Analytical method development and application to food and beverages

DR. EVA CSEPERKE VIZSOLYI

OFI- Österreichisches Forschungsinstitut für Chemie und Technik

Projektteam:
Eva Cseperke Vizsolyi, Yuliya Voronko, Barbara Zottl, Michael Washüttl/ OFI
Beate Raab, Martin G. J. Löder, Christian Laforsch / UBT
Kristina Enders, Dieter Fischer / IPF
Katharina Stollewerk, Isabel Niederkofler, Julian Drausinger / LVA
Benedikt Hufnagl / Hufnagl Chemometrics
Martina Zederbauer / LCNÖ der ecoplus; Gabriele Gedik / IVLV

MARII Workshop
Madrid
2025

OFI - Österreichisches Forschungsinstitut für Chemie und Technik

independent, accredited, austrian research and testing institute in the fields of materials applications & building renewal

OFI figures

1946	founded as private research and testing institute
116	employees
~ 18 Mio.	EUR turnover (2024)
~ 1.700	customers
~ 700	accredited testing, inspection and certification procedures

OFI – Department packaging, recycling & dangerous goods

Testing, research, development & advice in the packaging sector

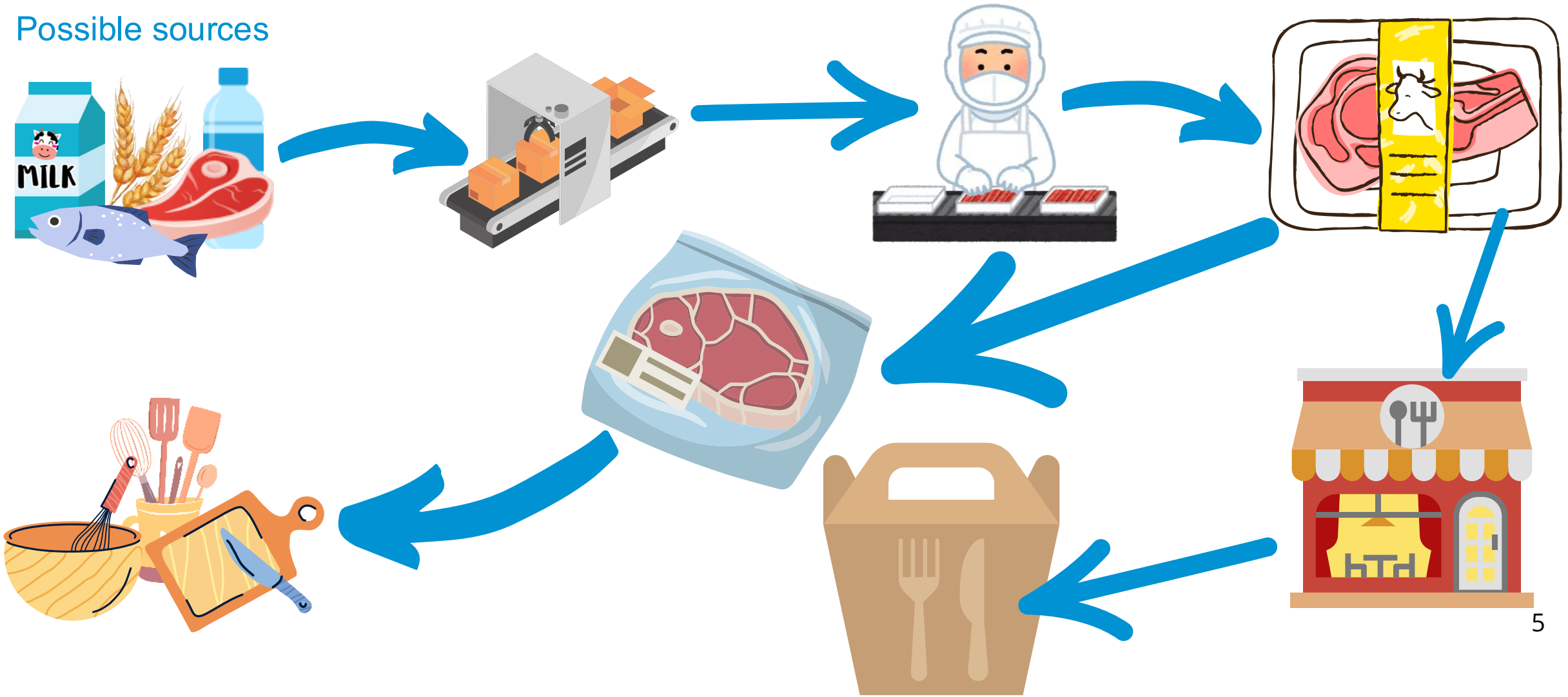
- ✓ 30 years of packaging expertise (extensive laboratory infrastructure and an interdisciplinary team)
- ✓ Packaging inspection, damage assessment and training
- ✓ Food law assessments (special area NIAS analysis), risk assessment and product protection
- ✓ Specialization in recycling and sustainability
- ✓ Transparent, quantitative assessment of the technical recyclability according to cyclos HTP (CHI)
- ✓ Customized development of individual, innovative packaging developments
- ✓ R&D-Expertise – participation in and management of international industrial branch projects

How do microplastics enter our food?

Microplastics are found everywhere around us

How do microplastics enter our food?

Possible sources



About the project from the beginning

Research partners

Funding

microplastic@food

Goal: Development of a metho
eva
mic

MICROPLEXFOOD

International project for the assessment of the presence/absence of microplastic in complex food and potential sources of origin

Associations

Industry partners

Food industry
Packaging industry

Project start: October 2023
Project end: February 2026

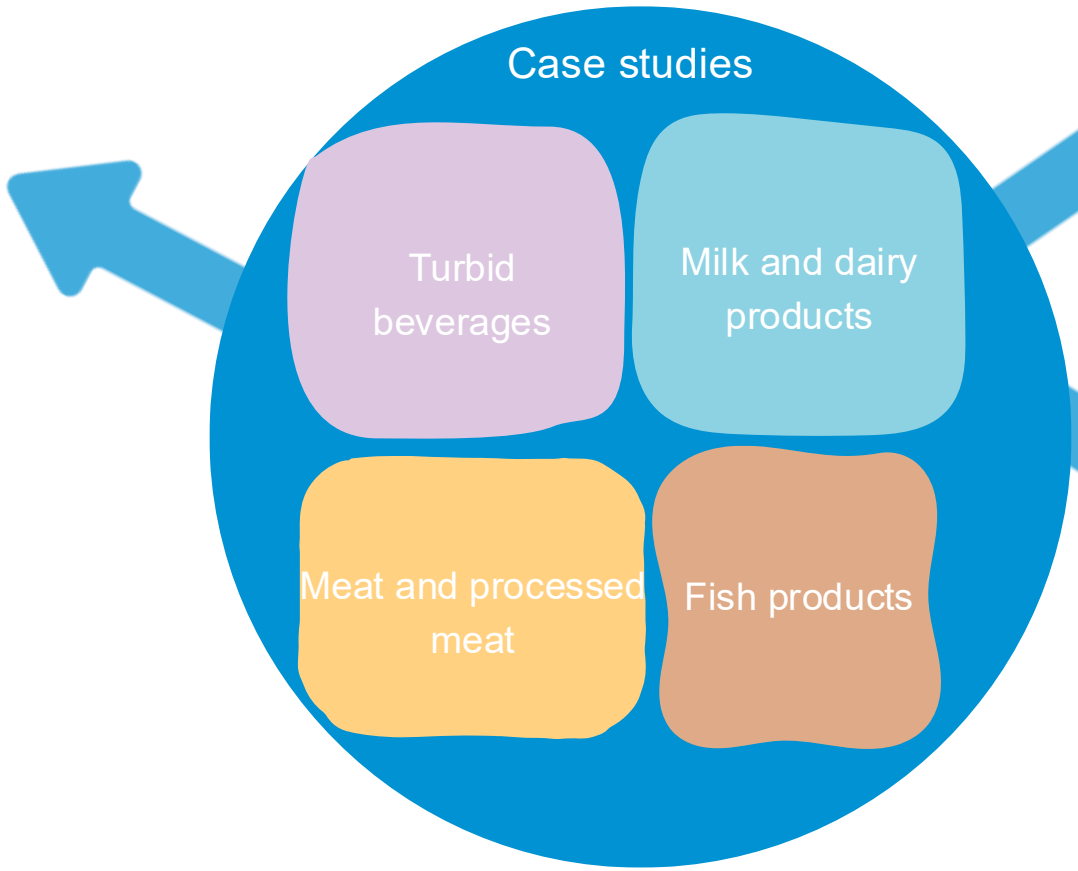
Project start: July 2021
Project end: June 2023

MICROPLEXFOOD

Standardisation

Sample preparation methods

SOPs



Analytical method development for spectroscopy and gaschromatography

Evaluation method development using machine learning

Goals and Deliverables

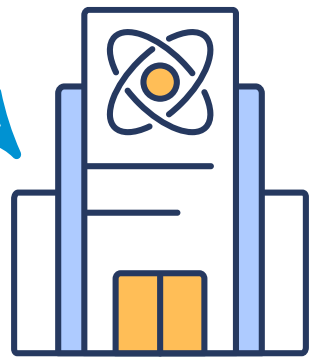
Goal:

to develop analytical methods for the detection and identification of microplastics in complex food matrices

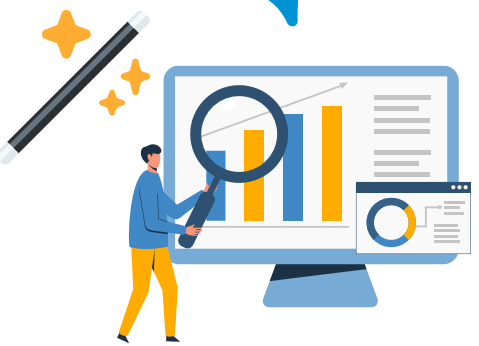
6 workpackages:

1. Project management
2. Development of purification protocols
3. Analytical methods
4. Data analysis, evaluation and visulisation
5. Analysis of real samples
6. Dissemination and Communication

to create SOPs which are widely applicable, aiming to help standardisation



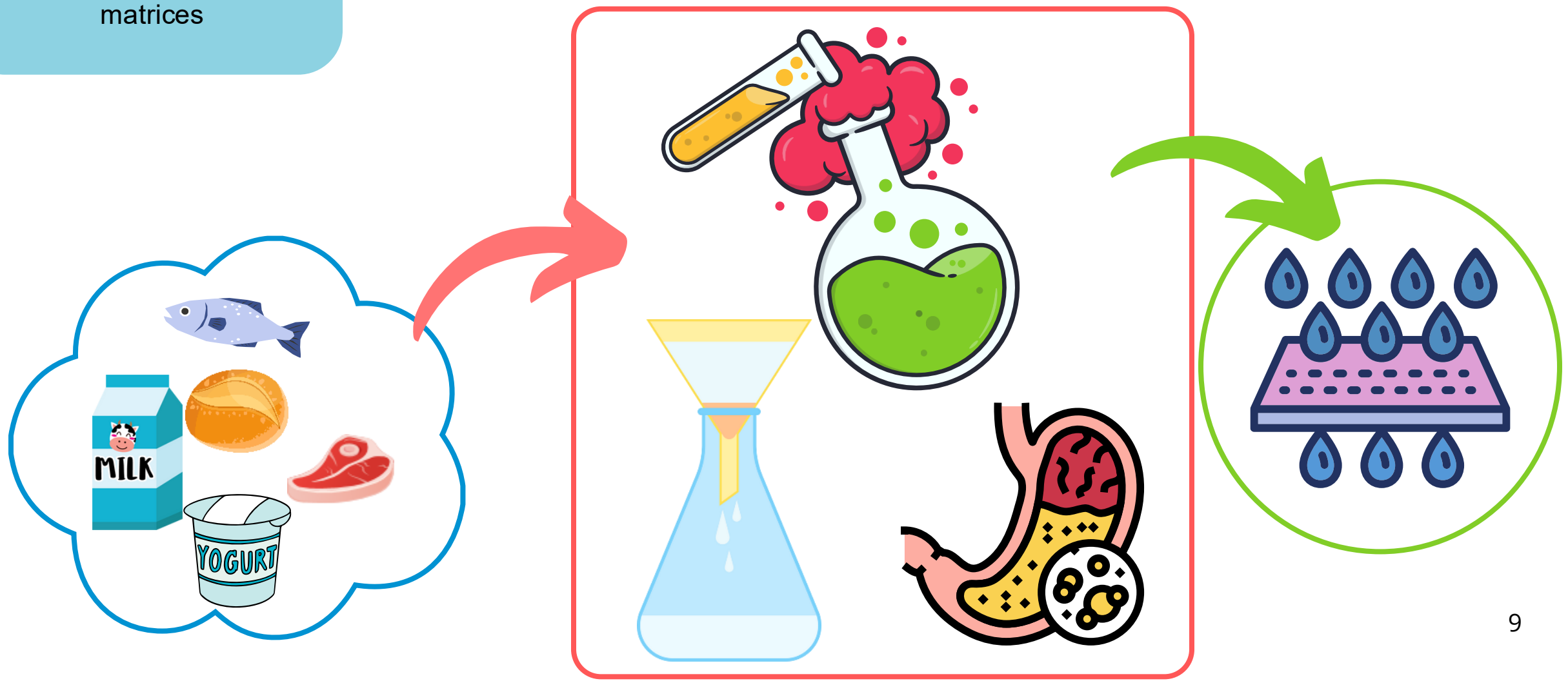
to combine particle based measurements (µFTIR and µRaman) with mass based measurement (PYR-GC/MS)



Mitigation strategy examples for the reduction of MP in the selected categories

WP2- Development of purification protocols

Creating the SOPs for sample preparation of complex food matrices



WP2- Development of purification protocols

Creating the SOPs for sample preparation of complex food matrices

Milk
Standard Protocol

Dilution with 10% SDS and water, 1 day

*

1st Enzyme step: Protease, 1 day

*

2nd Enzyme step: Protease, 1 day

*

10% SDS, 1day

*

Filter rinsing (35% ethanol with 0,1% H_3PO_4)**

*

Analysis with Raman
(transport abroad to other research centre)

Working protocol

filter after 1st step

filter after last step

Milk
With additional components

Dilution with 10% SDS and **30% H_2O_2**, 1 day

*

1st Enzyme step: Protease, 1 day

*

2nd Enzyme step: Protease, 1 day

*

3rd Enzyme step: Cellulolytic enzyme mixture 1, 1day

*

10% SDS, **4 days**

*

Fenton oxidation

*

Filter rinsing (35% ethanol with 0,1% H_3PO_4)**

*

Analysis with Raman
(transport abroad to other research centre)

Working protocol

filter after 1st step

filter after last step

*Filtration (5µm), **Verification for non-plastic destruction necessary

WP2- Development of purification protocols

Creating the SOPs for sample preparation of complex food matrices

Plain yoghurt
Standard Protocol

Dilution with 10% SDS and water, 4 days

*

1st Enzyme step: Protease, 1 day

*

2nd Enzyme step: Protease, 1 day

*

3rd Enzyme step: Protease, 1 day

*

Filter rinsing (35% ethanol with 0,1% H_3PO_4)**

*

Analysis with Raman
(transport abroard to other research centre)

filter after 1st step

filter after last step

Working protocol ✓

Yoghurt
With additional components

Dilution with 10% SDS and **30% H_2O_2**, 4 days

*

1st Enzyme step: Protease, 1 day

*

2nd Enzyme step: Protease, 1 day

*

3rd Enzyme step: **Cellulolytic enzyme mixture 1 and 2**, 1day

*

Fenton oxidation

*

SDS step: 10% SDS (and 30% H_2O_2), 4 days

*

Fenton oxidation

*

SDS step: 10% SDS (and 30% H_2O_2), 2 days

*

4th Enzyme step: Cellulolytic enzyme mixture 1 and 2, 1day

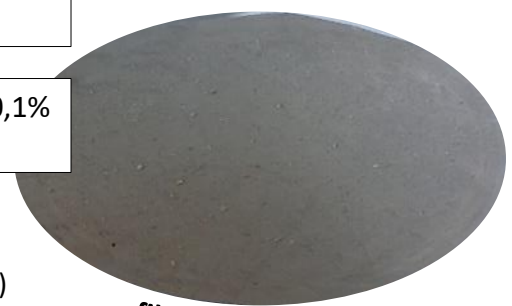
*

Filter rinsing (35% ethanol with 0,1% H_3PO_4)**

*

Analysis with Raman
(transport abroard to other research centre)

filter after 1st step



filter after last step

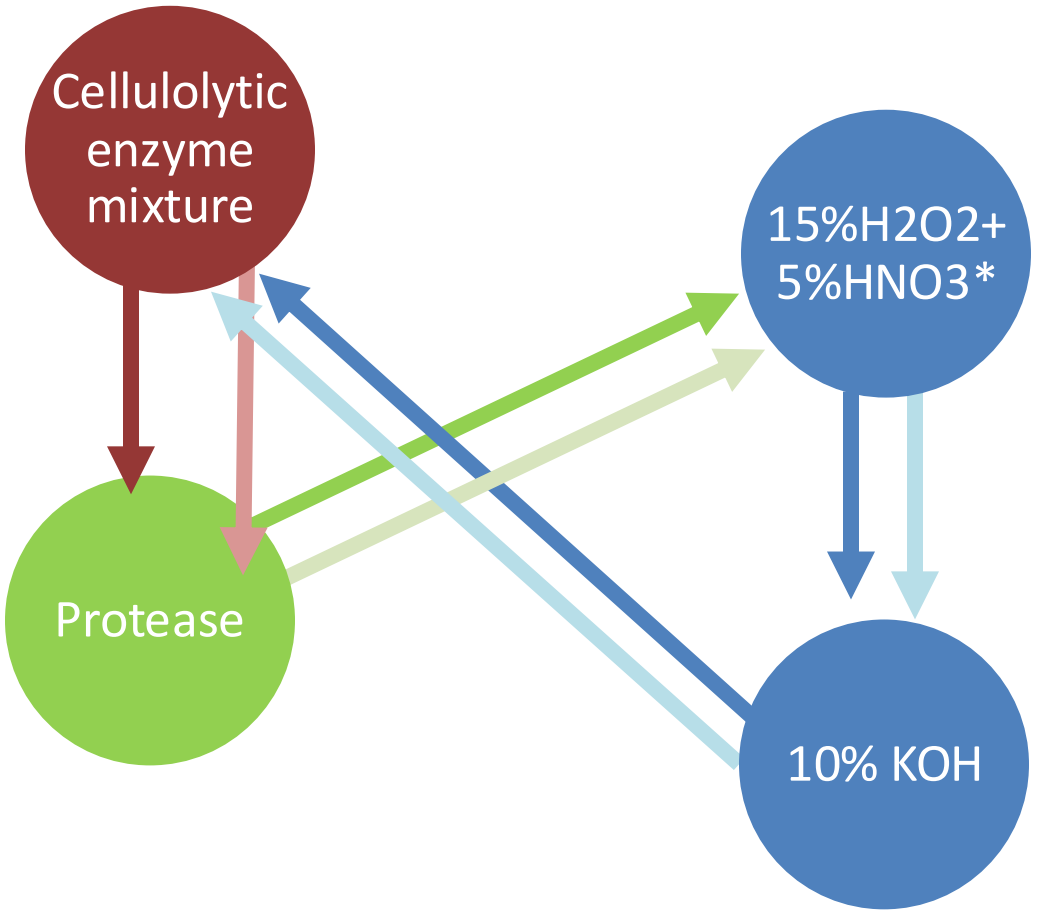
To be tested

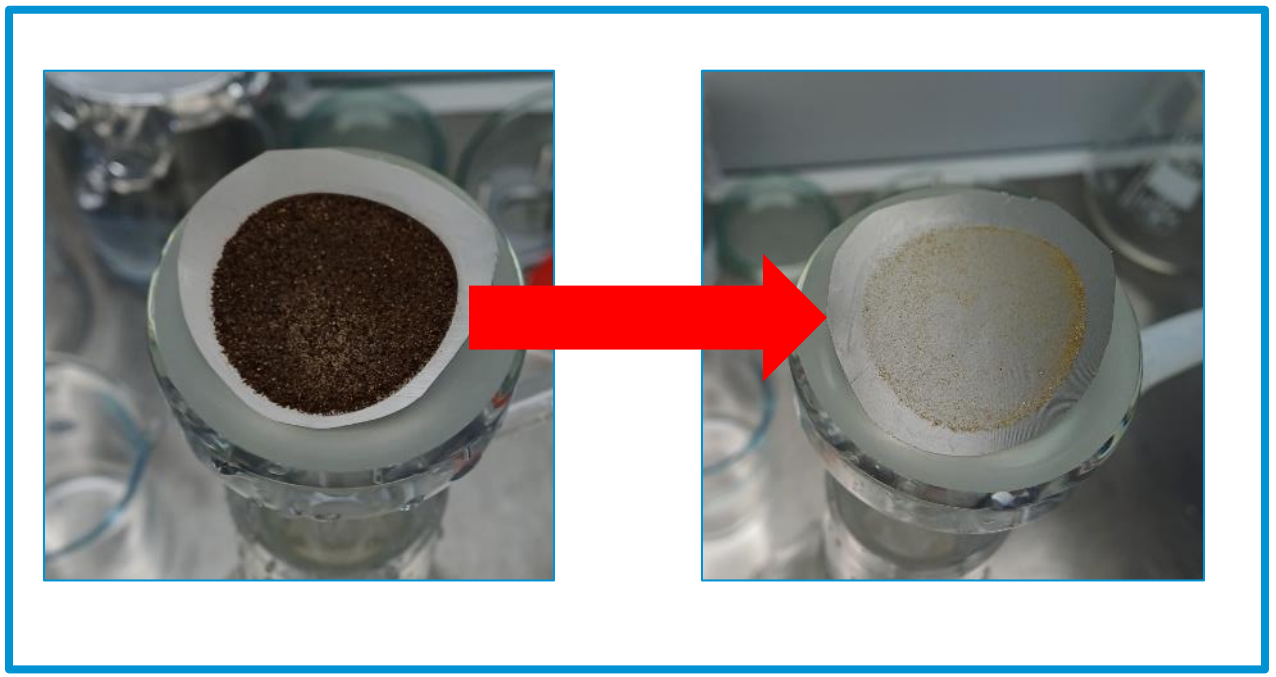
*Filtration (5µm), **Verification for non-plastic destruction necessary

Milled black pepper

WP2- Development of purification protocols

Creating the SOPs for sample preparation of complex food matrices





WP2- Development of purification protocols

Creating the SOPs for sample preparation of complex food matrices

Sample type	Analysed quantity	Protocol duration
Milk	1 litre	5 days
Milk with additional components	400 milliliters	8 days
Plain yoghurt	500 gram	8 days
Yoghurt with additional components	140 gram	14 days
Milled pepper	100 mg (2*50 mg)	18 days
Meat	10 g	14 days
Fisch	10 g	14 days

WP2- Development of purification protocols

Creating the SOPs for sample preparation of complex food matrices

Feedback loop from Analysis to digestion protocol development – necessary adaptations

The Aim:
Reduction of all unnecessary particle entry

1. Materials in use and devices/working processes
2. Matrix components of food product

	problem	solution/measurement	controlable /manageable	solved
Use of kitchen roll	Use of cellulose fibres	Change to Cleanroom wipers	controllable	yes
MilliQ-water withdrawal	The way of withdrawing	Optimization: water withdrawaling very close to outlet	controllable	yes
Sample transportation - packaging	Cross contamination from packaging material to sample	Use of aluminum foil, paper tape and cardboard filling material only	controllable	yes
Filtration device for working solutions	PSU filtration device leads to PSU particles in analysis	Filtration device made of glass	controllable	yes
Sample transportation - flasks	Glass flasks lead to particles in analysis	Currently no other solution considered	manageable (known particle entry)	yes
Airflow in working bench	Air flow could contribute to particle entry	Analysis of air samples in working bench	manageable (known particle entry)	yes
Process steps in protocol	Potential entry by each process step	Stage analysis of working process	manageable (known particle entry)	yes
Pressure flask used for rinsing in working process	Aluminum flask with internal thread	Stainless steel flask with extrenal thread	should be controllable	In progress

WP3- Analytical methods
Improving the µFTIR and µRaman methods
Development of the PYR-GC/MS method
Validation

Particle based measurement = results in particle number/sample

FTIR & Raman

Information about the particles (form, number/polymer type), size is available
Combination of optical and chemical information

High number of matrix-particles is disturbing

Particle size limits the measurements

Vs

Mass based measurement = results in mg/sample

PYR-GC/MS

Size of particles doesn't really matter

Mass-based results can be compared to other values easier

Extremely low sample size can be analysed

Matrix components cause distrurbance

Goal is to combine the two types of measurements

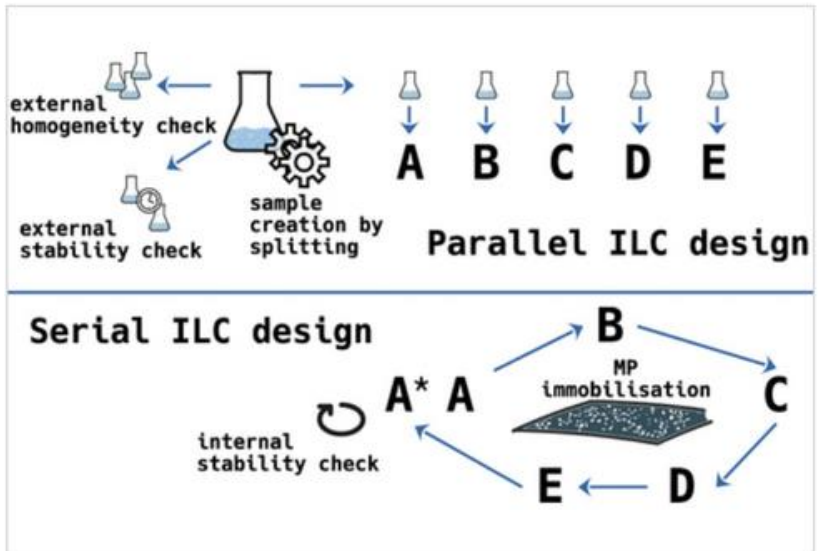
WP3- Analytical methods

Improving the µFTIR and µRaman methods

Development of the PYR-GC/MS method

Validation

ILC conducted in the previous project



Immobilised Ring Trial

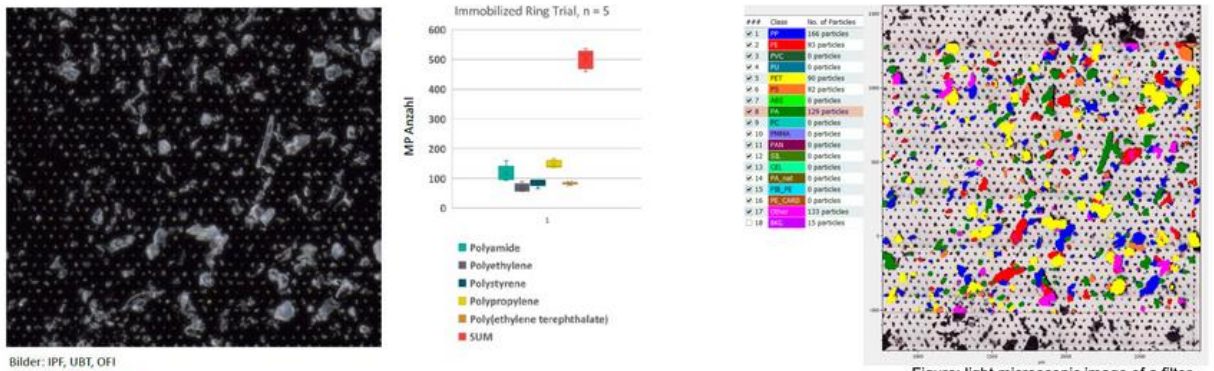


Figure: light microscopic image of a filter segment. IR image overlapped with the results from the machine learning program "Microplastics Finder"/Purency

Result:

- Analytical variation: 6% relative standard deviation

→ Low variation between the three institutes and spectroscopic methods

- Certain polymers lead to higher standard deviations (e.g. PA)

→ Improvement of the spectroscopic identification is necessary!

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What Goes Around Should Not Move Around: Immobilizing Microplastics as a New Approach for Analytical Ring Trials

Robin Lenz*, Kristina Enders, Eva Cseperke Vizsolyi, Mareike Schumacher, Julia Lötsch, Martin G. J. Löder, Gabriele Eder, Yuliya Voronko, José Manuel Andrade-Garda, Soledad Muniategui-Lorenzo, Christian Laforsch, Dieter Fischer*, and Matthias Labrenz*

WP3- Analytical methods

Improving the µFTIR and µRaman methods

Development of the PYR-GC/MS method

Validation

To validate the entire method, the validation of the instrument itself is not sufficient.

It is known that during the sample preparation, loss of particles is possible.

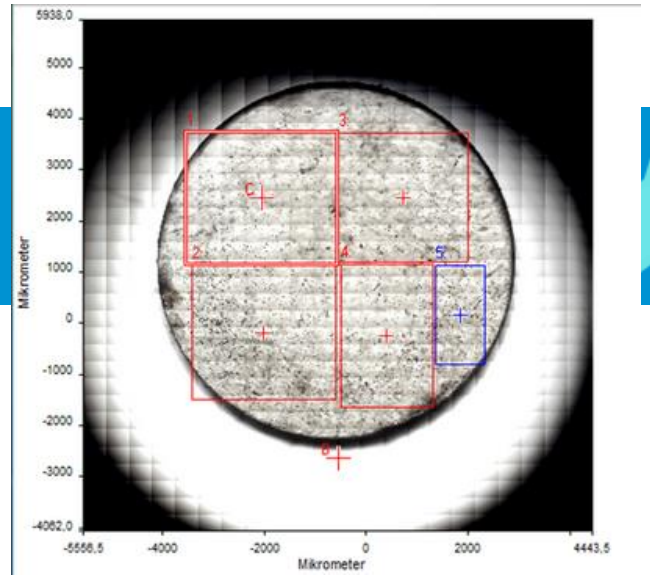
For this reason, we need not only reference particles, but reference materials with **exact particle number*, particle size* and polymer types.**

*in case of FTIR and Raman analysis

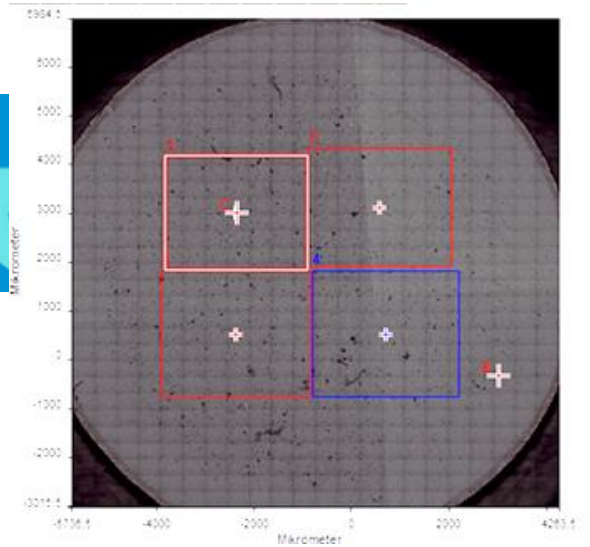
WP3- Analytical methods

Improving the µFTIR and µRaman methods

Development of the PYR-GC/MS method Validation



KBr Tablet



After filtration

First test: 1. Tablet measured; 2. Tablet dissolved, filtered and measured

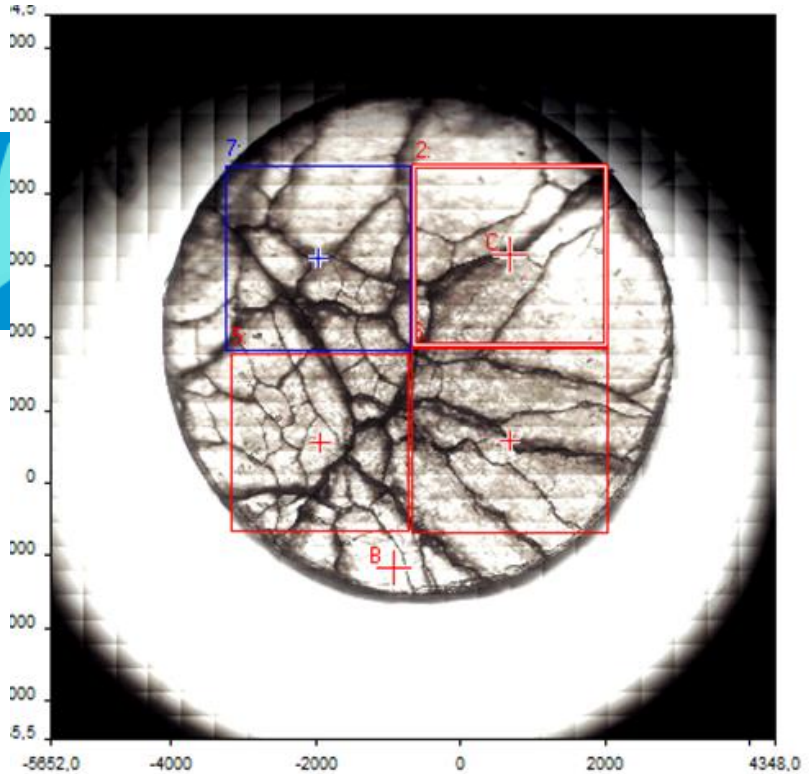
	Original particle number from manifacturer		Tablet		Filtrered
		100% recovery		71%	
PS	53	←	53	←	38
PET	83	←	84	←	53
		100% recovery		63%	

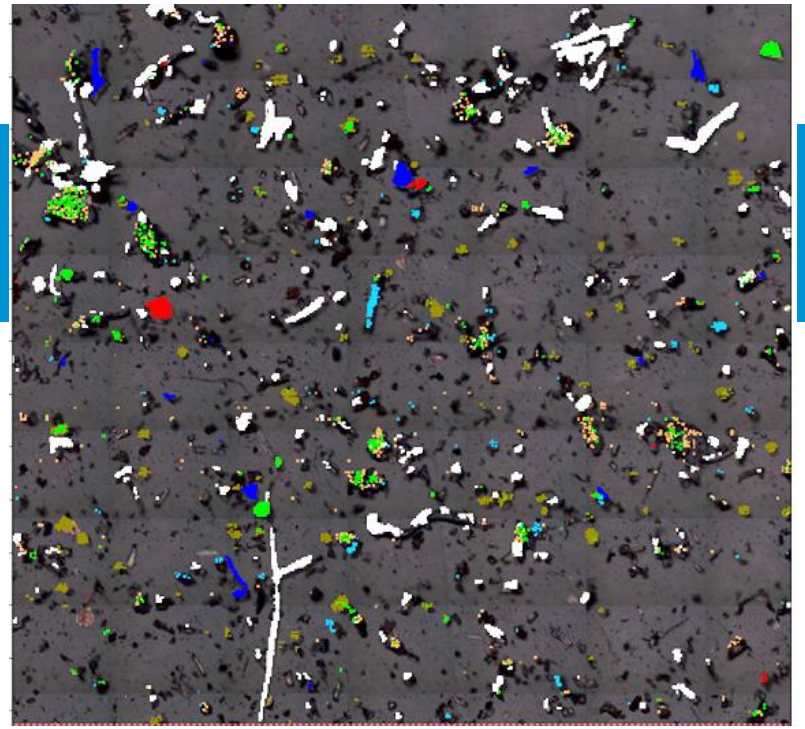
WP3- Analytical methods

Improving the μFTIR and μRaman methods

Development of the PYR-GC/MS method

Validation



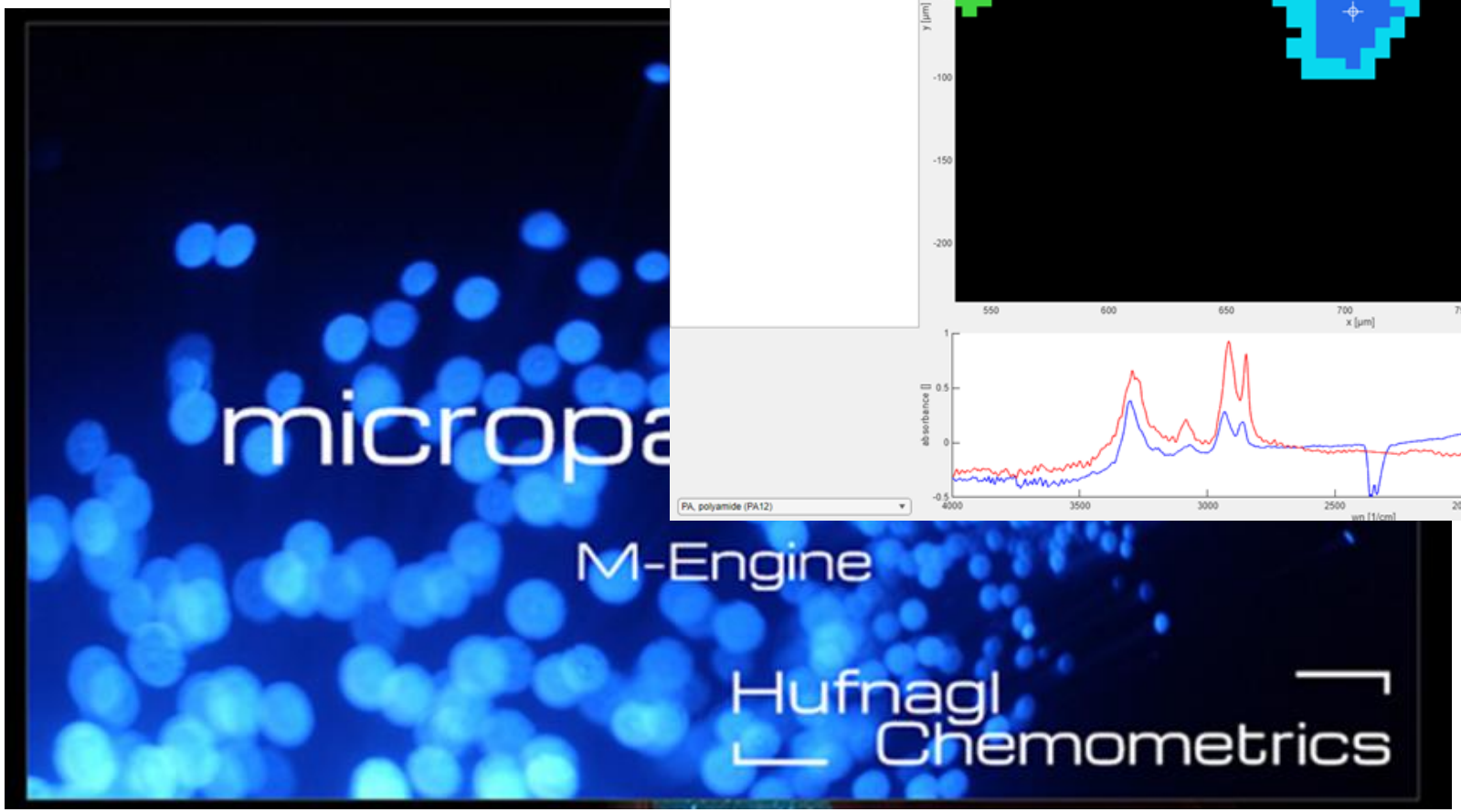


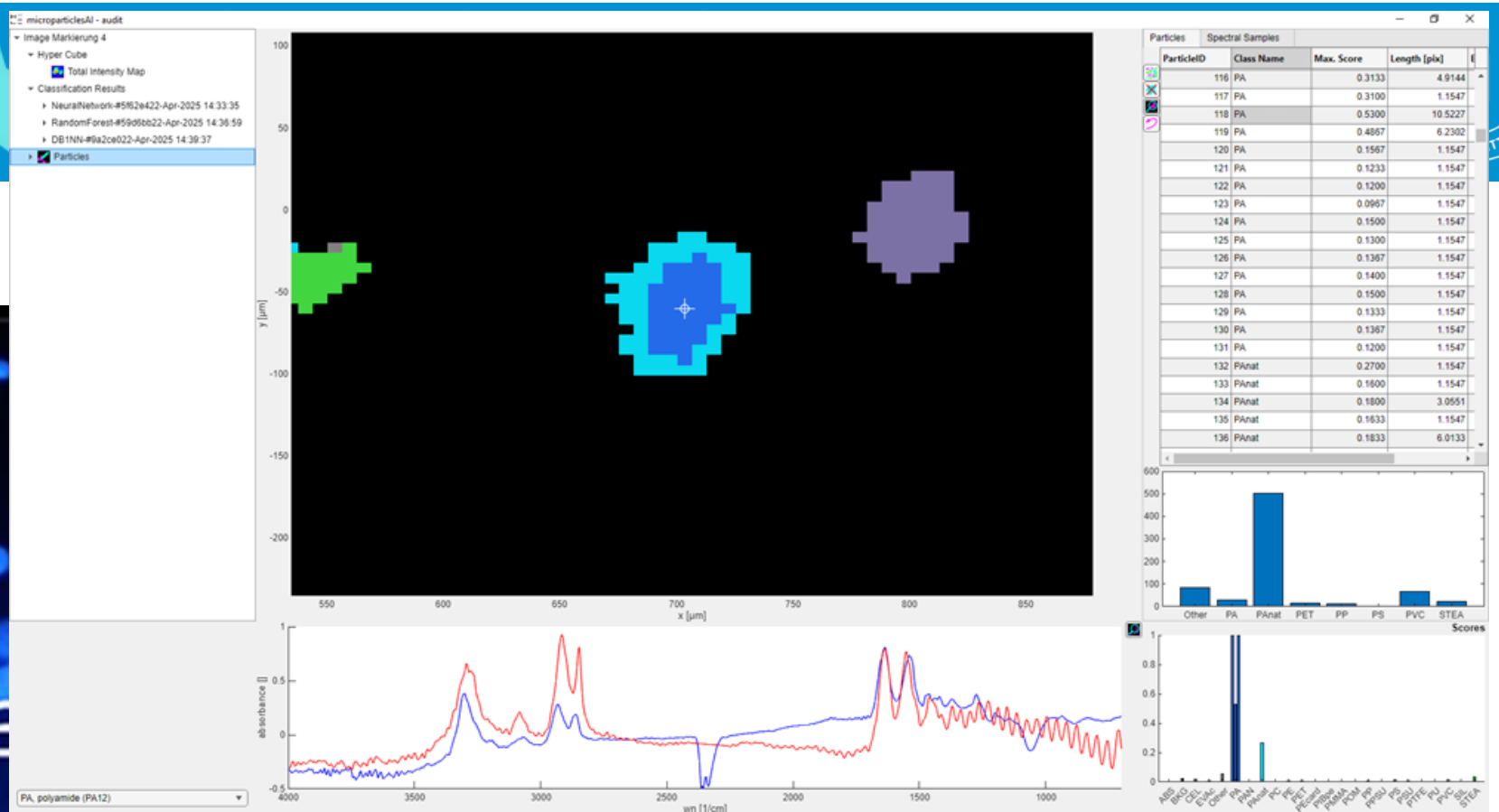
Test with matrix: 1. Tablet measured; 2. Tablet dissolved in matrix, filtered and measured

	Original particle number from manifacturer		Tablet		Filtered
PP	58	100%	58	74%	43
PE	44	61%	27	40%	11
PA	52	98%	51	33%	17

WP4- Data analysis, evaluation and visulisation

Training the machine learning program





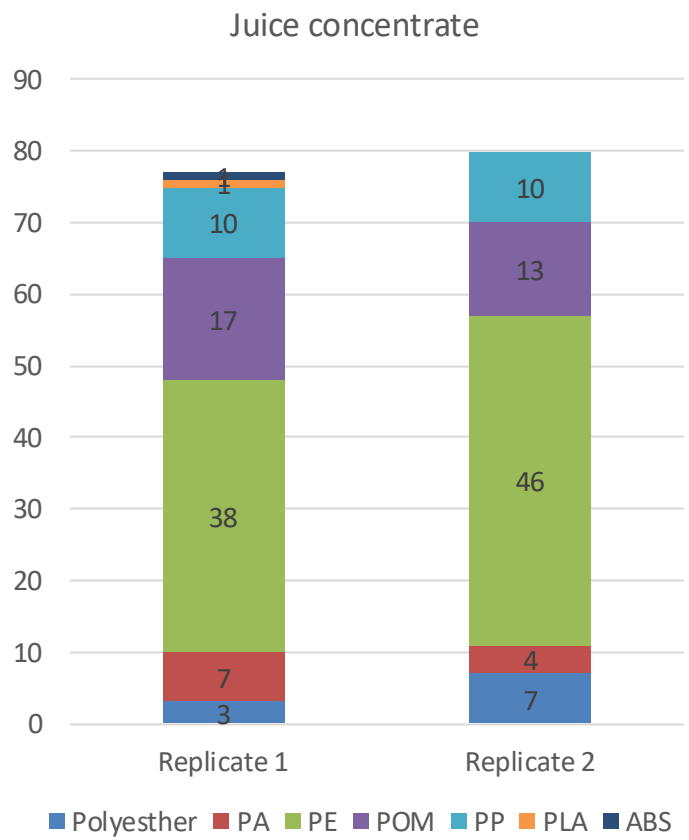
WP5- Analysis of real samples

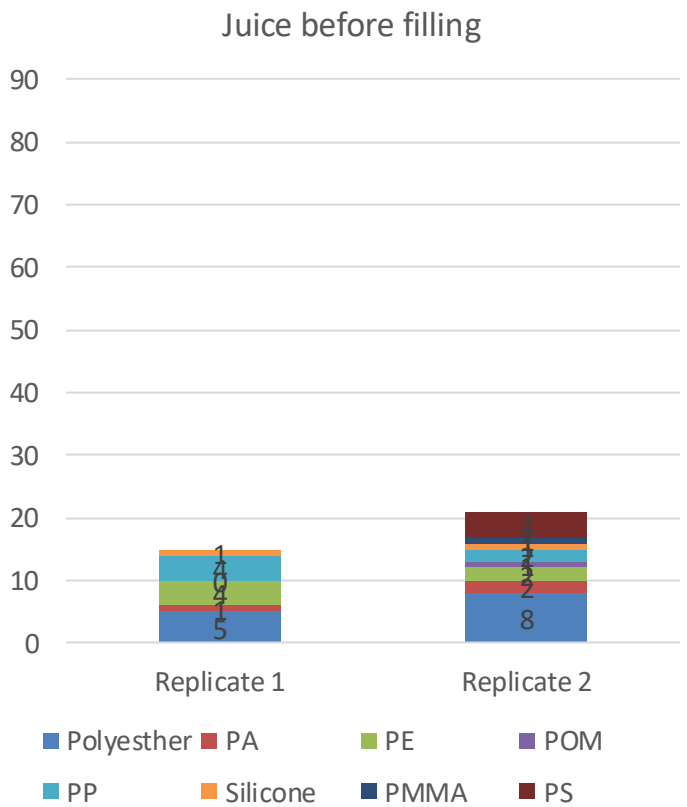
Measurement and evaluation of real samples

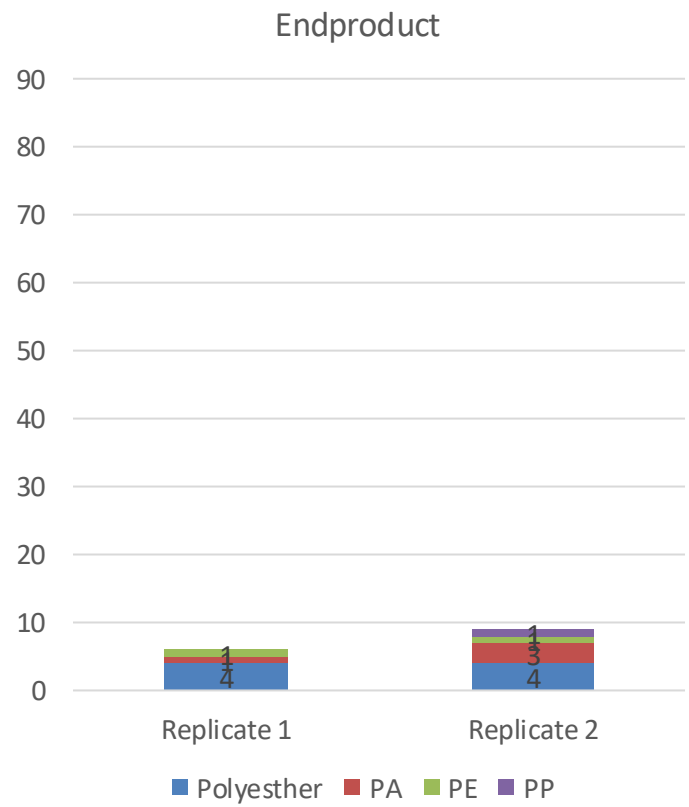
Mitigation strategy examples

Fruit juice

Packaging material
LDPE







MICROPLEXFOOD- Outlook

What is still left to do:

- Bread rolls
- Turbid beverages

Insights:

- We need proper reference materials
- Harmonization is needed in sample preparation and subsampling

CONTACT

BARBARA ZOTTL

barbara.zottl@ofi.at

+43 (1) 7981601633

Dr. Eva C. Vizsolyi

eva.vizsolyi@ofi.at

+43 (1) 7981601589

OFI

1030 Wien, Franz-Grill-Straße 5, Objekt 213

office@ofi.at | www.ofi.at