END-OF- LIFE COMPOSITES IDENTIFICATION BY SPECTROSCOPIC TECHNIQUES

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Keywords*: spectroscopy, composite, recycling, sorting, waste, circular*

1 Introduction

In the context of chemical recycling, spectroscopy technologies have a great potential to support the correct identification of composite materials. In this work, we investigate Laser Induced Breakdown Spectroscopy (LIBS) and hyperspectral imaging powered by machine learning techniques analysis for identifying and classifying composite materials used in Wind Turbine Blades, allowing for an accurate identification without the need of sample preparation or destruction.

In-situ material identification is one the challenges for chemical recycling of End-of-Life glass fiber composite (GFRP) waste from wind turbine blades utilizing solvolysis methods [1]. Recent research [2][3] reviewed present status of the application of several spectroscopic methods and their scope in developing a photonic sensor-based sorting system because of their speed, specificity and need of only minimal human involvement for routine use. Chemical recycling of fiber-reinforced polymer matrix composites is a very active topic of research and development. In view of the appearance of emerging solutions, there is a need to develop finer identification techniques for multi-material waste polymers.

LIBS (Laser Induced Breakdown Spectroscopy) is a chemical characterization technology that provides an accurate and quick in situ chemical analysis and has achieved a promising performance for organic polymer identification. Hyperspectral imaging in SWIR range has emerged recently, as it combines the power of digital imaging and spectroscopy. Every pixel in a hyperspectral image provides local spectral information across a large number of narrow bands. That is, instead of collecting a single spectrum, this technique records a hyperspectral image (HSI) of the sample, providing a pre-screening of the local chemical composition and its distribution.

In this development, we analyze optical spectrum data from both spectroscopic techniques for the characterization of the most common composite materials used in the wind turbine blades prior chemical recycling development. A set of coupons with common resin&hardener material combinations were prepared for model development. Data acquisition and preprocessing steps to maximise the valuable information obtained from experimental spectroscopic data is presented. Multivariate analysis, data dimensionality reduction as well as classification and segmentation algorithms are evaluated and selected, and thus, the spectral data obtained by both techniques is being analyzed to classify the materials. This work focusses mainly on the material identification phase (which takes place after waste collection and before sorting and recycling), but we also analyze its implementation for sorting as well as future perspectives that can contribute to increase the circularity of composites value chains.

Acknowledgement

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promising tool for plastic waste management. TrAC Trends in Analytical Chemistry, 116534.

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31/07/2023, Belfast . ICCM23

Camilo Prieto (AIMEN)

This project has received funding from the European Union's Horizon Europe research and innovation programme under grant agreement No 101058089.

Outline

Introduction

Experimental setup

- Laser Induced Breakdown Spectroscopy (LIBS)
- Hyperspectral imaging (HSI)
- Materials: WTB composites and matrices

LIBS results

- Experimental protocol development
- Data analysis and classification

Hyperspectral results

- Optical characterization and data analysis

Conclusions and next steps

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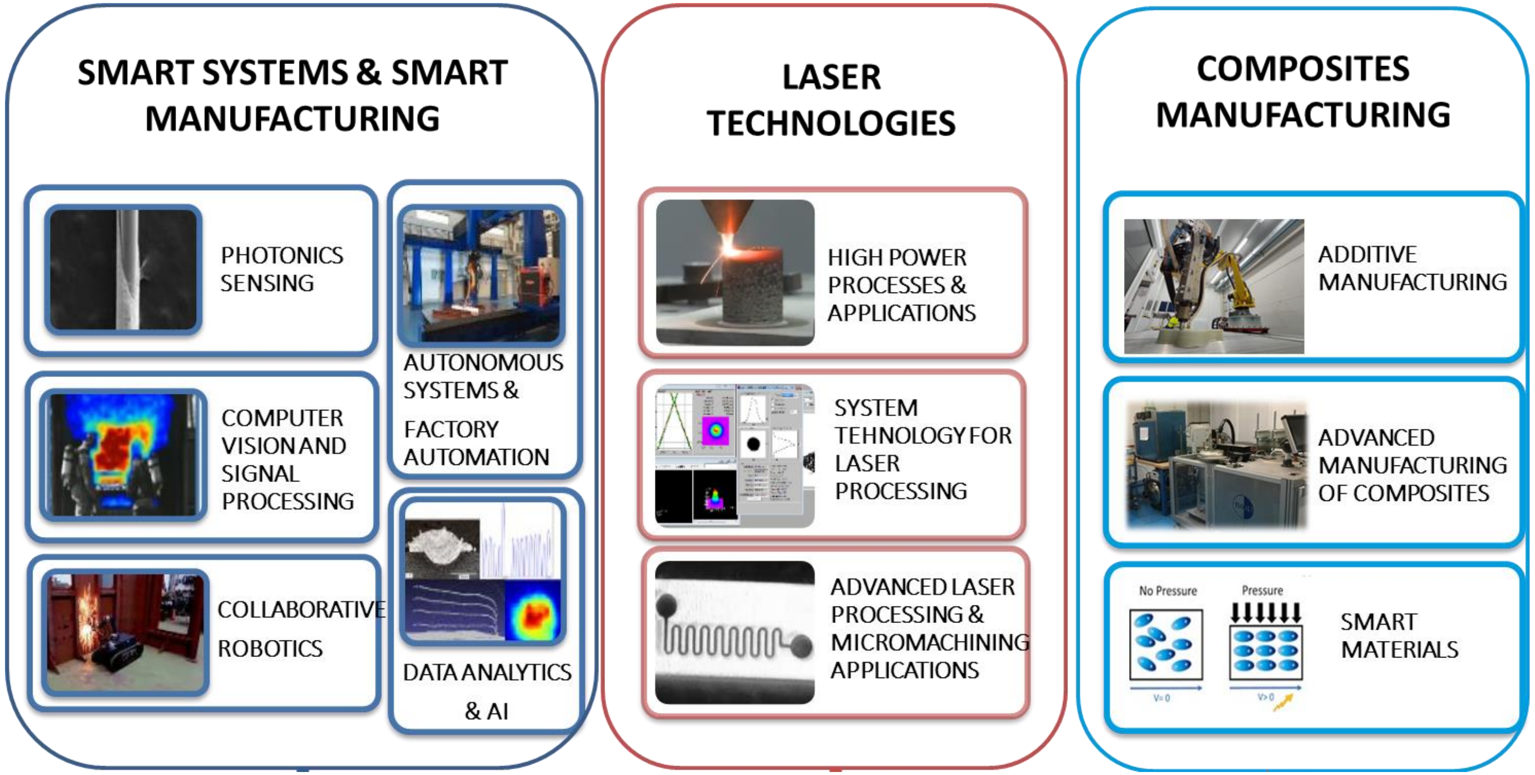
Introduction: AIMEN Technology Center

Industry supported, private centre. R&D and high-added value technology services

> 50 R&D projects per year.   Headcount: 280 *(50% in R&D)*

R&D Units



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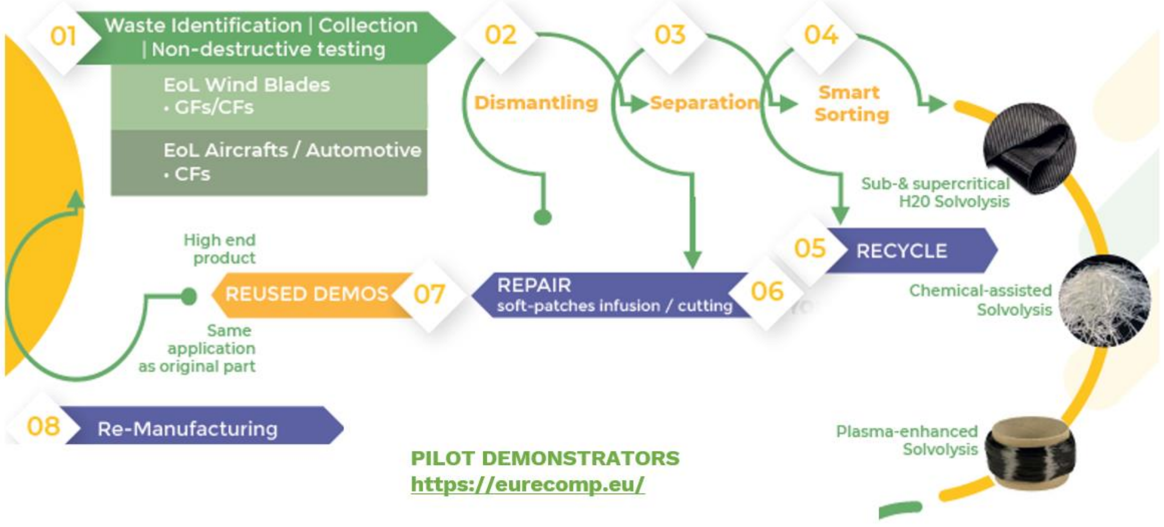
Introduction: EURECOMP project

EU funded collaborative research project with a strong focus on circularity, set out to provide sustainable methods towards recycling and reuse of composite materials

Chemical recycling of composites is a very active topic of research and development for fiber-reinforced polymer matrix: solvolysis



- In-situ material identification is one the challenges for chemical recycling of End-of-Life composite waste from wind turbine blades utilizing solvolysis methods
- **OBJECTIVE**: To develop innovative identification systems enabling reuse and recycling of complex composite materials by means of spectroscopic techniques Laser Induced Breakdown Spectroscopy (LIBS) and Hyperspectral Imaging (HSI) with machine learning

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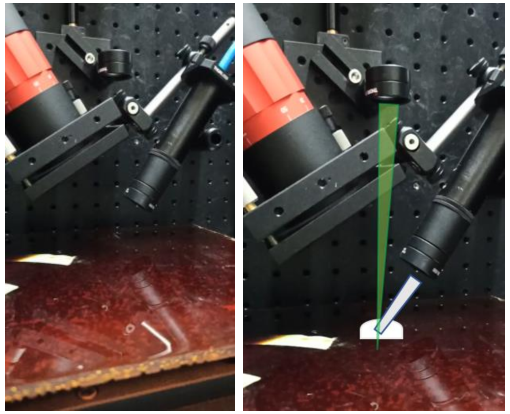
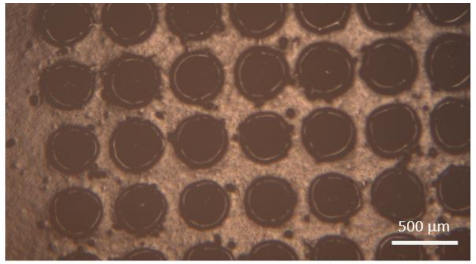
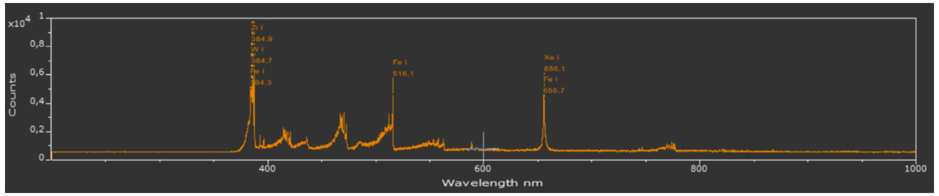
Experimental set up: LIBS

LIBS technique

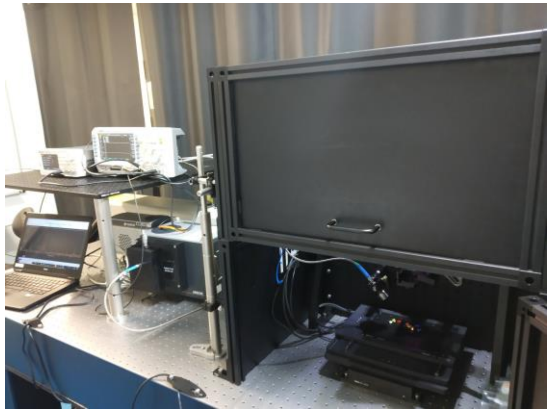


- Analysis of plasma generated during laser ablation: Atomic - molecular emission spectroscopic technique
- Provides information about chemical composition of sample





LIBS system



- Nanosecond laser. 515nm 10Hz
- Spectrograph: motorized 4-turret spectral gratings
- iCCD for temporal resolved plasmas signal acquisition
- Automated motion control system for LIBS datasets collection

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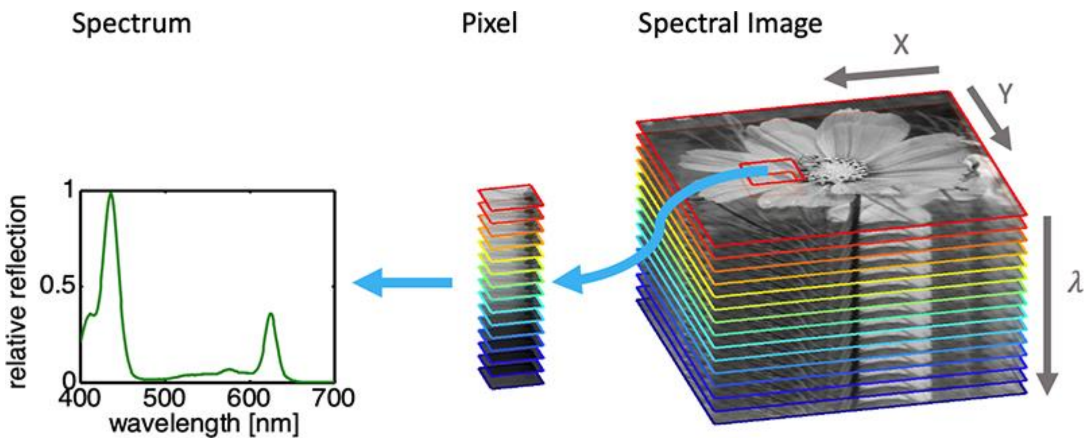
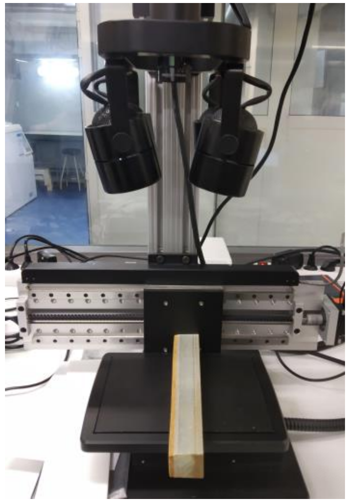
Experimental setup: Hyperspectral Imaging (HSI)



Multispectral / hyperspectral combines the power of digital imaging and spectroscopy.

Every pixel in the image provides local spectral information across a large number of spectral bands.

Hyperspectral imaging system at AIMEN



- Line scan (push-broom) workbench set up
  - 300 spectral bands: 400-1000nm
  - 2,1nm spectral resolution
  - 50mm objective, FoV:10x40cm
- Software for acquisition (optical characterization of WTB composites) and data analysis

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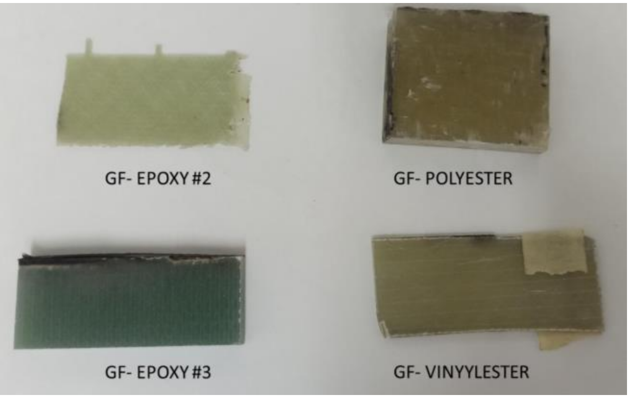
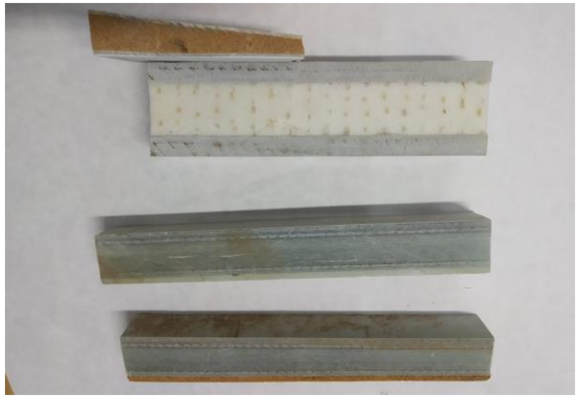
Experimental setup: Materials

EoL WTB GF-composites

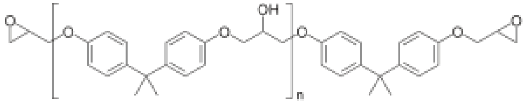
LIBS



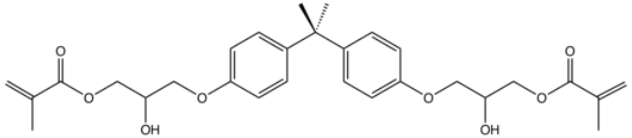
HSI



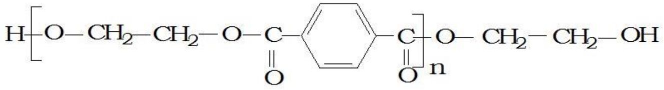
Epoxy



Vinylester

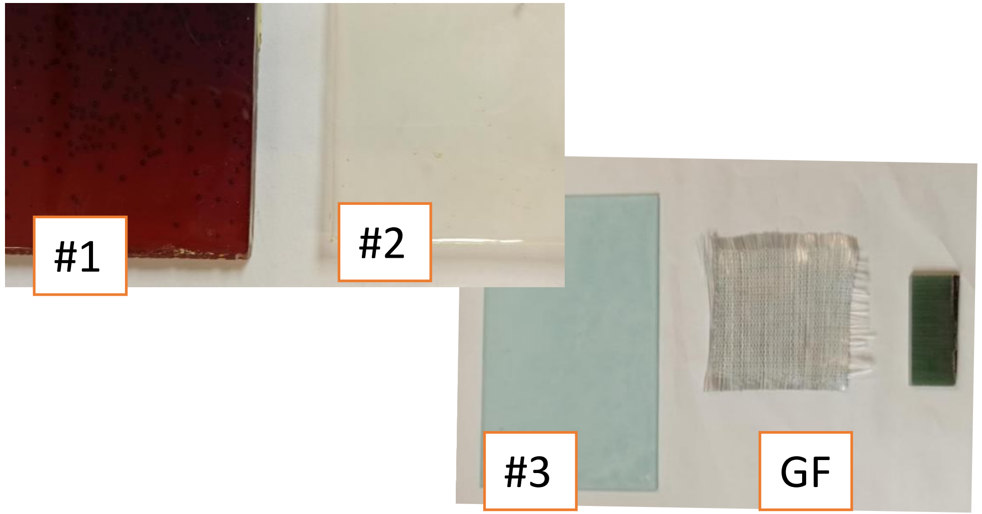


Polyester



Reference materials

Matrix (resin+hardener) identification



	Resin (Epoxy)	Hardener	Manufacturer
#1	Epikote	3R	Hexion/ Cidetec
#2	Infugreen	SD3304	Sicomin
#3	Epikote	MGS RIMH 137	Hexion

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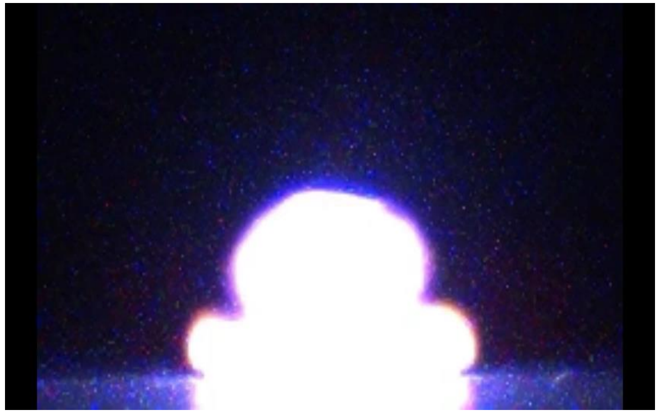
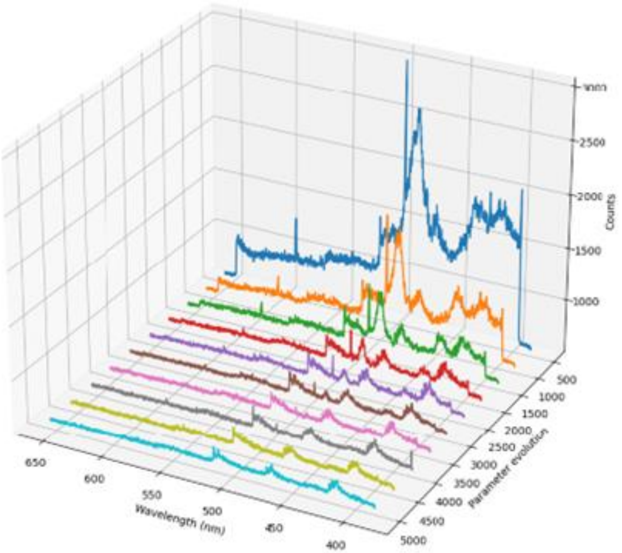
Materials information and LIBS spectra

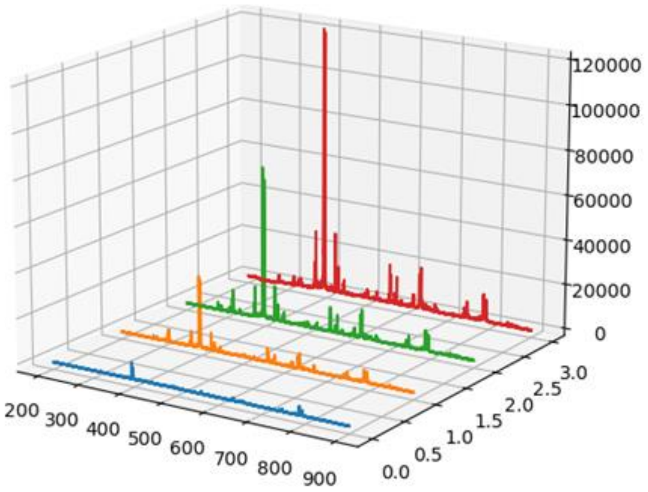
- LIBS spectra acquisition
- Preprocessing: spectra cleaning (remove outliers, remove background, etc); spectra normalization
- Data extraction and variable selection: (spectral descriptors and others feature extraction & selection); data distribution transformation (transform the data distribution to be as normal as possible)
- PCAs for dimensionality reduction; machine learning model creation (KNN classifier)

Spatio-temporal study of plasma for signal to noise optimization

- Laser energy/spot, collection optics
- Acquisition settings (grating, iCCD)







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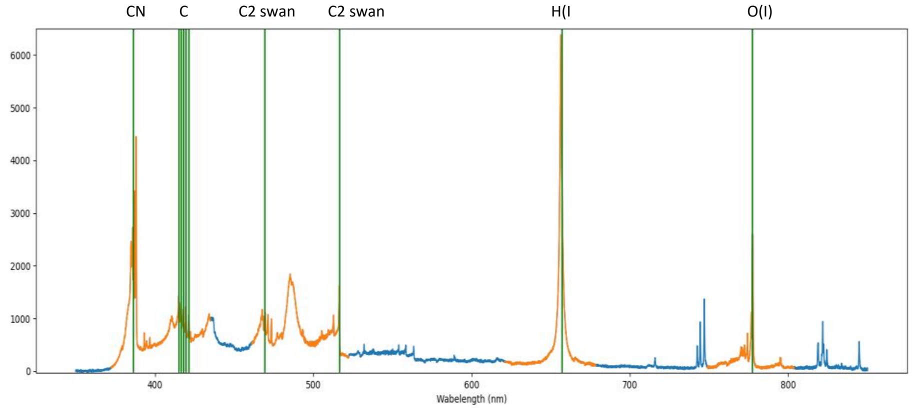
Experimental protocol: LIBS on composites

Reference LIBS spectra for polymer

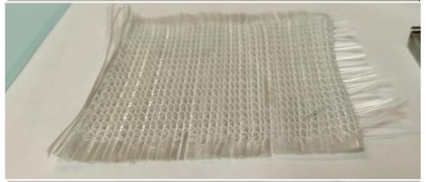


Spectral descriptors definition

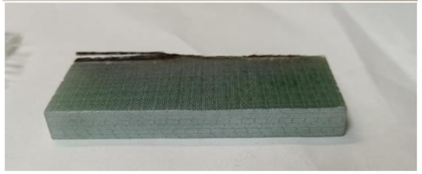
	name	type	b1	b2
0	CN_UVban_a	ABL	386.67	387.41
1	CN_UVban_b	ABL	387.42	388.65
2	C2_swanband_fm1	ABL	473.22	474.24
3	C2_swanband	ABL	516.23	516.75
4	H_I	ABL	650.19	663.74
5	N_I	ABL	746.55	747.39
6	O_I	ABL	776.51	778.42
7	CN_UVban_a	PBL	386.67	387.41
8	CN_UVban_b	PBL	387.42	388.65
9	C2_swanband_fm1	PBL	473.22	474.24
10	C2_swanband	PBL	516.23	516.75
11	H_I	PBL	650.19	663.74
12	N_I	PBL	746.55	747.39
13	O_I	PBL	776.51	778.42
14	CN_UVban_a	PWR	386.67	387.41
15	CN_UVban_b	PWR	387.42	388.65
16	C	PWR	414.92	421.85
17	C2_swanband_fm1	PWR	473.22	474.24
18	C2_swanband	PWR	516.23	516.75
19	H_I	PWR	650.19	663.74
20	N_I	PWR	746.55	747.39
21	O_I	PWR	776.51	778.42

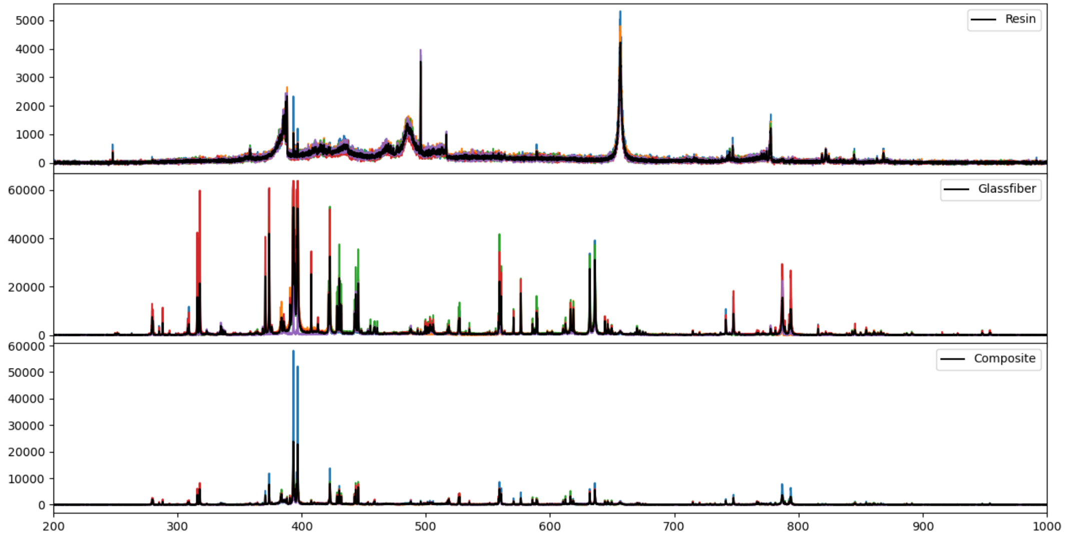
Matrix
Resin +hardener

Glass fiber



Composite





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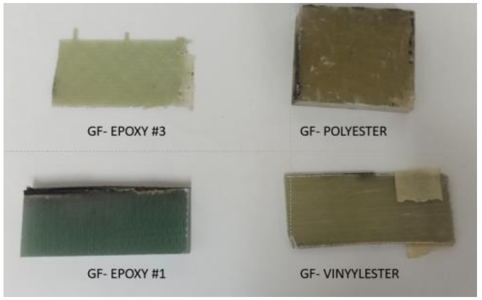
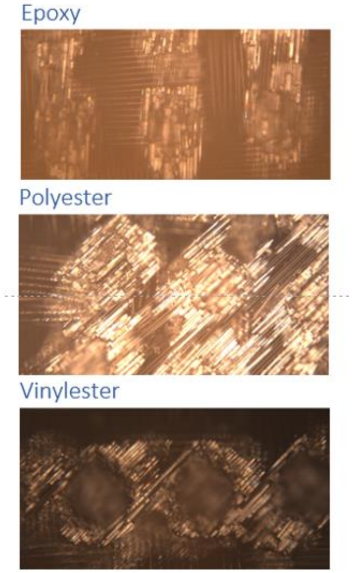
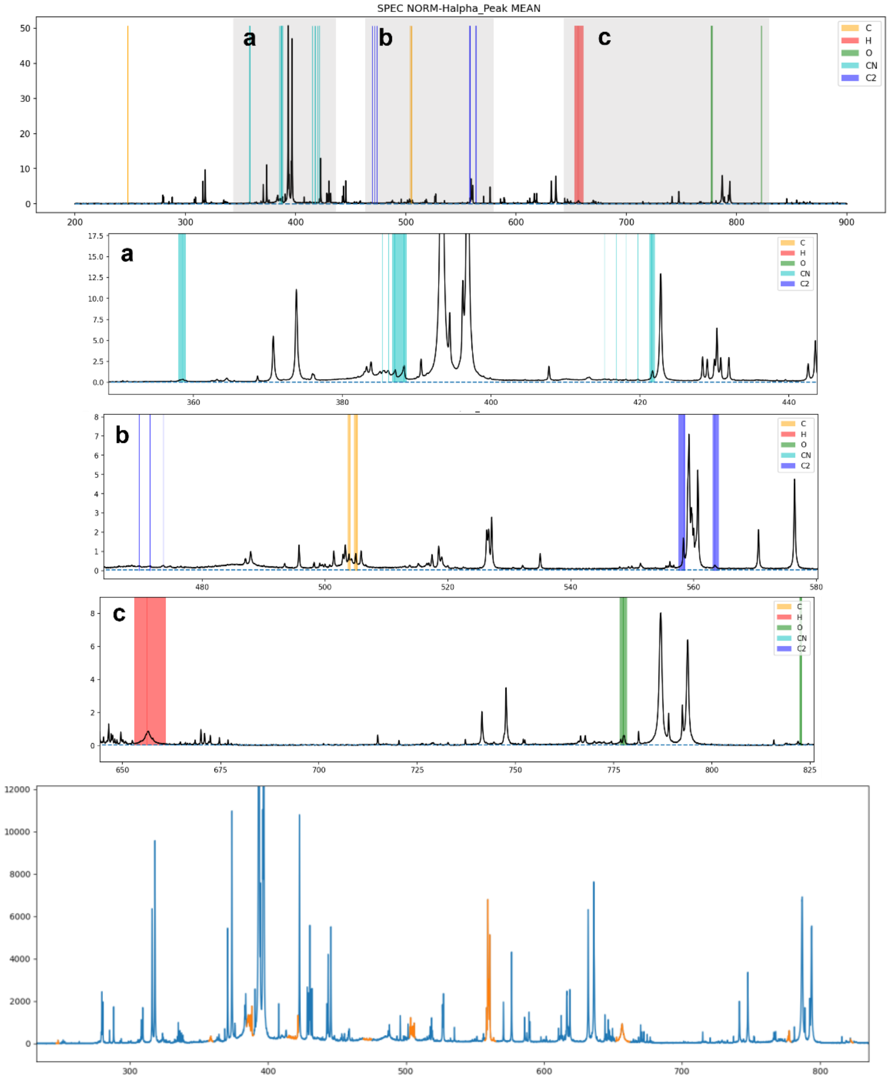
LIBS results: GF-composites identification



- 70 spectra per material
- optical microscope images (500mm pitch)



Spectra analysis - selection organics



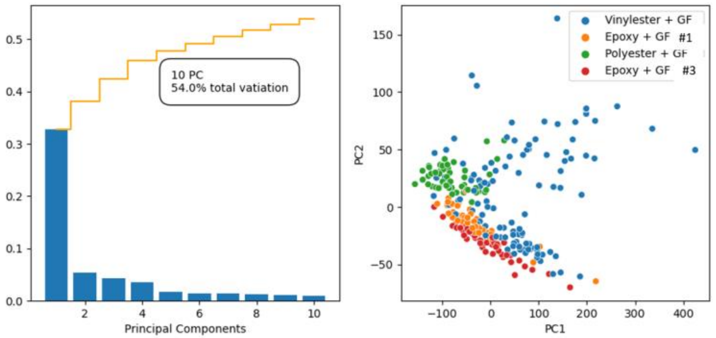
Descriptors

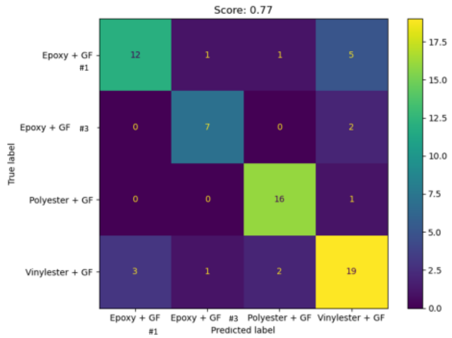
- area with baseline subtraction (ABL)
- line peak with baseline subtraction (PBL)
- line peak in a wave range (PWR)

Normalization

- maximum ; - sum =1000 ; - Std scaler;
- **H-alpha: 656,5nm**;

PCA and KNN





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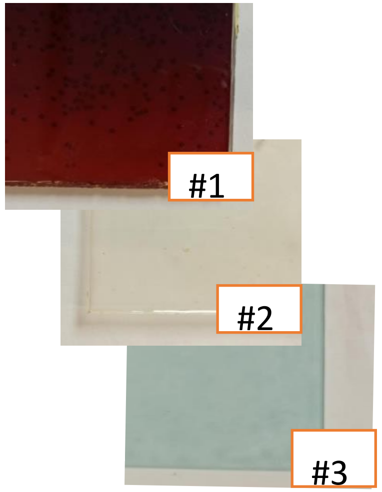
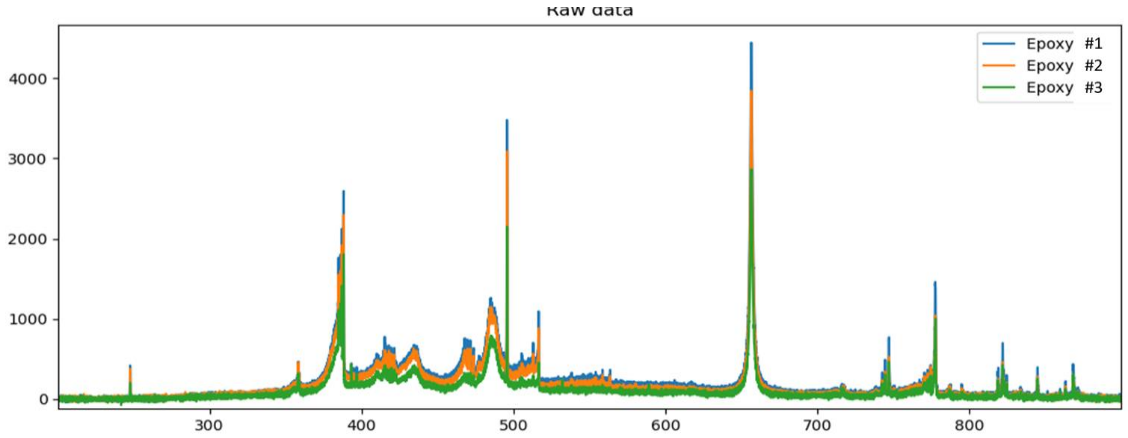
LIBS results: matrix identification

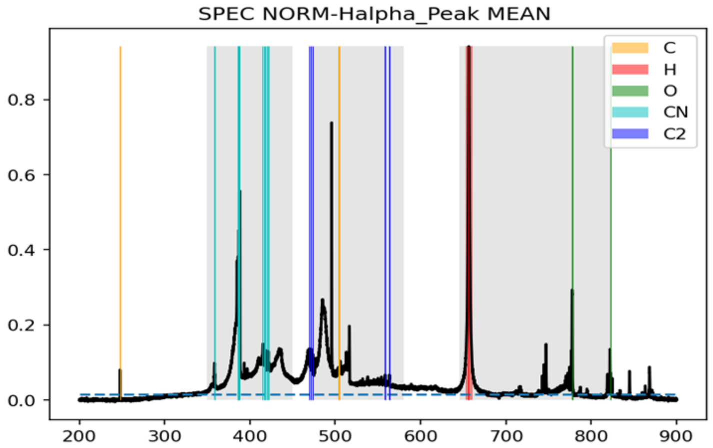


60 spectra per resin matrix

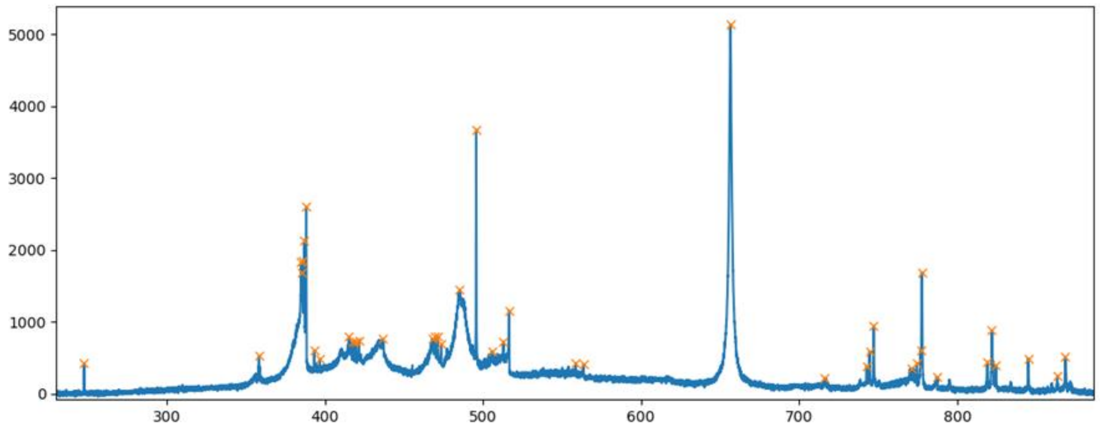
Raw spectra



Normalization



Feature extraction and selection



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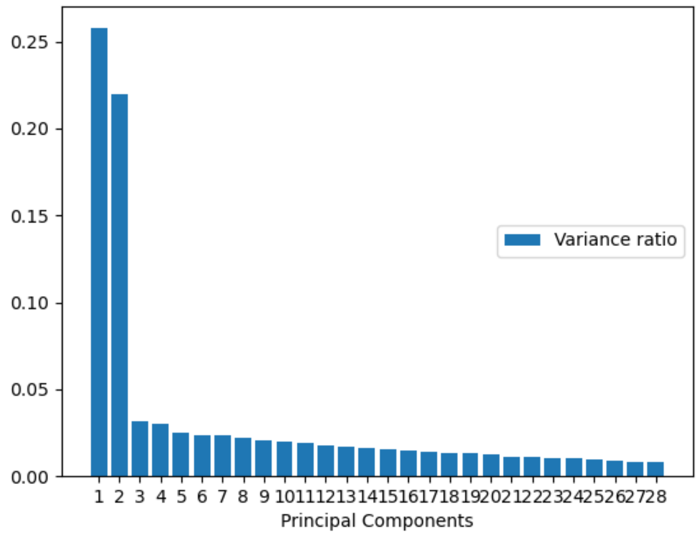
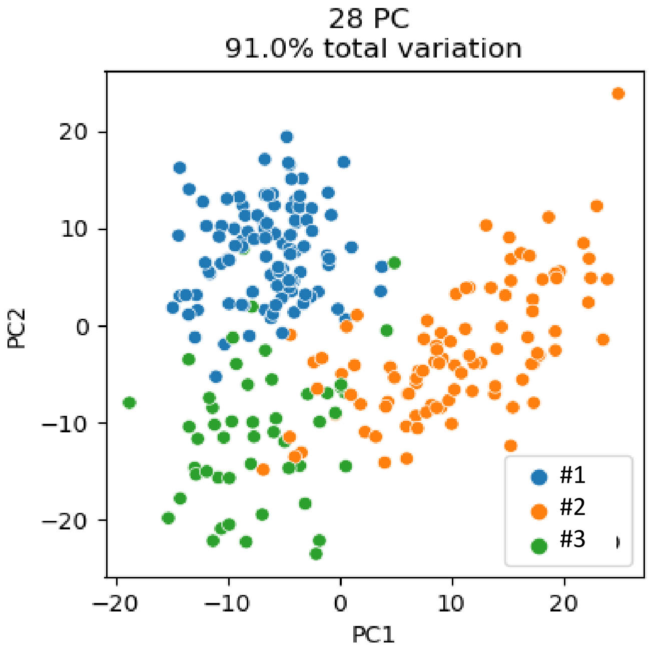
LIBS results: matrix identification. KNN classifier

Feature selection (spectral peaks/descriptors and ratios): **Variance threshold (0.03) + boxcox + ANOVA -> PCA (0.9 total variance) -> KNN (**K-nearest neighbours)

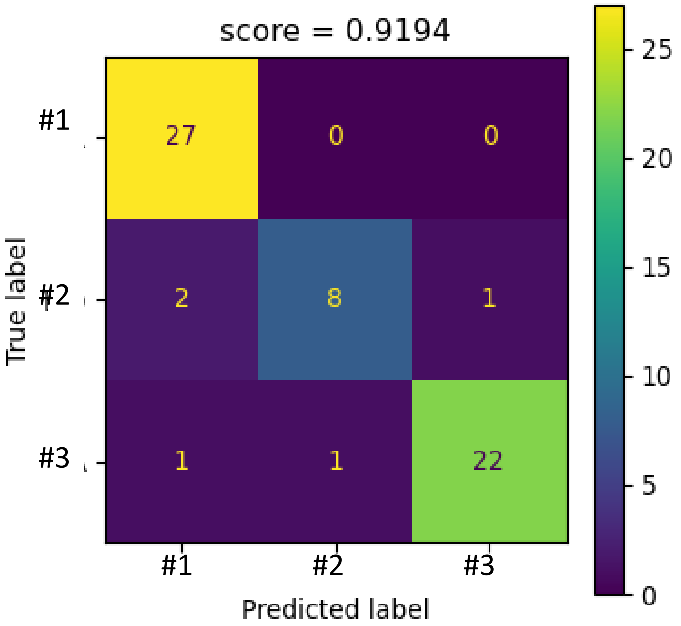
- ANOVA (Analysis of Variance) Analysis of Variance is a statistical method, used to check the means of two or more groups that are significantly different from each other
- Supervised Machine Learning model for classification: (KNN) 60 spectra per material (70 training/30 test)

PC- variance





KNN. Confusion Matrix



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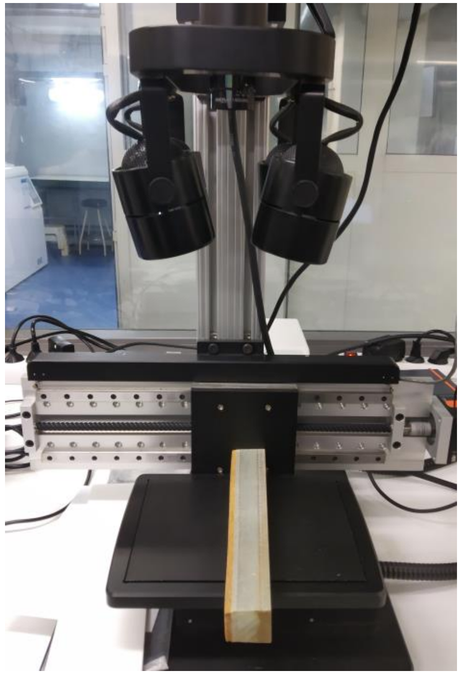
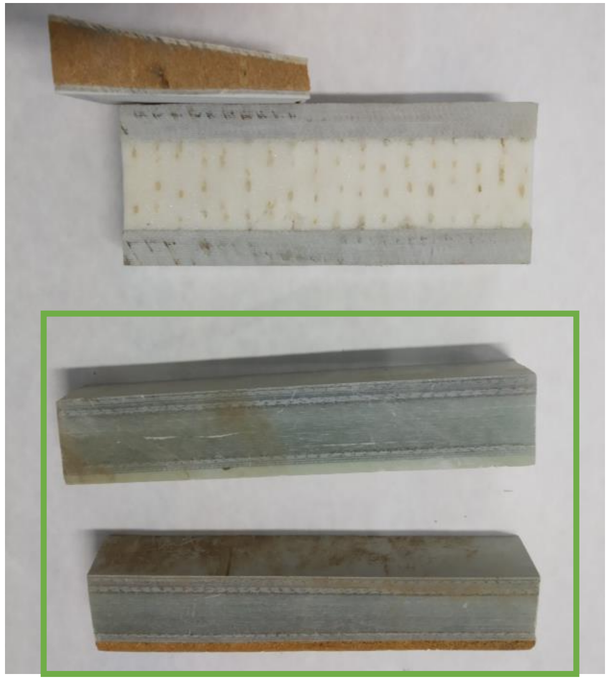
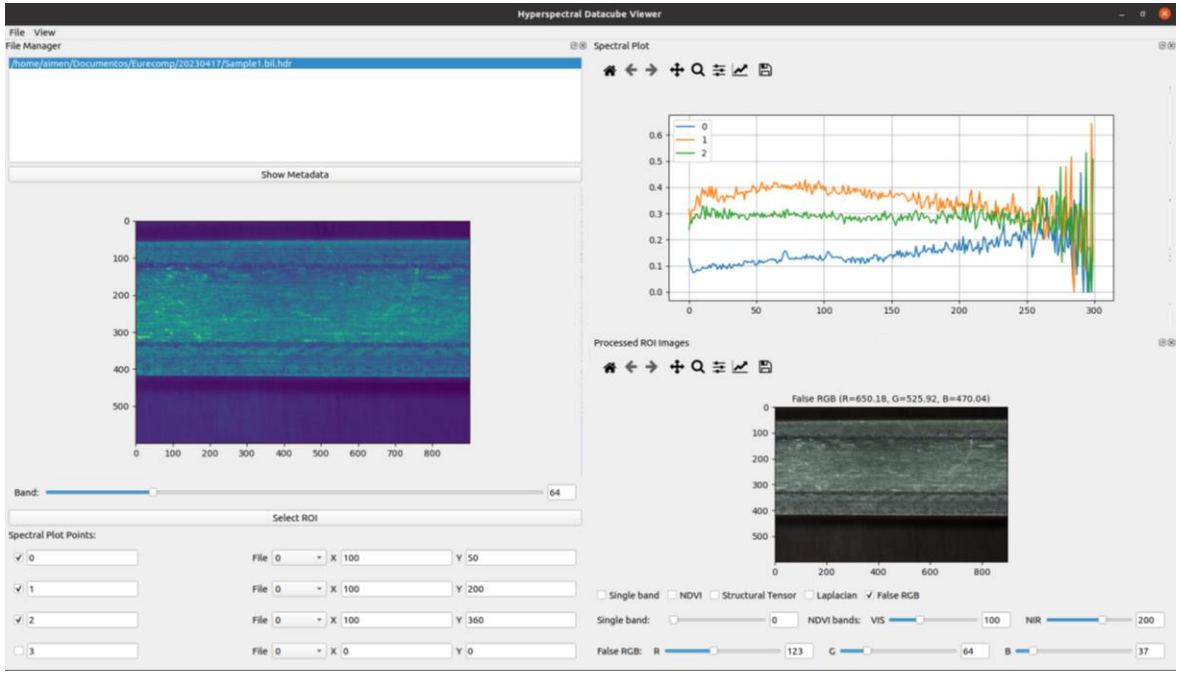
HSI results: GF- WTB composites

- Optical characterization: spectral reflectance and spectral bands selection
- Segmentation of layers, identification and classification of materials: useful for pre-screening materials family during WTB EoL operations







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Conclusions and future work

- ✓ LIBS system capable of identifying composite resin matrix -> recyclers to make decisions about the chemical process conditions
- ✓ HSI technique demonstrated as a potential approach for pre-screening/sorting EoL WTB composites -> circularity pathways

FUTURE WORK

- ➢ LIBS development: Extend material range of matrixes and composites investigated. Continue work with CF-composites. Explore other ML algorithms
- ➢ Transfer developments to multispectral imaging snapshot technology –under specification (lower number of bands and no movement between part and sensor required)

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Consortium

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Thank you!

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