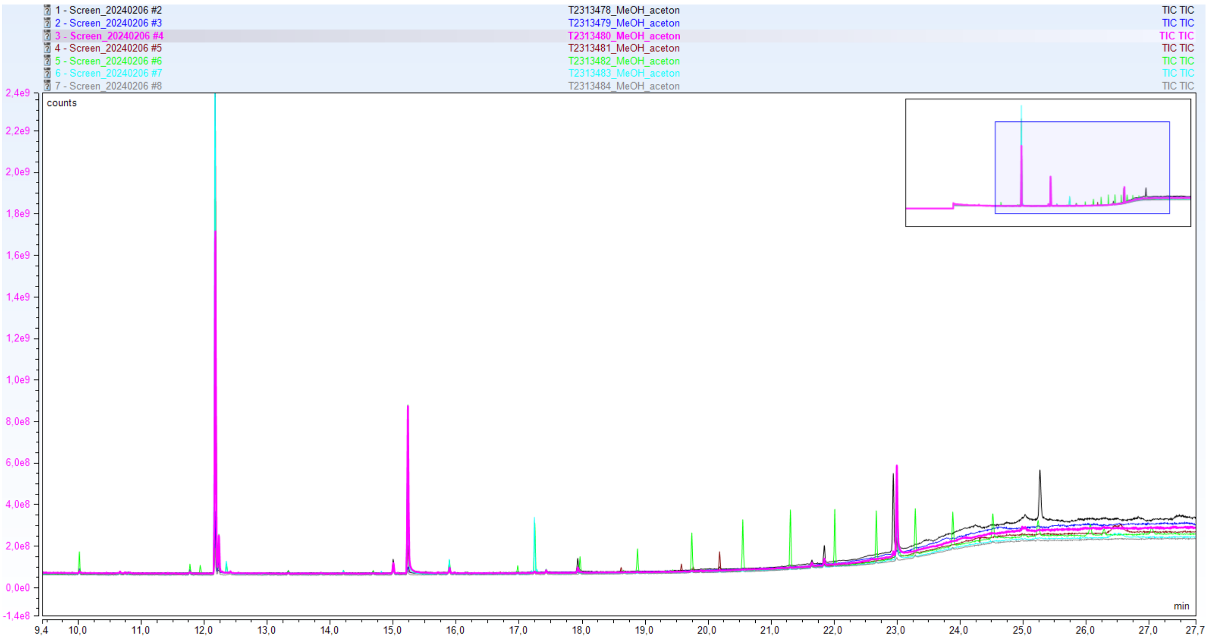


Figure 6: Chromatogram overlay of extracts using methanol/acetone. Note: The green signals on T2313482 are the result from siloxanes coming from the column.

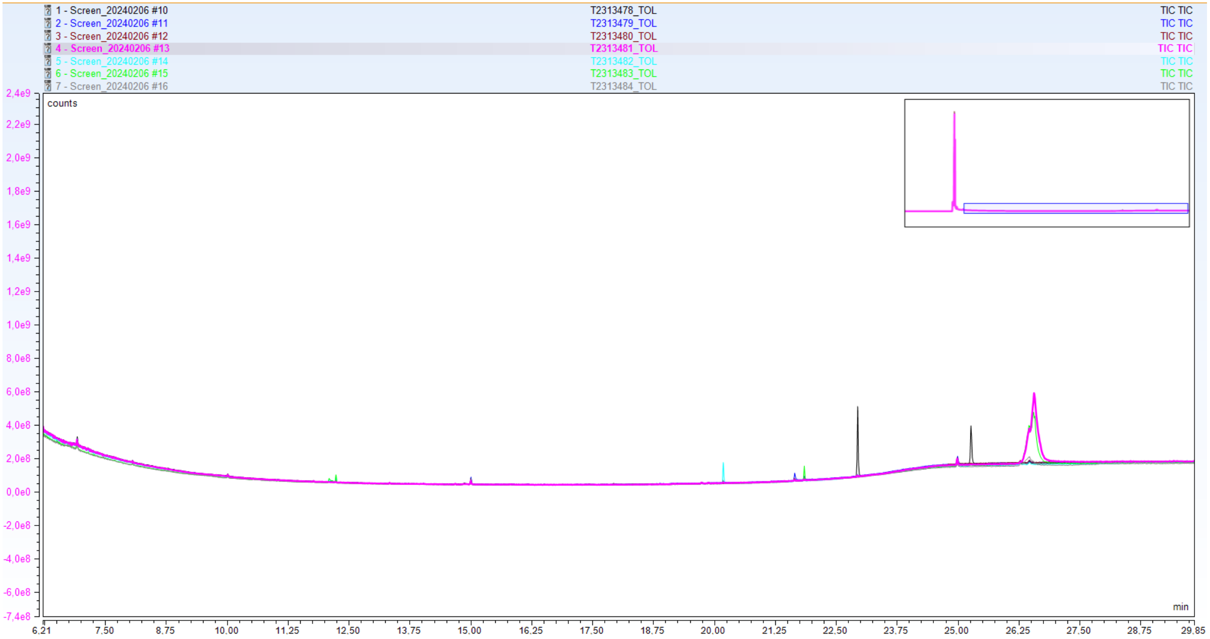


Figure 7: Chromatogram overlay of extracts using toluene.

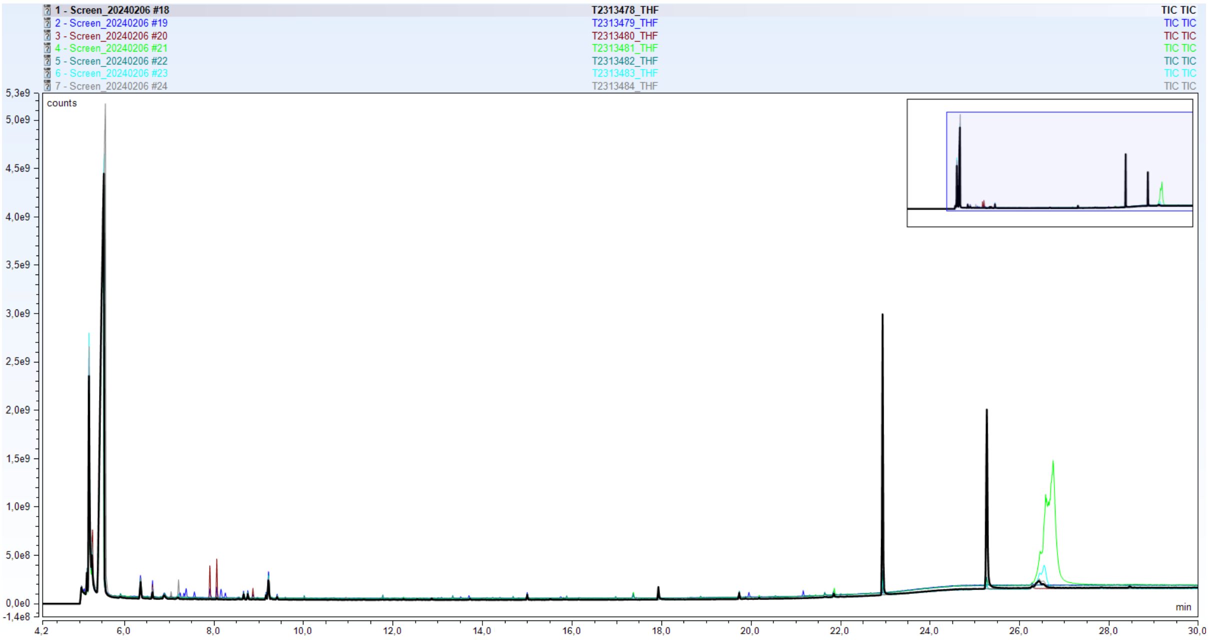
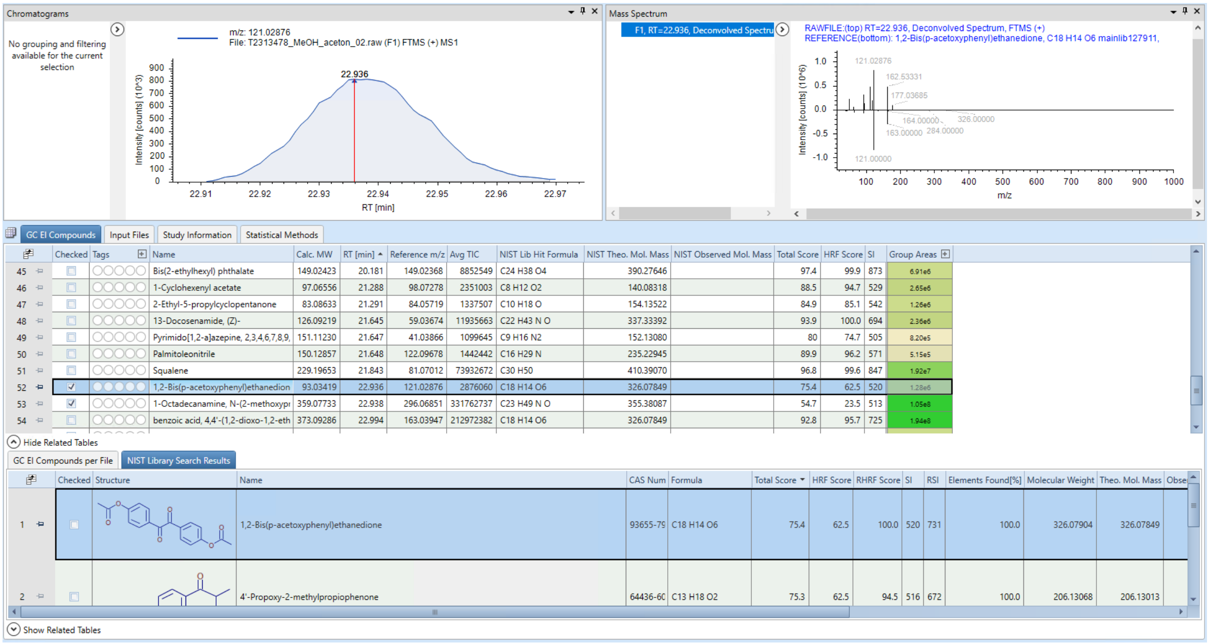


Figure 8: Chromatogram overlay of extracts using THF.

The data was also analyzed using Compound Discoverer^{TM} software, which is able to separate and identify overlapping component signals. But this also didn't give us more insight.



	Checked	Tags	Name	Calc. MW	RT [min]	Reference m/z	Avg TIC	NIST Lib Hit Formula	NIST Theo. Mol. Mass	NIST Observed Mol. Mass	Total Score	HRF Score	SI	Group Areas
45	☐	○○○○○	Bis(2-ethylhexyl) phthalate	149.02423	20.181	149.02368	8852549	C24 H38 O4	390.27646		97.4	99.9	873	6.91e6
46	☐	○○○○○	1-Cyclohexenyl acetate	97.06556	21.288	98.07278	2351003	C8 H12 O2	140.08318		88.5	94.7	529	2.65e6
47	☐	○○○○○	2-Ethyl-5-propylcyclopentanone	83.08633	21.291	84.05719	1337507	C10 H18 O	154.13522		84.9	85.1	542	1.26e6
48	☐	○○○○○	13-Docosenamide, (Z)-	126.09219	21.645	59.03674	11935663	C22 H43 N O	337.33392		93.9	100.0	694	2.36e6
49	☐	○○○○○	Pyrimido[1,2-a]azepine, 2,3,4,6,7,8,9,	151.11230	21.647	41.03866	1099645	C9 H16 N2	152.13080		80	74.7	505	8.20e5
50	☐	○○○○○	Palmitoleonitrile	150.12857	21.648	122.09678	1442442	C16 H29 N	235.22945		89.9	96.2	571	5.15e5
51	☐	○○○○○	Squalene	229.19653	21.843	81.07012	73932672	C30 H50	410.39070		96.8	99.6	847	1.92e7
52	☑	○○○○○	1,2-Bis(p-acetoxyphenyl)ethanedion	93.03419	22.936	121.02876	2876060	C18 H14 O6	326.07849		75.4	62.5	520	1.28e6
53	☑	○○○○○	1-Octadecanamine, N-(2-methoxypı	359.07733	22.938	296.06851	331762737	C23 H49 N O	355.38087		54.7	23.5	513	1.05e8
54	☐	○○○○○	benzoic acid, 4,4'-(1,2-dioxo-1,2-eth	373.09286	22.994	163.03947	212972382	C18 H14 O6	326.07849		92.8	95.7	725	1.94e8

	Checked	Structure	Name	CAS Num	Formula	Total Score	HRF Score	RHRF Score	SI	RSI	Elements Found[%]	Molecular Weight	Theo. Mol. Mass	Obse
1	☐		1,2-Bis(p-acetoxyphenyl)ethanedione	93655-79	C18 H14 O6	75.4	62.5	100.0	520	731	100.0	326.07904	326.07849	
2	☐		4'-Propoxy-2-methylpropiophenone	64436-60	C13 H18 O2	75.3	62.5	94.5	516	672	100.0	206.13068	206.13013	

Figure 9: Data analysis by Compound Discoverer^{TM} software.

In conclusion, the chromatograms do not show the appearance or disappearance of signals between the virgin material and the recycled materials, nor do they show a consistent decrease or increase of certain signals. Suggesting this method is not usable to detect possible NIAS in the RPET, rendering it not possible to distinguish between this virgin and recycled PET material.

4.2. Rheological properties

Figure 10 shows the viscosity of the polymer expressed in Pa.s plotted against shear rate (1/s). The initial plateau of all materials is related to the Newtonian behaviour, followed by a strong shear thinning relation in which the viscosity drops with increasing shear rate. Most importantly here is the height of the Newtonian viscosity (initial plateau) compared between the samples. This is an indication for the fluidity of the polymer melt and is related to molecular weight and melt interactions.

Firstly, it can be noticed that the viscosity of the PET 5140 IV is higher compared to the other two materials. This is related to the solid-state polycondensation process done on the samples which increases the molecular weight and hence the viscosity as well. This is measurable via intrinsic viscosity (IV) or via these melt plate-plate measurements.

Figure 10: Rheological properties (viscosity as function of shear rate) of PET 5530, PET 5140 nIV and PET 5140 IV for non-aged, 50, 100 and 200 hours QUV aged samples.

The effect of longer aging times is less obvious. It is well known that UV aging of PET either leads to crosslinking or chain scission[8], depending on the UV radiation, degradation kinetics and matrix of the PET, and hence changing the molecular weight of the polyester. As the viscosity relates a.o. to the molecular weight it is very difficult to assess or predict the outcome after QUV aging.

4.3. Mechanical properties: tensile and impact

Figure 11 shows the results of the tensile properties stiffness E_t, strength σ_m and elongation at break ε_b of PET 5530, PET 5140 nIV and PET 5140 IV after UV aging (0, 50, 100 and 200 hours). For all materials, the stiffness remains constant up to 200 hours of QUV aging. Regarding maximum strength,

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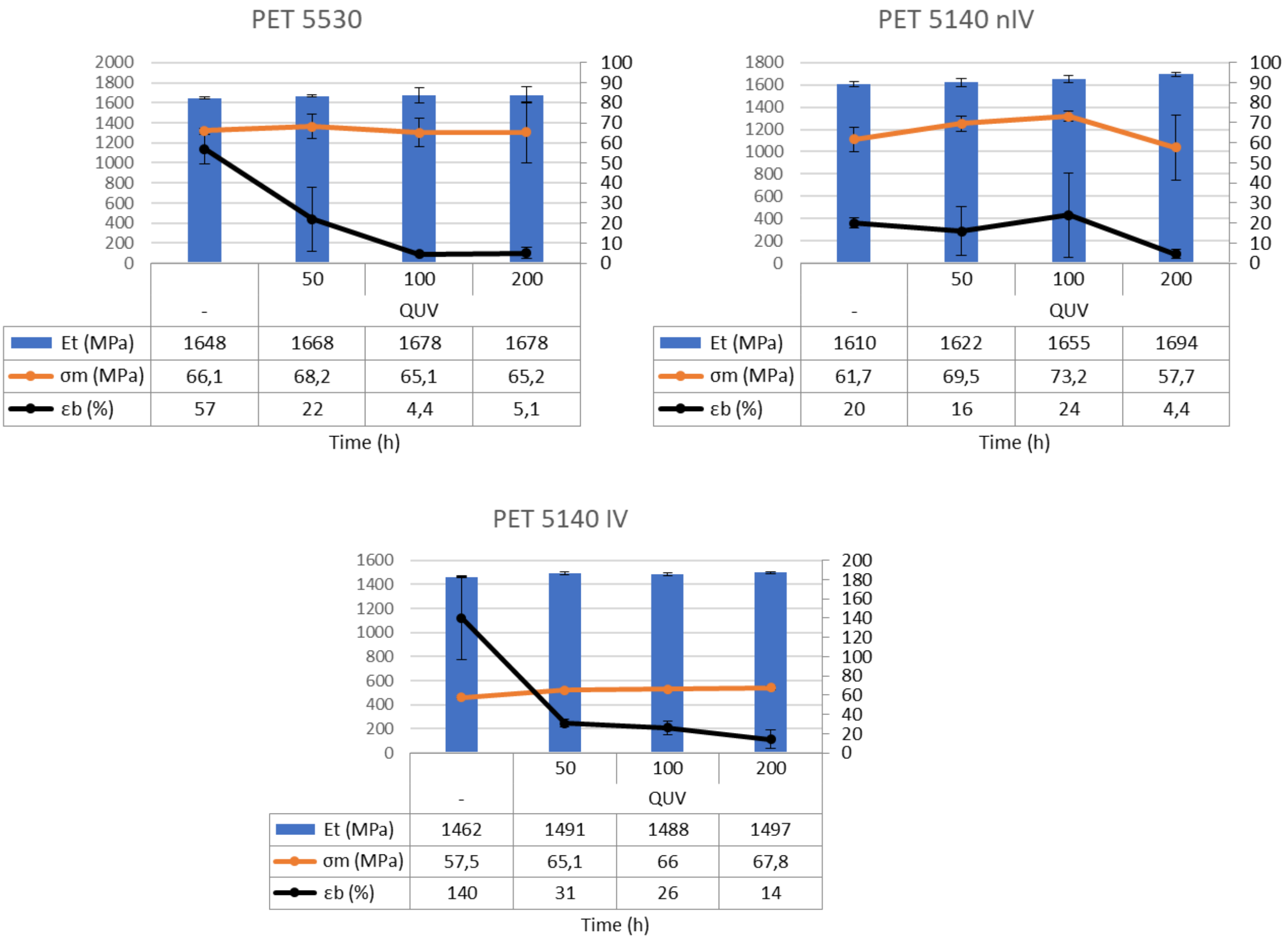
[8] Andersen, Emil, et al. "Real-time ageing of polyesters with varying diols." Materials Chemistry and Physics 261 (2021): 124240.

some more variation is noticeable, with a slight tendency of an increase for the PET 5140 IV material. The strain at break is strongly affected negatively for all samples. Depending on the initial ductility, this reduction is more (PET 5530 and PET 5140 IV) or less (PET 5140 nIV) pronounced.



PET 5530

	-	50	100	200
		QUV		
Et (MPa)	1648	1668	1678	1678
σm (MPa)	66,1	68,2	65,1	65,2
εb (%)	57	22	4,4	5,1

PET 5140 nIV

	-	50	100	200
		QUV		
Et (MPa)	1610	1622	1655	1694
σm (MPa)	61,7	69,5	73,2	57,7
εb (%)	20	16	24	4,4

PET 5140 IV

	-	50	100	200
		QUV		
Et (MPa)	1462	1491	1488	1497
σm (MPa)	57,5	65,1	66	67,8
εb (%)	140	31	26	14

Figure 11: Tensile properties (stiffness E_t, strength σ_m and elongation at break ε_b) of PET 5530, PET 5140 nIV and PET 5140 IV for non-aged, 50, 100 and 200 hours QUV aged samples.

Figure 12 shows the impact properties of PET 5530, PET 5140 nIV and PET 5140 IV for non-aged, 50, 100 and 200 hours QUV aged samples. For all samples, a severe reduction in impact resistance is noticed. The samples become more brittle because of degradation during aging.

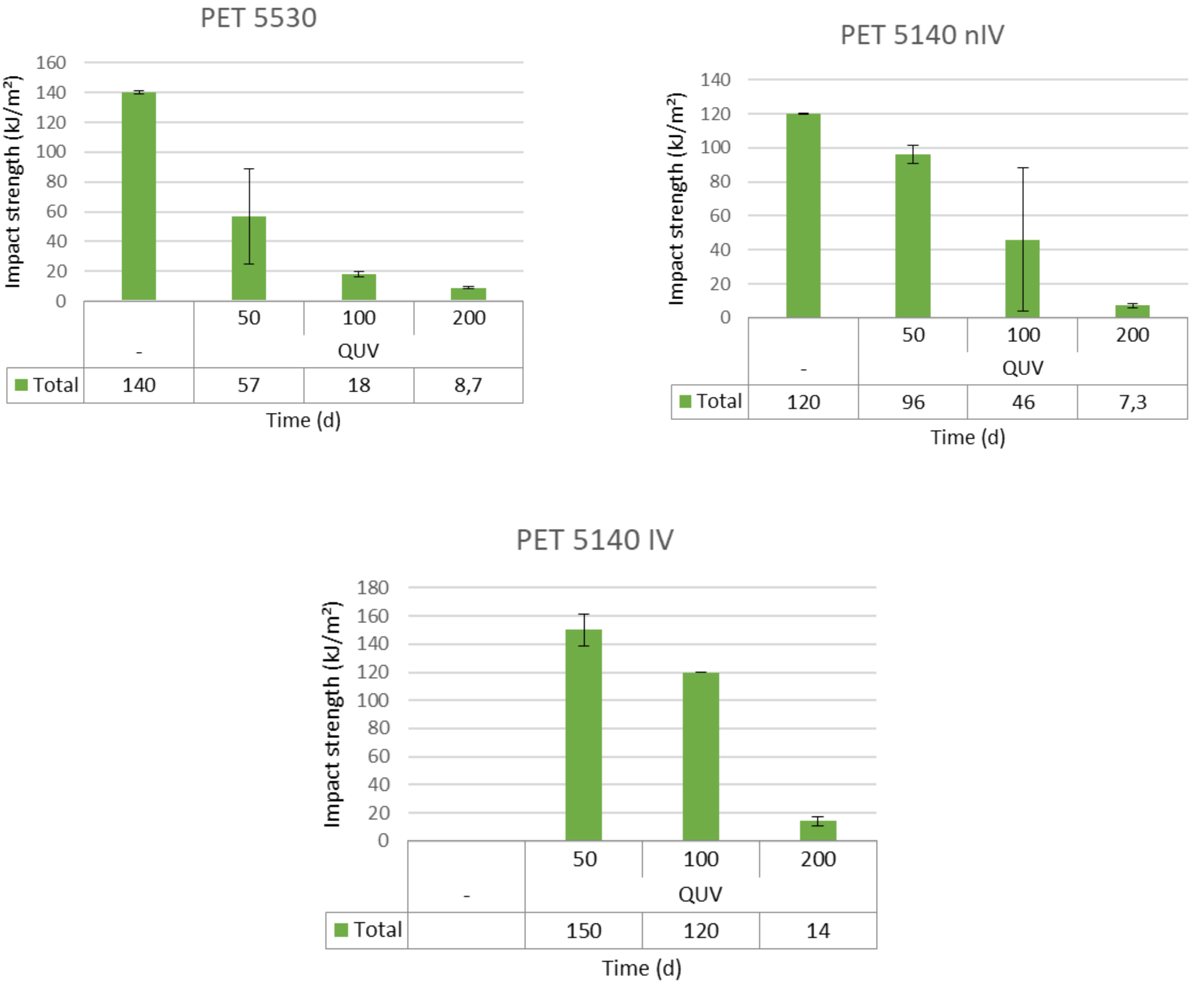


Figure 12: Impact properties of PET 5530, PET 5140 nIV and PET 5140 IV for non-aged, 50, 100 and 200 hours QUV aged samples. The initial (virgin) impact value for PET 5140 IV is not measurable (>200 kJ/m²).

4.4. Thermal properties: DSC and TGA

Table 7 gives an overview of the thermal properties derived from DSC and TGA measured on all non-aged and aged materials.

Table 7: Thermal properties derived from DSC and TGA

Sample	QUV aging (hours)	DSC						TGA
		Tg (°C)	Tc (°C)	Tm (°C)	ΔHm (J/g)	ΔHm° (J/g)	Xc (%)	Td (°C)
PET 5530	0	83	194	254	44,75	140	32	377
PET 5530	50	83	194	254	45,66	140	33	382
PET 5530	100	82	197	254	48,79	140	35	381
PET 5530	200	83	196	254	47,75	140	34	376
PET 5140 nIV	0	83	194	254	46,89	140	33	368
PET 5140 nIV	50	83	194	254	47,07	140	34	376
PET 5140 nIV	100	82	194	254	49,58	140	35	371
PET 5140 nIV	200	83	197	254	49,8	140	36	381

PET 5140 IV	0	84	184	252	40,75	140	29	373
PET 5140 IV	50	84	184	252	40,98	140	29	382
PET 5140 IV	100	84	186	252	41,94	140	30	382
PET 5140 IV	200	84	187	253	42,09	140	30	382

For all samples, glass transition temperature (Tg) remains constant, however a slight increase in crystallization (Tc) temperature can be noticed. This is related to faster crystallization (shorter chain length due to aging), and this is also noticed in an increased crystallinity (Xc). Regarding thermal degradation temperature (Td), only small changes can be noticed, which are not conclusive.

5. Results: Hyperspectral imaging

In Figure 13, the spectral fingerprints of each analyzed sample are shown. They are practically identical since the material is the same, indicating that differentiation is not easy. The goal of this analysis is to distinguish the fingerprints from each other, and to achieve this, it is necessary to look for the characteristic and unique peaks (maximum and minimum) and slopes of the spectral fingerprint of each sample that allow it to be differentiated from the rest.

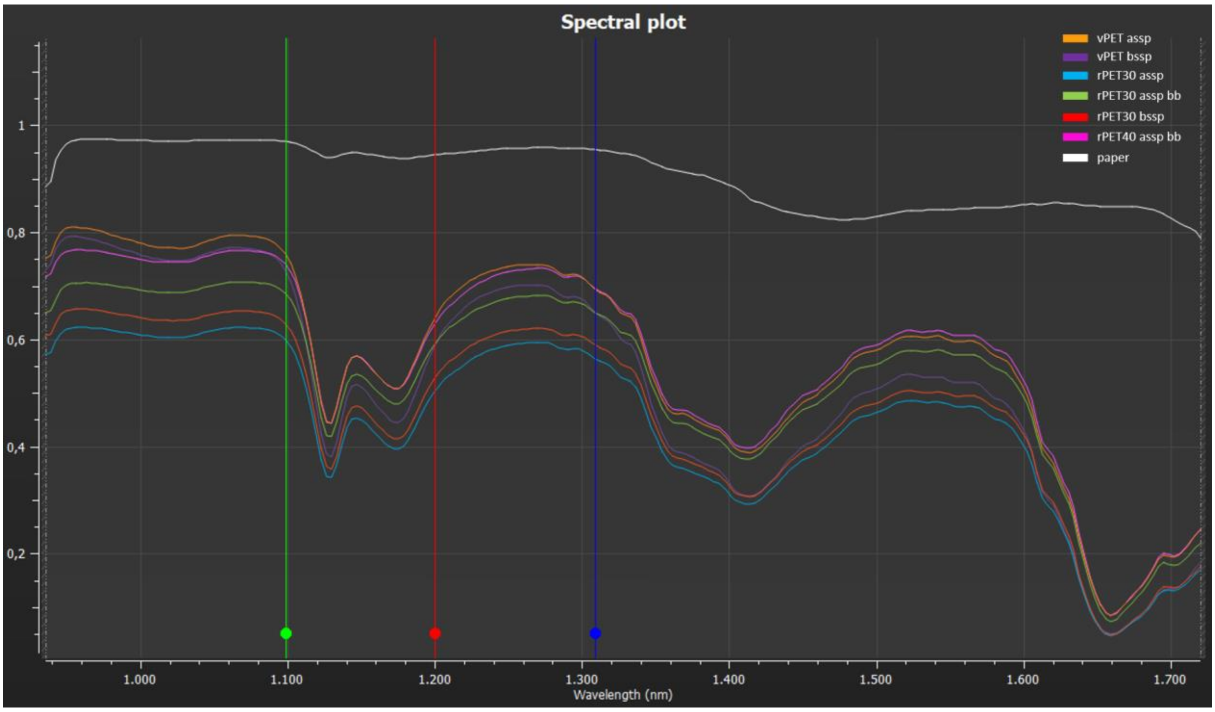
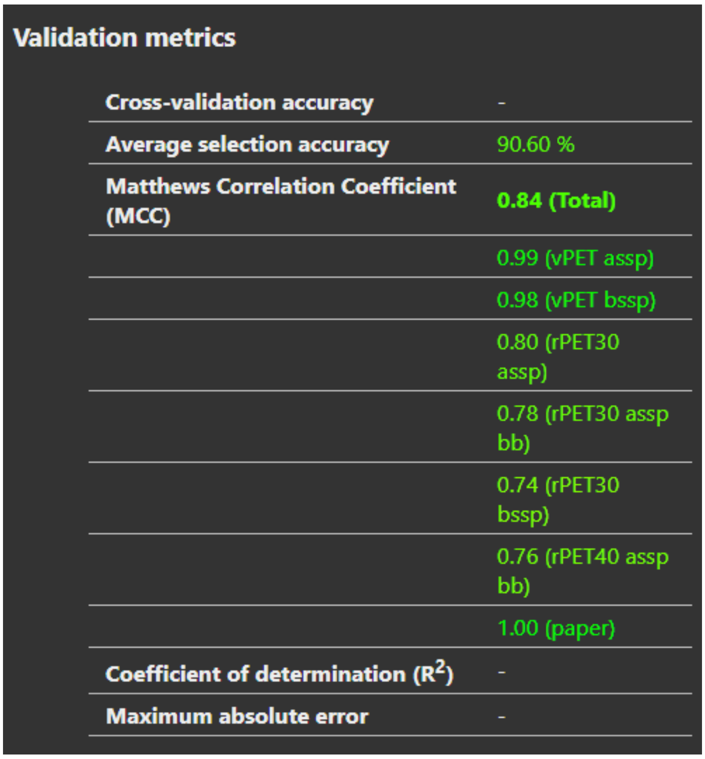


Figure 13: Spectral fingerprints of vPET assp, vPET bssp, rPET30 assp, rPET30 assp bb, rPET30 bssp, and rPET40 assp bb.

The y-axis, representing the sample's reflection intensity, cannot be considered in absolute value because it is very sensitive to the amount of light the sample receives. If the environment is not highly controlled with constant light, it will induce error. Instead, its relative value will be used to find these differences. To highlight the differences between spectral fingerprints and minimize noise, preprocessing functions such as Mean Centering, Standard Normal Variable, Savitzky-Golay filter, and Derivate have been used. An appropriate combination of these functions improves the classification results. The model used to differentiate the samples is a Partial Least Squares Discriminant Analysis (PLS-DA) algorithm. This algorithm is suitable for the use case due to its ability to handle multicollinear and high-dimensional data, its discrimination capability, and its robustness against noise and redundancy.

Figure 14 presents the data obtained from training the PLS-DA algorithm with virgin plastic (vPET) and recycled plastic (rPET) samples at different stages of the production process (see section 3.1). The goal is to create a model that can distinguish between the different types of samples and determine if hyperspectral technology in the NIR is suitable.

For the study, the following metrics have been used: 1) the Matthews Correlation Coefficient (MCC) (see Equation 1), 2) precision (see Equation 2), and 3) recall (see Equation 2). These metrics are calculated by combining the results of True Positive (TP), True Negative (TN), False Positive (FP), and False Negative (FN) obtained by comparing the model's predictions with the ground truth or sample label (see Table 8). Together, these metrics provide a robust evaluation of the algorithm in terms of balance, specificity, and sensitivity, which are critical aspects in the classification and analysis of spectral fingerprints.



Validation metrics

Cross-validation accuracy	-
Average selection accuracy	90.60 %
Matthews Correlation Coefficient (MCC)	**0.84 (Total)**
	0.99 (vPET assp)
	0.98 (vPET bssp)
	0.80 (rPET30 assp)
	0.78 (rPET30 assp bb)
	0.74 (rPET30 bssp)
	0.76 (rPET40 assp bb)
	1.00 (paper)
Coefficient of determination (R^2)	-
Maximum absolute error	-

Figure 14: Results from PLS-DA algorithm training.

Table 8: TP, TN, FP, FN confusion matrix

		Actual	
		Positive	**Negative**
Predicted	**Positive**	True positive	False positive
	Negative	False Negative	True negative

Equation 1: Matthews Correlation Coefficient

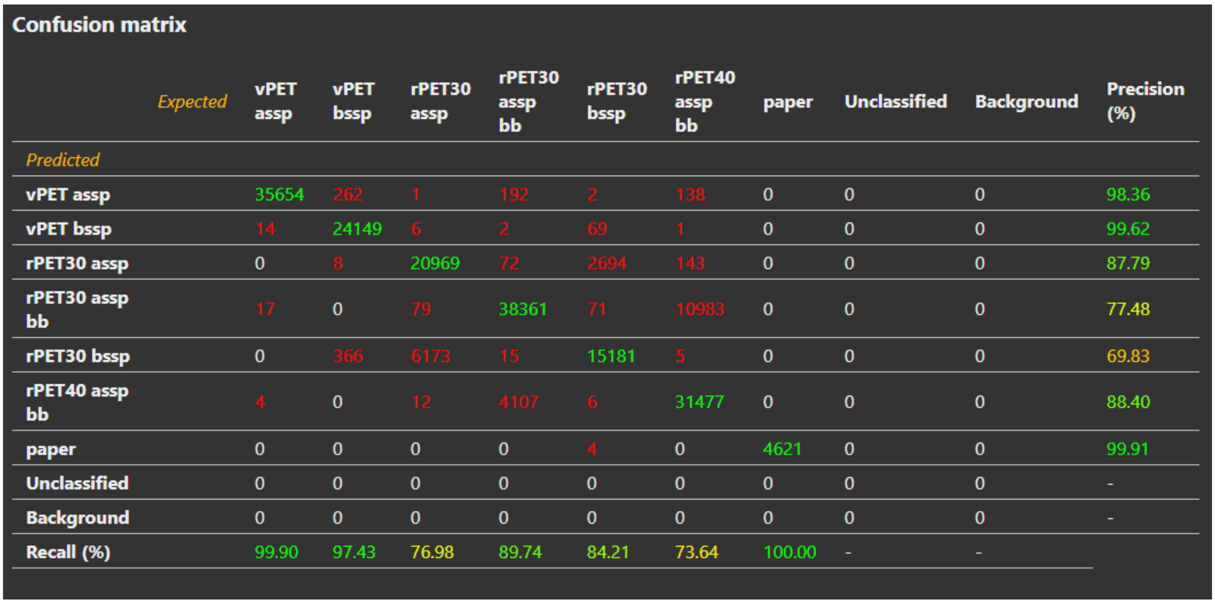
$$MCC = \frac{TP * TN - FP * FN}{\sqrt{(TP + FP) * (TP + FN) * (TN + FP) * (TN + FN)}}$$

Equation 2: Precision and Recall

$$Precision = \frac{TP}{TP + FP} \qquad Recall = \frac{TP}{TP + FN}$$

According to the results shown in Figure 14, the model's average precision is 90.60%, and the MCC obtained is 0.84, suggesting a good capability of the model to discriminate between the different classes. The MCC is a more informative measure than precision because it considers all values of the confusion matrix (true positives, true negatives, false positives, and false negatives). Therefore, it is the reference metric to quantify the model's capability. Thus, the values obtained indicate a high discrimination capacity of the model for most classes, especially for virgin PET samples, which have MCC values very close to 1.

For a more detailed analysis, the confusion matrix, an essential tool for evaluating a classification model's performance, has been used as it provides detailed information about the model's correct and incorrect predictions. The confusion matrix of the PLS-DA algorithm is presented in Figure 15. To evaluate this matrix, the other two previously mentioned metrics, precision and recall (Equation 2), are analyzed. Precision is the proportion of instances correctly classified as positive (TP) among all instances classified as positive (TP + FP). It measures the accuracy of the model's positive predictions. Recall, also known as sensitivity or true positive rate, is the proportion of instances correctly classified as positive (TP) among all instances that are actually positive (TP + FN). It measures the model's ability to find all positive instances.



Confusion matrix

Expected	vPET assp	vPET bssp	rPET30 assp	rPET30 assp bb	rPET30 bssp	rPET40 assp bb	paper	Unclassified	Background	Precision (%)
Predicted										
vPET assp	35654	262	1	192	2	138	0	0	0	98.36
vPET bssp	14	24149	6	2	69	1	0	0	0	99.62
rPET30 assp	0	8	20969	72	2694	143	0	0	0	87.79
rPET30 assp bb	17	0	79	38361	71	10983	0	0	0	77.48
rPET30 bssp	0	366	6173	15	15181	5	0	0	0	69.83
rPET40 assp bb	4	0	12	4107	6	31477	0	0	0	88.40
paper	0	0	0	0	4	0	4621	0	0	99.91
Unclassified	0	0	0	0	0	0	0	0	0	-
Background	0	0	0	0	0	0	0	0	0	-
Recall (%)	99.90	97.43	76.98	89.74	84.21	73.64	100.00	-	-	

Figure 15: Confusion matrix assessing precision and recall results of each sample.

Thus, the PLS-DA model results show high precision and discrimination capacity for most PET sample classes, both virgin and recycled, at different stages of the production process. Virgin PET samples (vPET assp and vPET bssp) exhibit the highest precision and recall, indicating that the model has an excellent capacity to correctly identify these classes. Recycled samples (rPET30 and rPET40) have slightly lower precision and recall, especially in the bssp stage, suggesting that further analysis of these samples is needed.

In summary, the trained PLS-DA model is effective for classifying the different PET samples, with the potential to further improve precision and discrimination capacity in some specific areas. To corroborate this study, the trained model was tested on the samples. The results can be seen in Figure 16.

Figure 16: Trained model testing on the samples. On the left, an image taken with a normal camera. On the right, inference results where each colour corresponds to a different class.

6. Conclusion and outlook

6.1. Controlled aging and chemical analysis

The controlled aging and chemical analysis of several PET samples revealed the following key findings:

- Progressive discoloration was observed in all materials with aging, but the samples remained physically intact. Images of PET 5530, PET 5140 nIV, and PET 5140 IV aged up to 400 and 800 hours will be analyzed in future studies.
- The thermal extraction process identified benzyl benzoate in both virgin and recycled PET samples. However, benzyl benzoate, commonly used as a textile additive, cannot serve as a reliable marker for recycled content. Semi-quantitative analysis showed no meaningful differences between virgin and recycled PET.
- Viscosity was found to be dependent on the interplay between chain scission and crosslinking.
- The stiffness of PET samples remained constant up to 200 hours of QUV aging. While maximum strength showed some variation, a significant reduction in strain at break was observed for all samples, with PET 5530 and PET 5140 IV being more affected.
- There was a severe reduction in impact resistance across all samples, indicating increased brittleness due to aging.
- The glass transition temperature (Tg) remained constant for all samples. However, there was a slight increase in crystallization temperature (Tc) and crystallinity (Xc), suggesting faster crystallization due to aging. Changes in thermal degradation temperature (Td) were minimal and inconclusive.

In summary, while some analytical methods show promise, further research is needed to develop reliable markers for distinguishing recycled content. These findings emphasize the complexity of using chemical and physical properties to identify recycled materials, highlighting the need for continued investigation and refinement of these techniques.

6.2. Hyperspectral imaging

These months of the project have served as an initial phase where the NIR hyperspectral technology has been tested on various PET samples. This technology operates with considerable reliability for classifying different types of plastics, such as PVC, PP, PET, etc. [see Section 2]. However, in this task, the objective was to study its potential in identifying the unique features of each sample of a single material, PET in this case, at different stages of its lifecycle (recycling, process stages, etc.). As seen in Figure 13, since it is the same material, the spectral fingerprint is very similar. Therefore, using AI, the technology's ability to find nuances that allow one sample to be differentiated from another has been studied. The results obtained so far indicate that it is appropriate to continue exploring this identification with this technology. The results are not yet valid to determine if the technology is useful due to the insufficient representativeness of sample variability, but they are promising since it has managed to differentiate samples with very similar spectral fingerprints with an MCC of 0.84.

6.3. Outlook

This was the first version of the work done in T5.4 and will be updated in a final version D5.7 to be submitted in M42.

Regarding the controlled aging and chemical analysis, CTB will continue to analyze the 400 and 800 QUV aged samples. Furthermore, the chemical analysis work will be expanded by analyzing new samples from post-consumer origin (bottle grade polyesters and textile grade polyesters). Also known samples with recycled content will be made by CTB to be used for the analysis (see Table 9).

Regarding the work using HIS, CTB and CIRCE have defined the work to be carried out in the coming months. This work will consist of quantifying the percentage of virgin/recycled PET in each sample. To this end, CTB has been working on this and has provided CIRCE with samples containing different percentages of virgin/recycled PET (see Table 9). CIRCE will use hyperspectral technology in the NIR and VNIR to achieve this distinction. Additionally, a conveyor belt will be used to simulate the classification process in a semi-industrial environment.

Table 9: New samples.

Sample	Virgin/recycled PET (%)	Weight (kg)
M00	100/0	1.8
M01	90/10	1.4
M02	80/20	1.7
M03	70/30	1.9
M04	60/40	1.8
M05	50/50	1.9
M06	40/60	1.9
M07	30/70	1.8
M08	20/80	1.8
M09	10/90	1.8
M10	0/100	1.9

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