Exposure assessment of microplastic in the **food and packaging** industry and an outlook on **paints and lacquers**

Outline

Exposure assessment in the **food and packaging industry**

Project **‘microplastic@food’**

Key facts

Results: SOPs (+Round Robin Test) – Mineral waters – Process line testing

Follow-up project **‘MICROPLEXFOOD’**

Key facts and planned case studies - Goals

Exposure assessment in the **paint and lacquers industry**

Project **‘MicroPaint’**

OFI – Packaging, Recycling and Dangerous Goods

Testing, Assessment, Development and Consulting in the packaging sector

- ✓ Longstanding packaging expertise (30 years of expertise in the packaging sector), comprehensive laboratory infrastructure, interdisciplinary team
- ✓ Packaging assessment, training courses and damage expert assessment
- ✓ Food law expert assessment (special area of expertise: NIAS analysis), risk assessment and product protection
- ✓ Specialisation on recycling and sustainability
- ✓ Transparent, quantitative assessment of the technical recyclability according to cyclos HTP (CHI)
- ✓ Customer specific development of individual and innovative packaging designs
- ✓ R&D expertise – project work and lead in (inter)national) industrial projects

Projects at OFI

Safety and Quality

- Xenohormone
- Migratox
- PolyCycle
- SafeCycle
- **Microplastic@food**

Recycling, Circularity and Sustainability

- Flex4Loop
- Pack2the Loop
- Reflex

Packaging development

- InCanPres
- Active and intelligent packaging
- Packloop

Occurrence in Food (According to Literature)

- Microplastic particles have been found in different types of food, but its **sources are mostly unknown**
- Occurrence found in ….

  …different salt samples…

  …in honey and sugar….

  …mineral water and beer…

BUT:
…studies in food are extremely difficult and prone to errors due to the complex matrix and other contamination,…!
…occurrence before, during and after processing has to be thoroughly investigated!

Figure: WHO report 2022

Project ‘microplastic@food’

Research partners

ofi – Fortschritt in guten Händen
Universität Bayreuth
ipf – Leibniz-Institut für Polymerforschung Dresden

Funding

cornet – Collective Research Networking
FFG – Forschung wirkt.
Bundesministerium für Wirtschaft und Klimaschutz

microplastic@food

Goal: Development of a method to evaluate the presence of microplastic particles in food

Project start: July 2021
Project end: October 2023

Associations

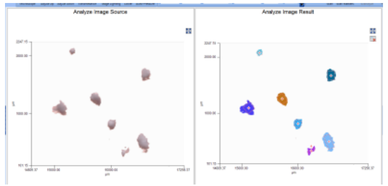
eco plus – lebensmittel cluster niederösterreich
IVLV – Industrievereinigung für Lebensmitteltechnologie und Verpackung e.V.

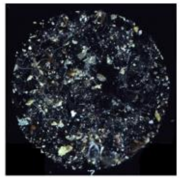
Industry partners

Food industry (Beverages, public water suppliers, etc.)
Packaging industry
Production plants
Analytics
Associations

Project Goals ‘microplastic@food’

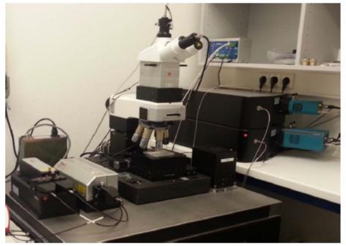
Development on an **analytical method** for the detection and identification of microplastic in the food and packaging industry





Assessment of validated and reliable data in four case studies:

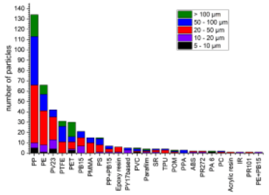
- Packaging and filling plants
- Water and filtered beverages
- Food surfaces
- Soluble foods



Raman: alpha 300R+ / WITec

Evaluation of **sources of origin** and definition of **counter measures** and strategies to prevent the presence of microplastic in food

FTIR: spotlight 400/PERKIN-ELMER



J.Brandt, L.Bittrich, F.Fischer, E. Kanaki, A.Tagg, R.Lenz, M. Labrenz, E.Brandes, D.Fischer & K. DOI: 10.1177/0003702820932926.

Goals and Deliverables

Development of SOPs for microplastic analysis for the food and beverage sector

Development of a validated method for analysis with computer-aided evaluation of the samples (qualitative and quantitative) **= automatization**

Analysis of final products and filling plants to gather information on **causes for contamination**

Development of a catalogue of '**counter-measures** for microplastic in food, beverage and packaging'

Work Plan

AP2

Development of a standardized method for the **sample preparation** and concentration of microplastic particles

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AP3

Comparison and optimization of methods for the measurement of microplastic particles

→ Analysis of **reference samples**

ipf ofi

AP4

Automatization with computer-aided analysis

ipf Purency

AP5

Analysis of **packaging materials and filling plants** to determine their contamination with microplastic particles and assess potential sources of origin

ofi

AP6

Application of the developed method in defined food **product categories**

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AP7

Preparation of a guidance document with recommendation for standardized methods and counter measures to prevent microplastic contamination in food and packaging

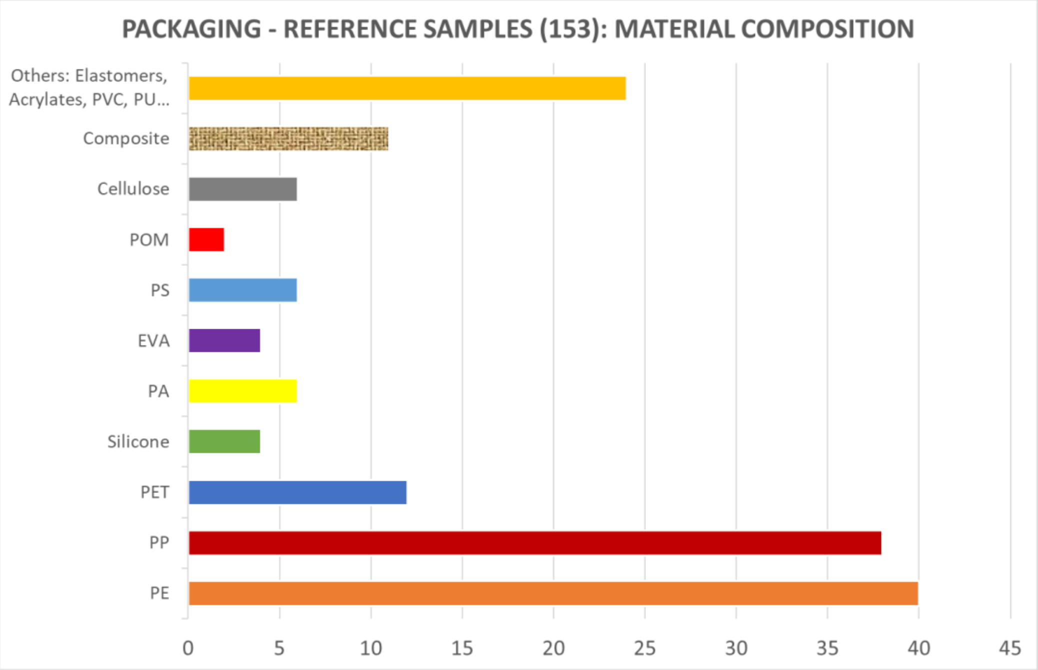
Setting Up a Database

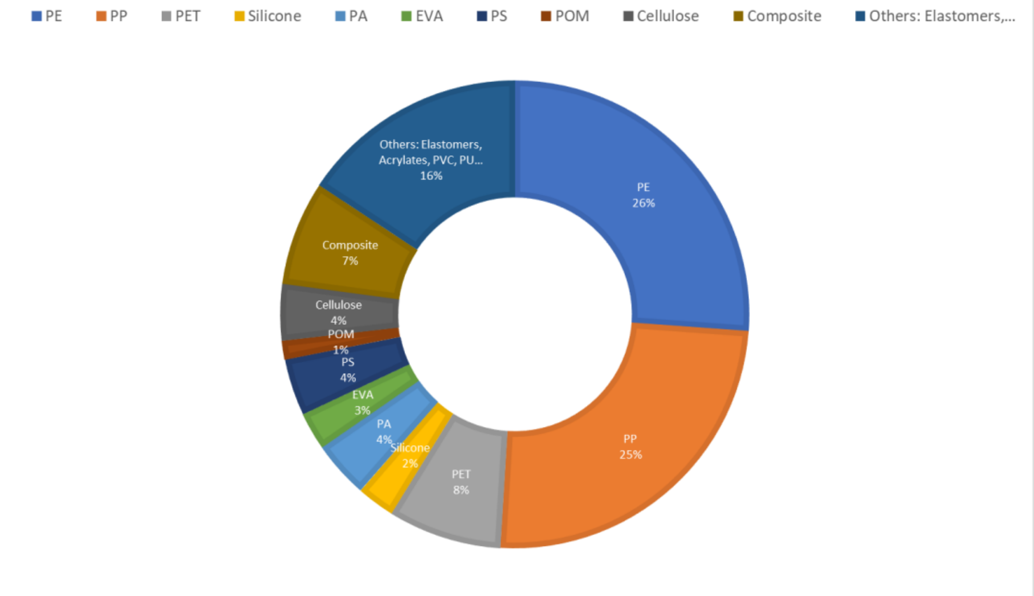
More than >300 different colored and printed packaging materials were provided by the industry partners

More than > 153 reference samples spectrums for the 16 selected polymers were cumulated

Result: standardized protocol for sample collection and preparation.

→Interlaboratory comparison

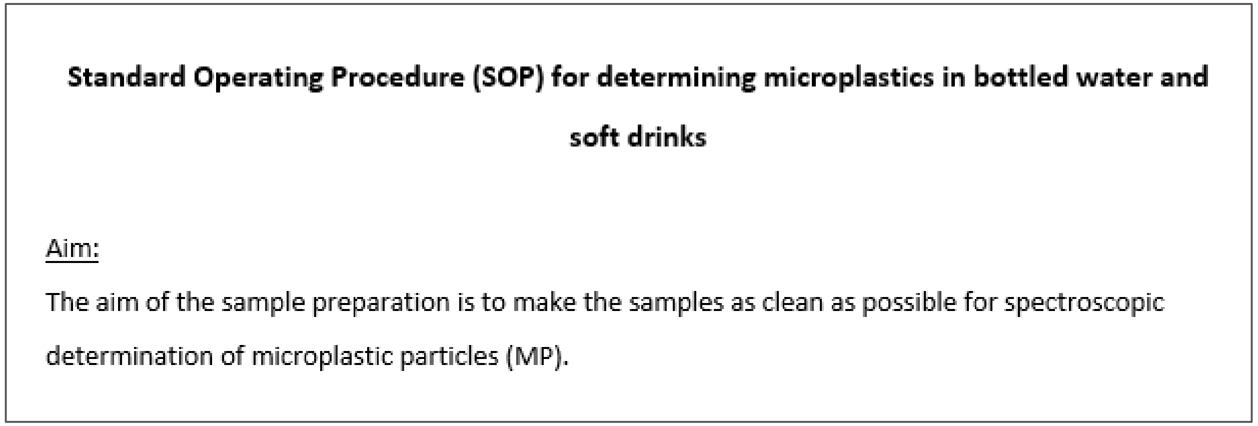




Standard Operating Procedure (SOP)

– Preparation steps
- Gentle cleaning of outside packaging to avoid inside contamination
- Homogenisation of the sample through gentle shaking
- Opening of bottle and removal of closure
- Clear liquids (such as water) are directly filtered
- Total volume of a packaging unit has to be filtered!!!

Figure: Steel filter



Standard Operating Procedure (SOP) for determining microplastics in bottled water and soft drinks

Aim:

The aim of the sample preparation is to make the samples as clean as possible for spectroscopic determination of microplastic particles (MP).

Sample Preparation

Samples:
- Packaging rinsed with ultrapure water
  - Bottles, cups, containers, trays (inner surface = contact to foodstuff)
  - Screw caps, preforms (total surface)
- Analysis of liquids: (mineral)waters or clear liquids from glass bottles, plastic bottles or beverage carton


Protocol:

– Shaking of bottle/container and filtration of total content

– Filtration of the liquid samples or the rinsed media on Si- or Ano-Disk filter

– Reflushing of the equipment with ultrapure water



Spectroscopy/Analysis

IR-Transmission-Imaging and/or particle-based Raman measurements

- 3 Filter / Sample types: first duplicates -> triplicates for uncertainties
- For each sample series a blank (=ultrapure water) is analysed

Spectroscopic Analysis:

- FTIR – Microscopy/Spectroscopy (Imaging) ; ~22-28% analysed
  -> extrapolated to 100%
- Raman-Microscopy/Spectroscopy (particle based measurement); 100% analysed

Data evaluation (automatisation; with visualisation):

- Purency „Microplastics Finder“ (Machine Learning Software)
- Software GEPARD (Gepard-Enabled PARticle Detection): open source

For each IR-image (~2 x 2 mm) ~100.000 individual specta are recorded

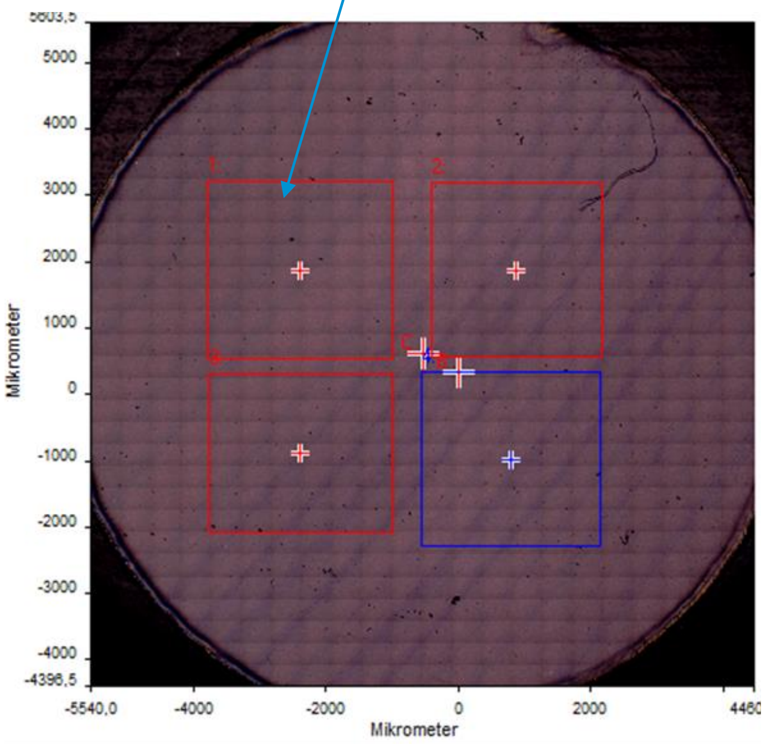
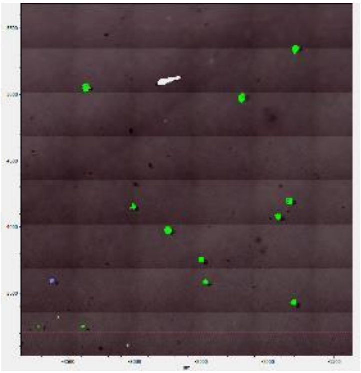
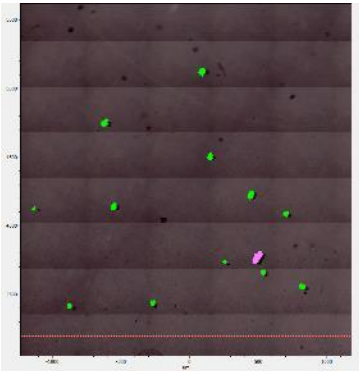
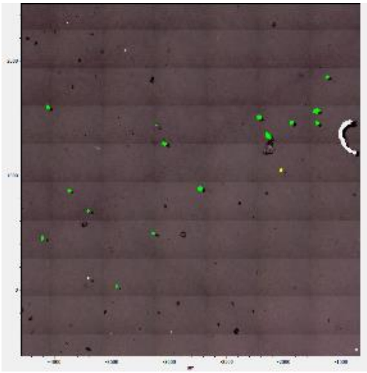
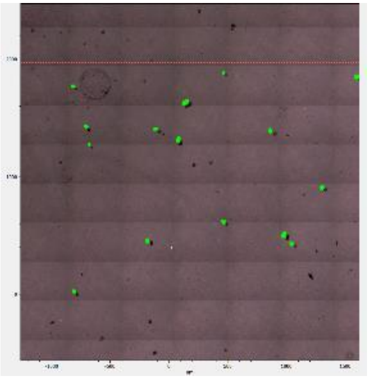


Figure: Filter loaded with sample with 4 measurement areas (25-30% of the surface) for FTIR –Imaging (Transmission)

Interlaboratory Trial

Aim:
Development of a validated method for the quantification of microplastic (for food packaging)
Verification and assurance of the intercomparability

different analytical methods
(µRaman, µFTIR)
different analysis software
(Gepard/ParticleScout; Purency/Microplastics Finder)

Assessment of the **analytical fluctuation** margin to develop a basis for real food-microplastic measurement

Methodology comparison of spectrometric methods and internal ‚ring trial':

OFI: FTIR Imaging / Perkin Elmer FTIR-microscope / transmission

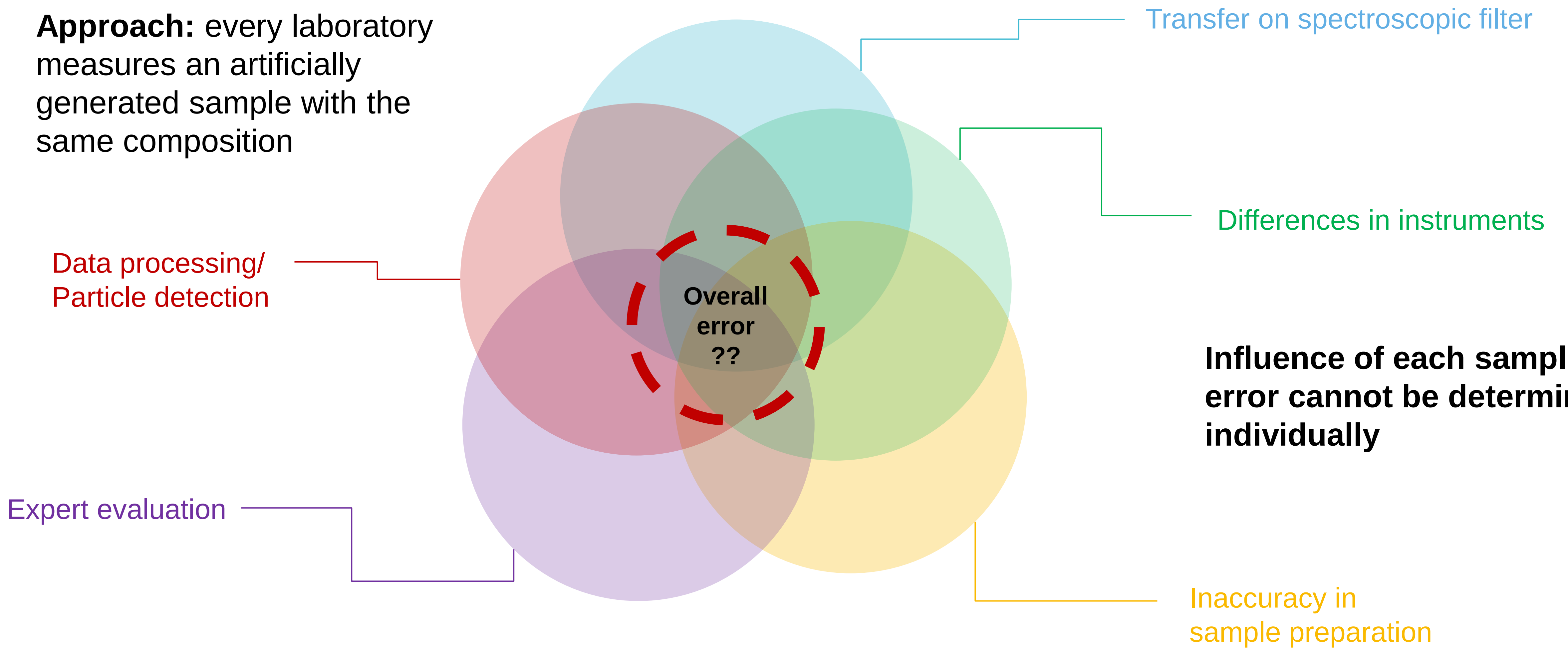
IPF: particle-based Raman-measurement / Witec

IPF: particle-based FTIR-measurement imaging / Perkin Elmer

UBT: FTIR Imaging / Bruker FTIR microscope

Interlaboratory Calibration

Approach: every laboratory measures an artificially generated sample with the same composition

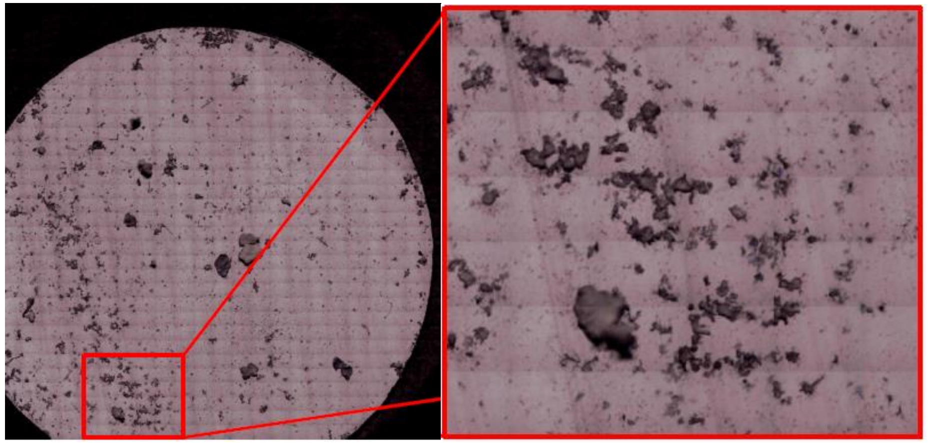


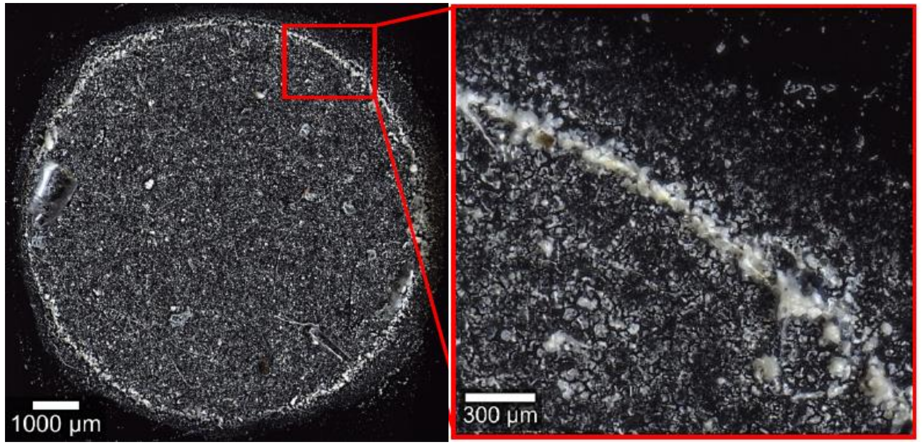
Influence of each sample error cannot be determined individually

Traditional Ring Trial

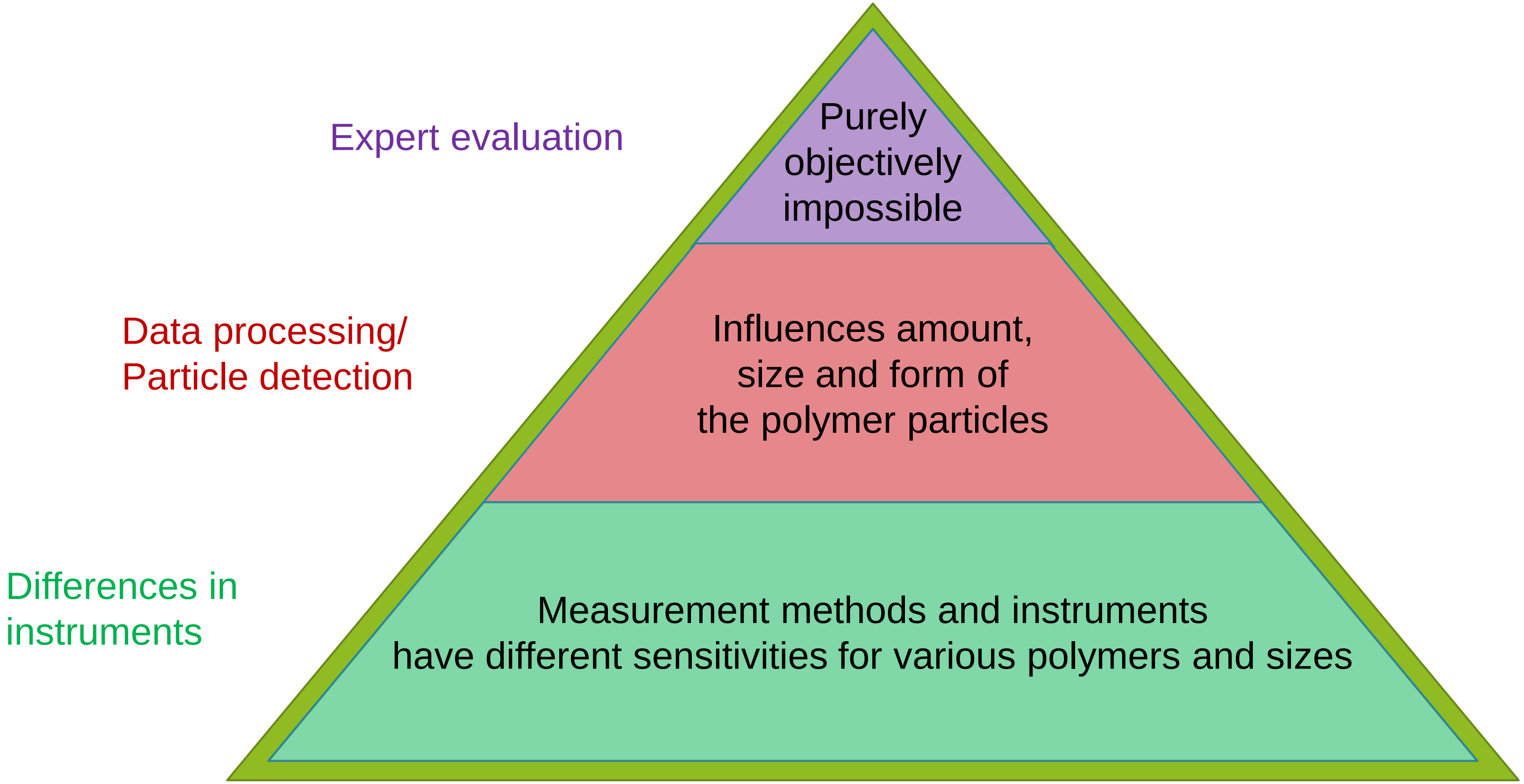
1. Separation in solid phase and send as suspension
   Problem: particle homo-aggregation

2. Addition of 0,005% Triton for transportation in frozen state
   Problem: Crystallistion and particle hetero-agglomeration



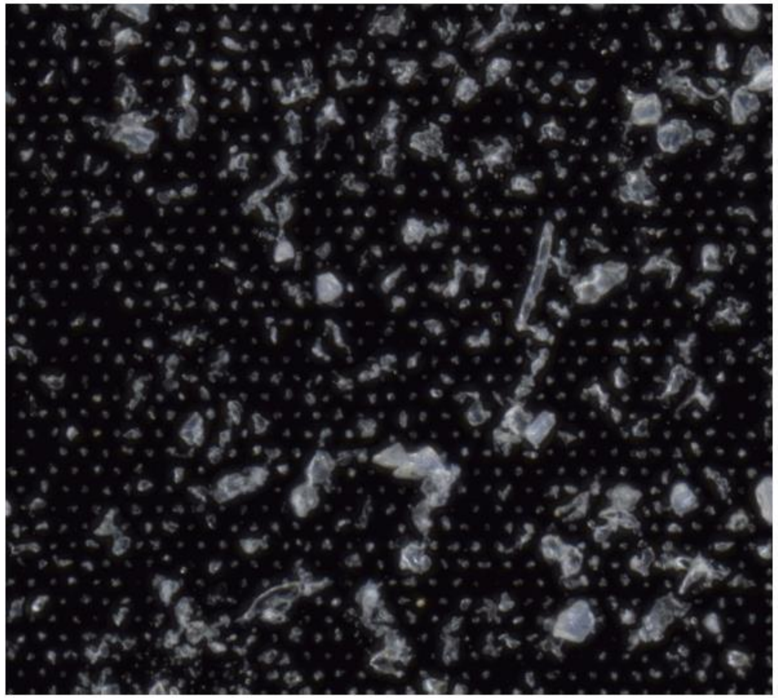


Error Effects?

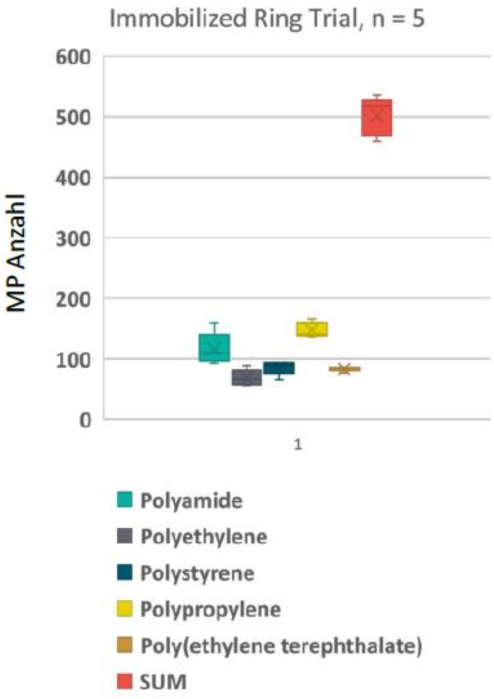


Minimise these effects through
particle immobilisation!

Immobilised Ring Trial



Bilder: IPF, UBT, OFI



###	Class	No. of Particles
☑ 1	PP	166 particles
☑ 2	PE	93 particles
☑ 3	PVC	0 particles
☑ 4	PU	0 particles
☑ 5	PET	90 particles
☑ 6	PS	92 particles
☑ 7	ABS	0 particles
☑ 8	PA	129 particles
☑ 9	PC	0 particles
☑ 10	PMMA	0 particles
☑ 11	PAN	0 particles
☑ 12	SIL	0 particles
☑ 13	CEL	0 particles
☑ 14	PA_nat	0 particles
☑ 15	PIB_PE	0 particles
☑ 16	PE_CARD	0 particles
☑ 17	Other	133 particles
☐ 18	BKG	15 particles

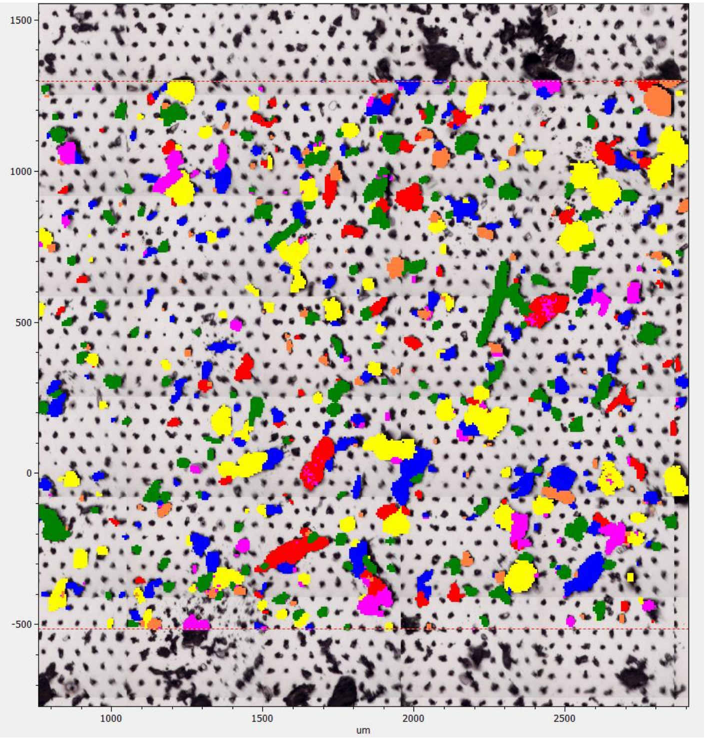


Figure: light microscopic image of a filter segment. IR image overlapped with the results from the machine learning program "Microplastics Finder"/Purency

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!

Case Study: Water and Filtered Beverages

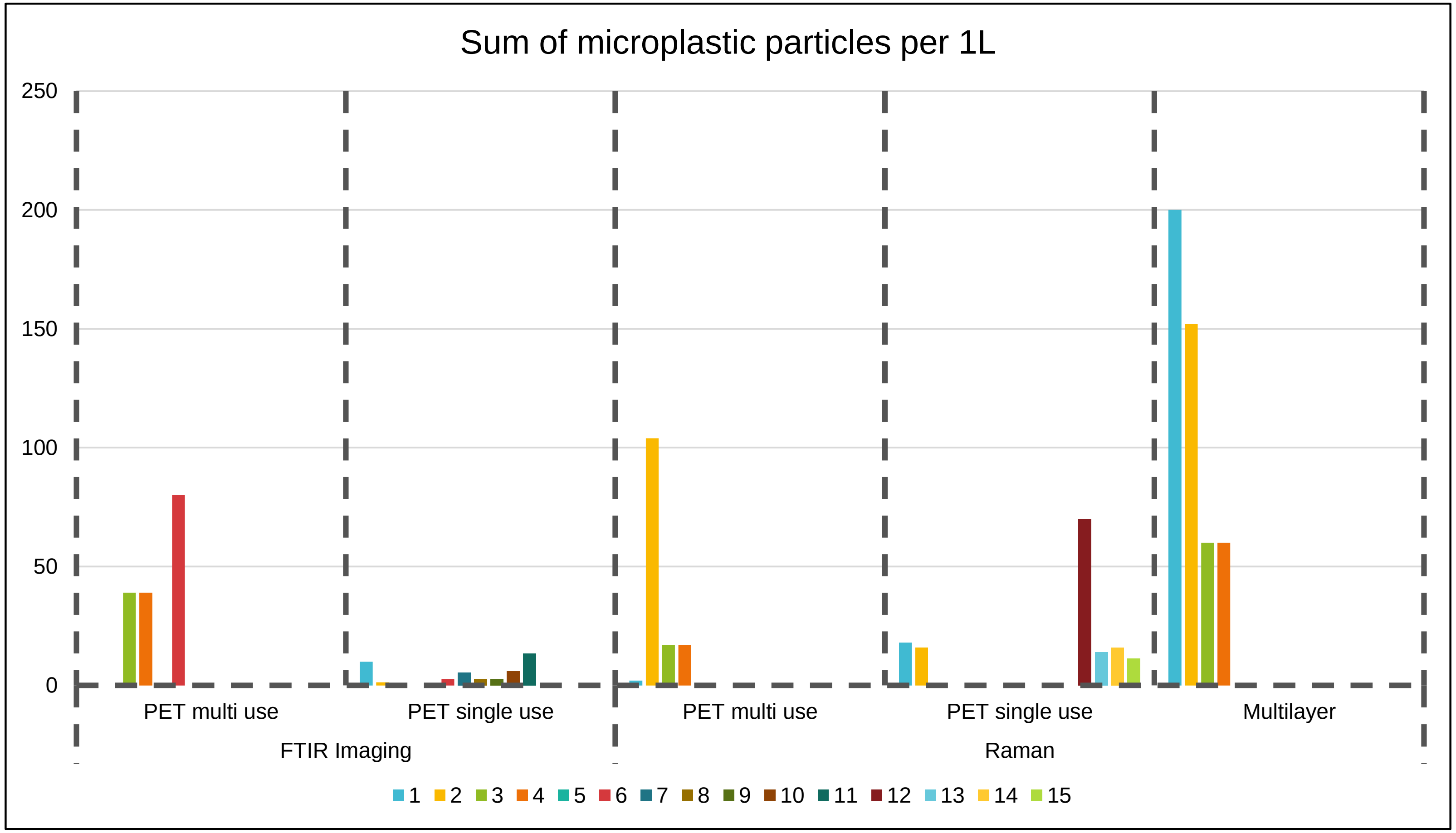
Samples

- Packaging (bottles, closures, preforms)
- Mineral water (glass, plastic, single use, multiple use, multilayer)
- Fruit juices
- Beer and wine
- Water from filling plants and wells
- Blanks from the laboratory
- Blanks from the customers/filling plants

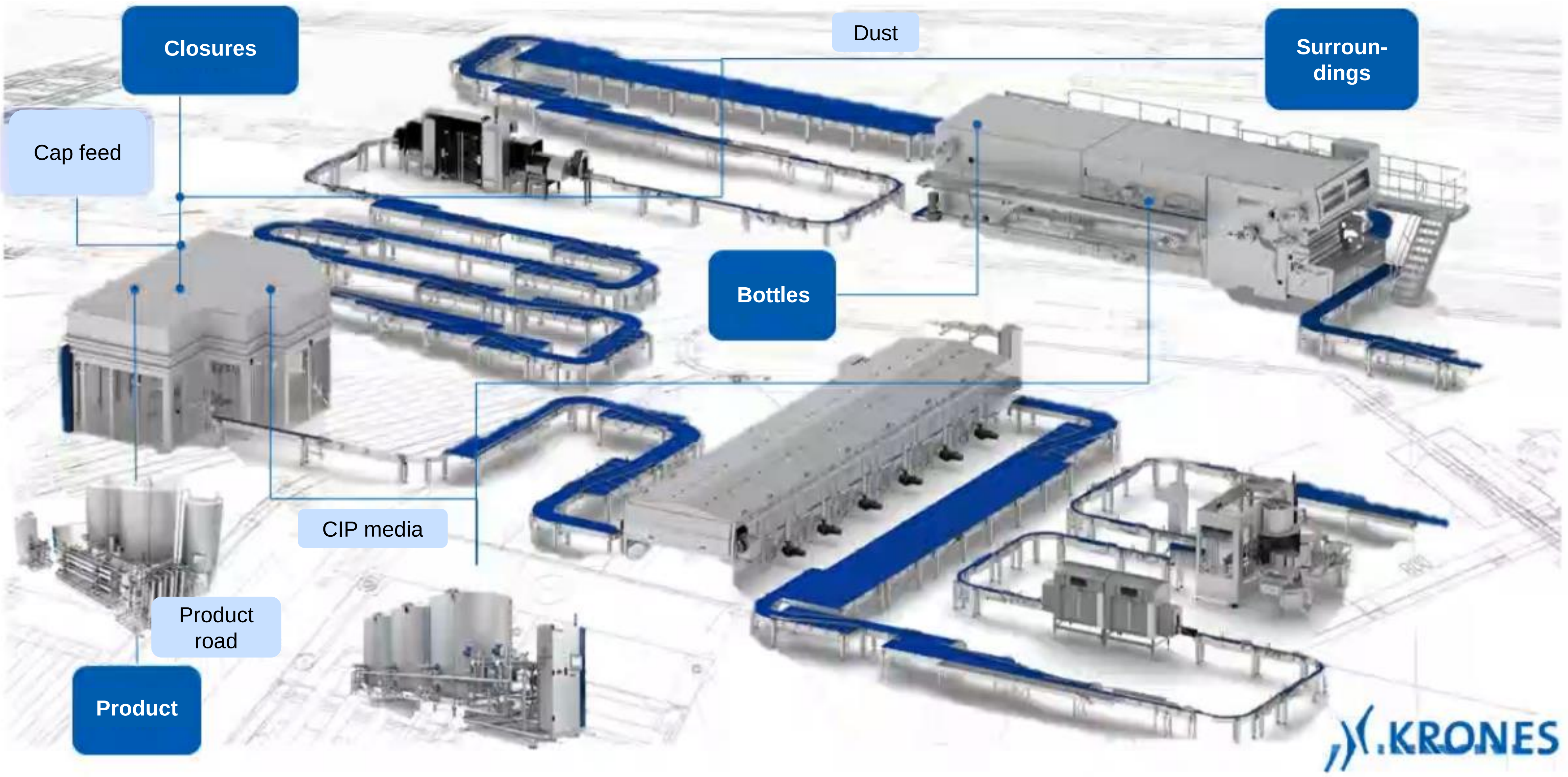
Triplicate assessment of each sample!

Case Study: Mineral Waters

- Microplastic particles were found in nearly every sample in different concentrations
- Multi use bottles generally lead to higher results than single use bottles
- Multi use glass bottles lead to high results in comparison to PET bottles
- Raman leads to higher results than FTIR



Possible sources for microplastic – e.g. multi-use glass



Other Beverages

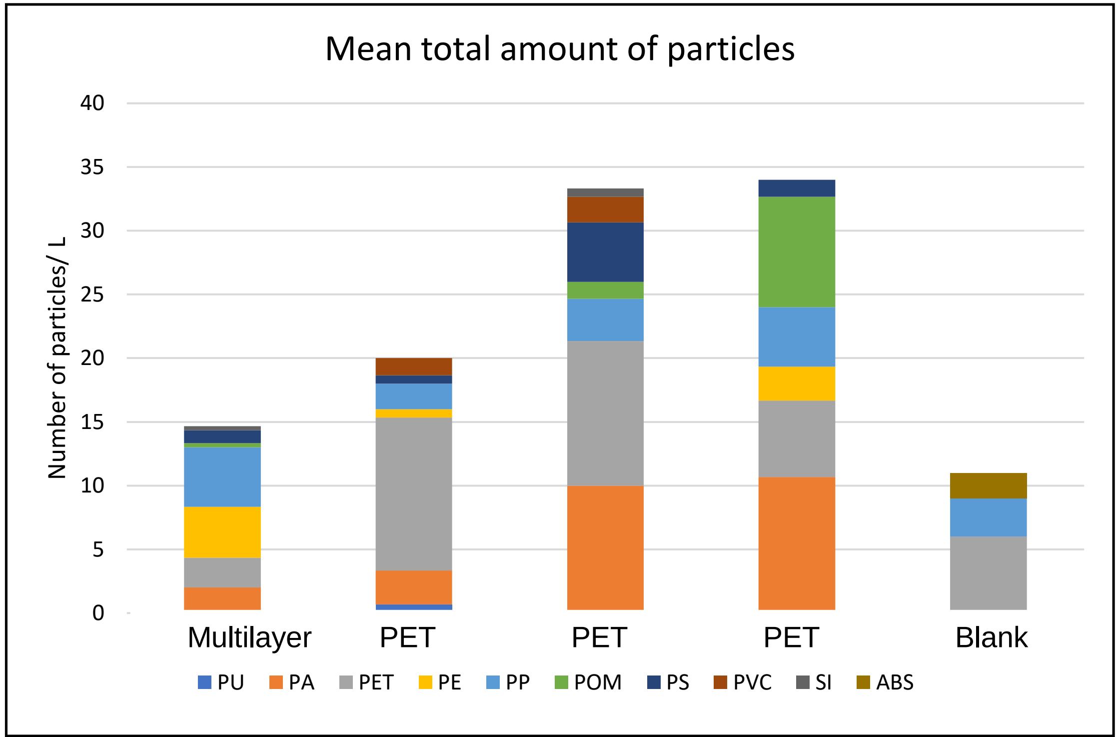
4 different beverages measured in triplicates, no background measurements at the filling plant

Sample preparation:

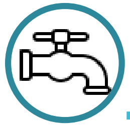
- Pre-filtration
- Oxidation step

Result:

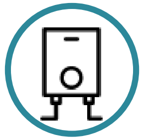
- CO_2 content has an effect on the results
- Average of 15-20 particles/bottle
- Packaging not the main source for the particles



Microplastic Evaluation Along the Process

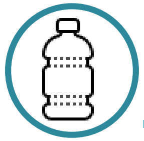


Water supply 1,200 ml



Water supply after processing (before the filling machine) 1,200 ml

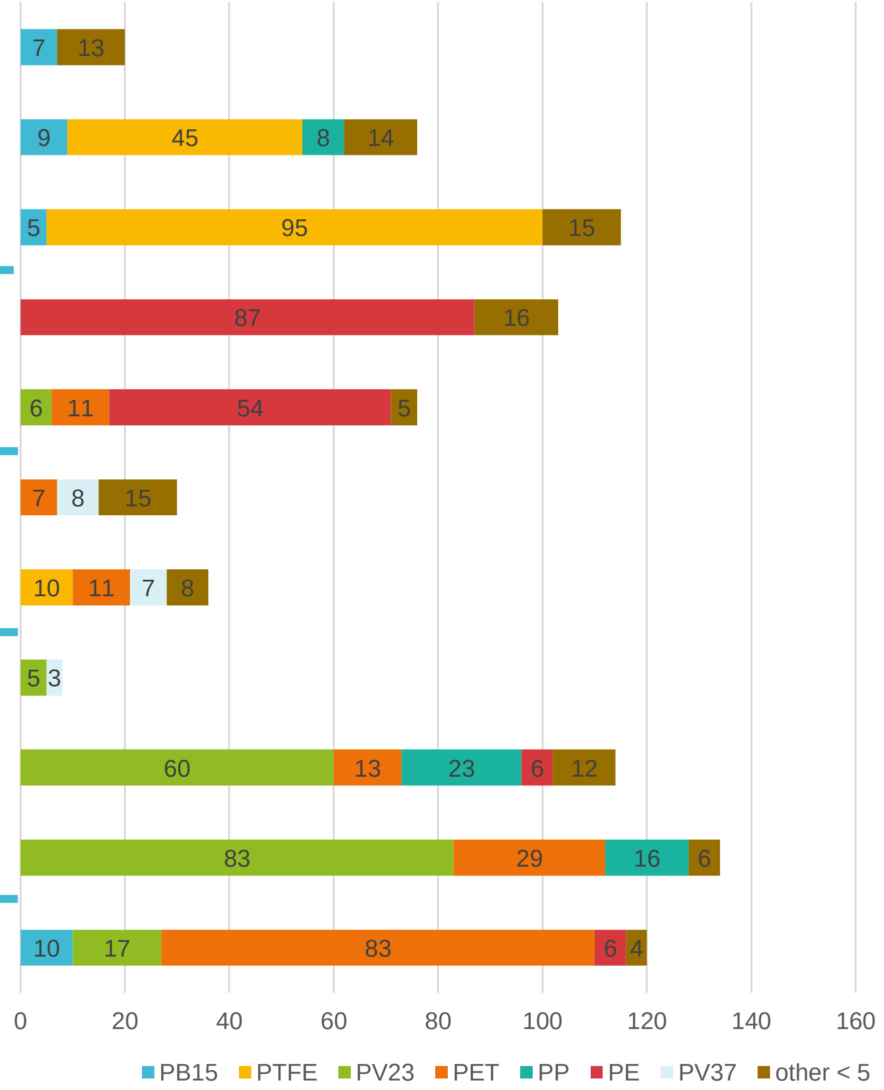
Commercial drinking water multilayer 500 ml



Commercial drinking water single use PET 500 ml

Commercial drinking water multi use PET 500 ml

Surrounding air company (1h, Ø11 cm)



Microplastic Evaluation Along the Process

Water supply 1,200 ml

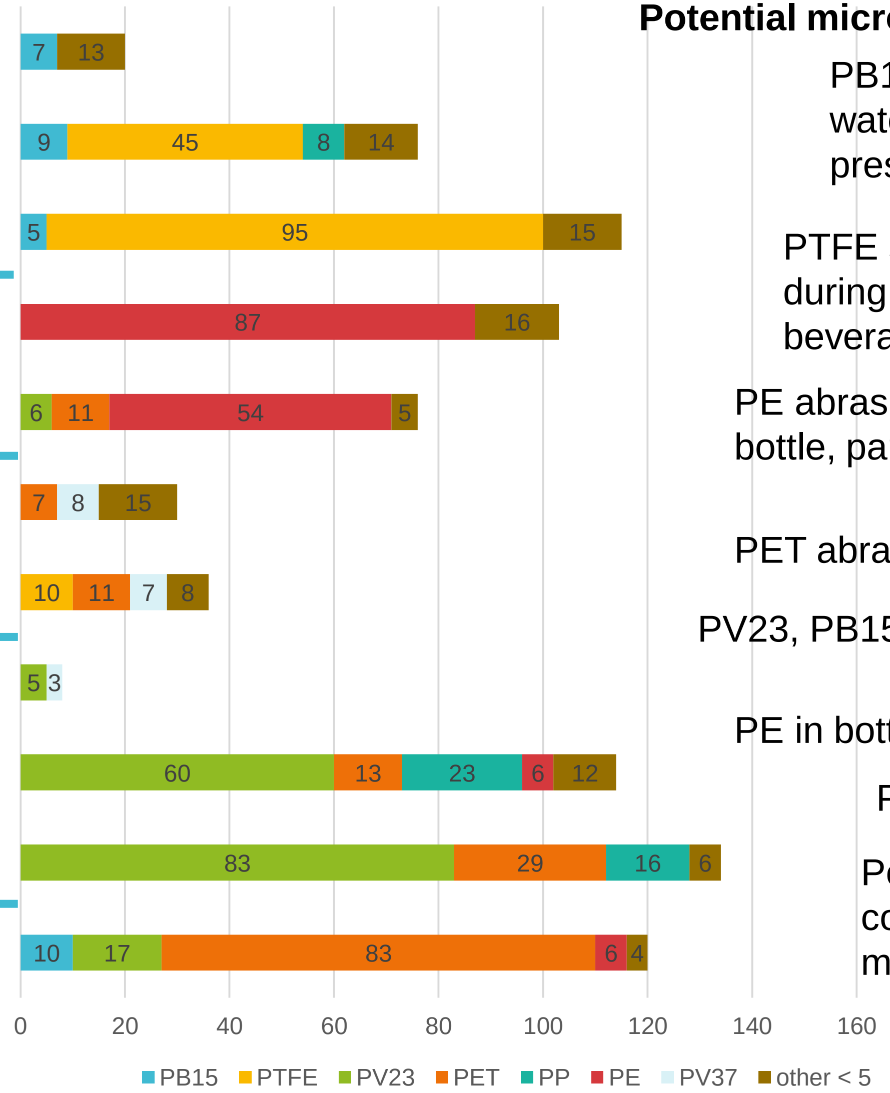
Water supply after processing (before the filling machine) 1,200 ml

Commercial drinking water multilayer 500 ml
Cap: PE+TiO_2
Bottle outside: PE+$CaCO_3$
Inside: PE

Commercial drinking water single use PET 500 ml
Bottle: PET
Label film: PP + PB15 + PV23
Cap: PE + PB15

Commercial drinking water multi use PET 500 ml
Bottle: PET
Label film: PE + PB15 + PV23
Cap: PP + PB15
Sealing: EVA

Surrounding air company (1h, Ø11 cm)



Potential microplastic sources

PB15 is often part of PE water pipes and is still present after processing

PTFE sealing and coatings during processing, but in beverages below detection limit

PE abrasion from inside of the bottle, partly from the cap

PET abrasion from bottles

PV23, PB15 part of label printing

PE in bottles from label film

PP from cap

Possible cross-contamination through microplastic in surrounding air

Conclusion Process

- Initial product shows low microplastic contamination
- PTFE in final product not measurable -> perhaps from tap sealings in sample collection?
- All packaging systems show the presence of microplastic through the packaging (e.g. labels, closures)
- More complex PP closures have a higher microplastic potential (greater surface)
- Potential for microplastic reduction through optimisation in accordance with the production process
- Air samples show high potential for optimisation concerning microplastic
  → A lot of PET found, which was processed at the same time

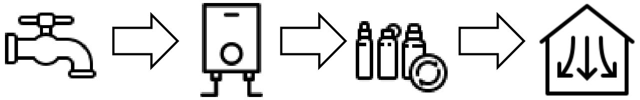
Coming up…

Analysis of rinseable products

e.g. cheese, tofu,…

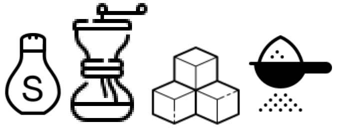
Process analysis

In-depth assessment of filling plant (e.g. for mineral water)



Analysis of salt and sugar products

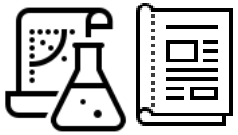
e.g. salt or sugar grinders,…



Publications

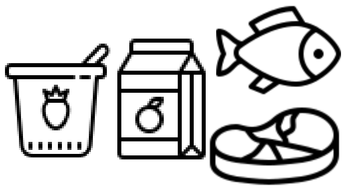
On methodology, intercalibration, results for case study mineral water,…

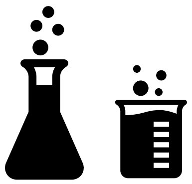
Peer-reviewed and industrial journals

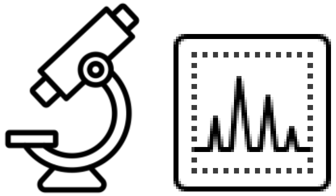


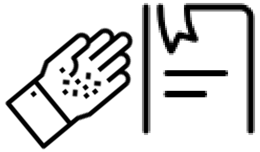
Follow-up ‘MICROPLEXFOOD’

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









Complex foods
(turbid beverages, fruit juices, dairy products, fish and processed meat products)

Sample preparation
(Enzymatic or chemical digestion)

Sample measurement /analysis
(µFTIR, µRaman, Pyrolysis-GC/MS)

Formulation of countermeasures
(Guidance document)

Project 'MICROPLEXFOOD'

Research partners

Funding

Follow-up project:

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

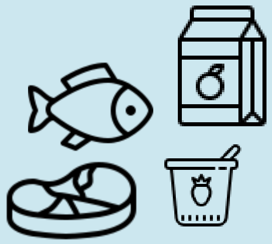
Project start: Fall 2023
Duration: 2 years

Background

- During the project ‘microplastic@food’ methods have been developed and knowledge was gained on several product groups.

- **BUT:** to set up the method rather „simple“ foods, such as mineral water, solvable foods and food surfaces with rinse-off methods were considered

How can this methodology be used for food, which are **more complex** (= and must be chemically or enzymatically digested), or contain **particles** that might **interfere with the measurement** (= food particles are wrongly identified as microplastics)?



Goals and Deliverables

Precise analysis of complex food matrices on the presence of plastic particles through the use of **enzymatic (and if necessary chemical) digestion** methods within the defined use cases.

Further **development of the spectroscopic methods** to improve the distinction of the microplastic particles from the sample matrices.

Precise distinction of detected particles in the **case studies**.

Definition of the specific sources through the analysis of the products, the process environment and the packaging.

Work Plan

AP2

Sample preparation of complex food (enzymatic and chemical methods)

LVA

AP3

Analytical methods – further development for complex food

ipf

AP4

Data analysis and visualisation

ofi

Purency

AP5

Microplastic analysis of real samples including source analysis

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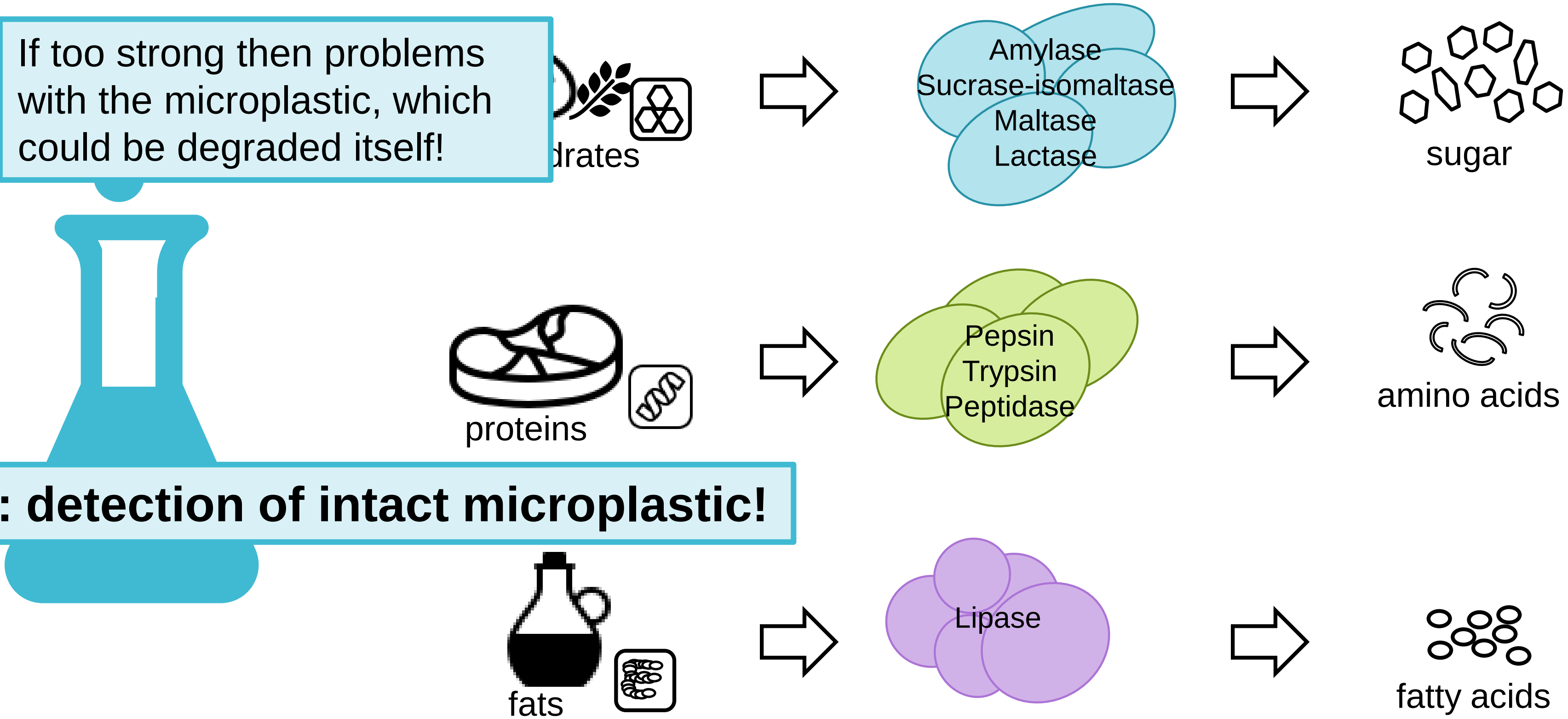
Enzymatic Degradation – Dissolution of the Food Matrix

– E.g.: with
  - acids
  - alkalis
  - oxidizing agents
  - heat
  - ultrasound (supporting)
  - enzymes

If too strong then problems with the microplastic, which could be degraded itself!



Our goal: detection of intact microplastic!

Case Studies

Case I: turbid beverages and fruit juice

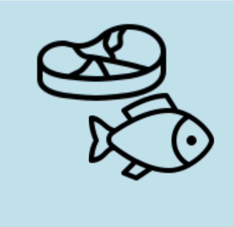
= food with pectin, cellulose and starch

Contains floating particles, which hinder the analysis and possible sources within the process

Case II: complex dairy products

= food with high water, protein and sugar content

Influence of particles in products, which might hinder the analysis (= distinction of e.g. fruit yogurt particles and microplastics) and sources within the process



Case III: fish and processed meat products

= food with high protein and/or fat content

Potential sources:

processing (e.g. machinery, processing aids, etc.) and water, in which the fish is present

Further:

References and samples for comparison (such as packaging) and water and clear beverages

Project ‚MicroPaint‘

Research partners

OFI · FELMI-ZFE

Planned Funding

FFG – Forschung wirkt.

MicroPaint

Austrian industry project to assess the sources and levels of contamination with microplastics in the lacquer and paint industry

Project start: Q1 2024
Duration: 3 years

Industry partners

Paint industry
Lacquer industry
Analytical companies

Microplastic from Paint and Lacquers

- About 75,000 to 300,000 t of microplastic are released in the environment in the EU each year (EU 2018)
- One of the identified main causes are abrasion from paint and lacquer system during their use period
- Estimation (EUNOMIA 2018 and EU study 2022): 482,000 t/year from building paint, street paint, marine paints and lacquers are released as microplastic in the environment

Releases in the environment in the EU

(updated figures from 'draft IA study')

Source	Quantity (tons/year)
Paints	482 000
Tyres	450 000
Pellets	134,260 – 198,190
Textiles	1,649 – 61,078
Geotextiles	6,000-19,750
Detergent capsules	18 009
For reference, in the marine environment (IA SUPD, 2018)	
Microplastics	*75 000 – 300 000*

Pew & Systemiq, Breaking the Plastic Wave (2020)

Globally terrestrial and ocean leakage, and still rising:

All plastics:
42 million ton, 2016

Microplastics:
4,4 million ton, 2016

Four sources of microplastics considered: tyre abrasion, production pellets, textiles, personal care products

European Commission

→ Currently the amounts are mainly based on mathematical models

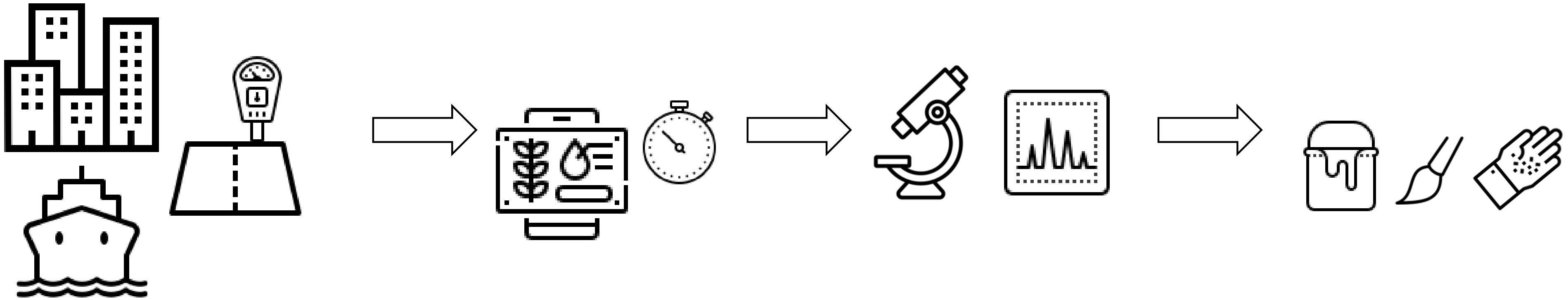
Motivation

- The paint and lacquer industry are in a tight spot and have to gather information on the causes for their microplastic release and formulate counter-measures to eliminate/reduce the contamination

- Qualitative and quantitative measurements are essential to gather information on the current amount that is being released

Goal: ensure the contamination of the environment through microplastic release from lacquers and paints is minimized

Project outline



Building paint, marine paint and street paint

Simulation and aging of paints and lacquers

Analysis of microplastic in the laboratory and in the environment

Measures to minimise microplastic contamination

How much microplastic can potentially be released from defined paint and lacquer systems?

Facades, corrosion protection on buildings, marine paints, etc.

Development of an **aging and simulation method**

Different mechanisms such as rain, sunlight, temperature deviation (spalling), mechanical (and possible biological) abrasion

Comparison of results from the laboratory with environmental measurements

Definition of counter-measures to minimise microplastic contamination through paints and lacquers

Project Goals

Development of a suitable sample preparation and analytical method for the identification and quantification of microplastic particles out of paints and lacquers

Development of an **aging test**, which gives information on the tendency of paints and lacquers to release microplastic particles into the environment

Development of suitable **simulation tests** (in the laboratory) and outdoor weathering tests (OFI weathering station and public buildings) -> comparison laboratory and reality

Method Development

Development of a suitable sample preparation and analytical method for the identification and quantification of microplastic particles out of paints and lacquers

- FITR and Raman spectroscopy/microscopy to analyse:
  - reference lacquer systems (e.g. based on polyurethane, epoxide, acrylate, alkyd resins
  - colourless lacquers -> liquid and solid (as powders) after hardening on defined surfaces (metal, wood,…) with defined abrasion (rain, spalling, wind,…)
- Targeted addition of pigments to ensure the traceability (‚colour-tracer‘) and optimise sensitivity and accuracy of the method
- Optimisation of sampling and the analysis of different surfaces (wood, metal,…) for the suitable collection of microplastic particles and optimisation to lower sensitivity and detection limits

Aging Tests

Development of an **aging test**, which gives information on the tendency of paints and lacquers to release microplastic particles into the environment

- Analysis of relevant environmental impacts, such as rain, sunlight, deviation in temperature and mechanical abrasion at laboratory scale regarding the formation of microplastic particles
- Assess the degradation and dissolution mechanisms of paints and lacquers
- Testing of the influence of proper maintenance (e.g. use of care products,…) to reduce the release of microplastics

Simulation Tests

Development of suitable **simulation tests** (in the laboratory) and outdoor weathering tests (OFI weathering station and public buildings) -> comparison laboratory and reality

- Development of suitable simulation tests (in the laboratory) and outdoor weathering tests (OFI weathering station and public buildings)
- Analysis of relevant environmental impacts, such as rain, sunlight, deviation in temperature and mechanical abrasion at laboratory scale regarding the formation of microplastic particles
- Concordance of developed aging test and outdoor weathering station
- Testing of the influence of proper maintenance (e.g. use of care products,…) to reduce the release of microplastics

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