MICROPLASTICS FOR BREAKFAST

Mikroplastika za doručak

Prvi susret istraživača mikro i nanoplastike iz Srbije, Crne Gore & Bosne i Hercegovine

Preparation and characterization of microplastics for environmentally relevant laboratory research

Assist. prof. dr. Ula Putar

University of Ljubljana, Faculty of Chemistry and Chemical Technology

Vrčin, 10. 4. 2025

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Faculty of Chemistry and Chemical Technology

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Introduction

University of Ljubljana, Faculty of Chemistry and Chemical Technology, Ljubljana, Slovenia

Chair of Chemical Process, Environmental and Biochemical Engineering

PLANTerastic research team:
https://planterastics.fkkt.uni-lj.si/

- > 10 years of microplastic research
- > 35 published papers
- 4 book chapters
- 6 national research project
- 1 international research project

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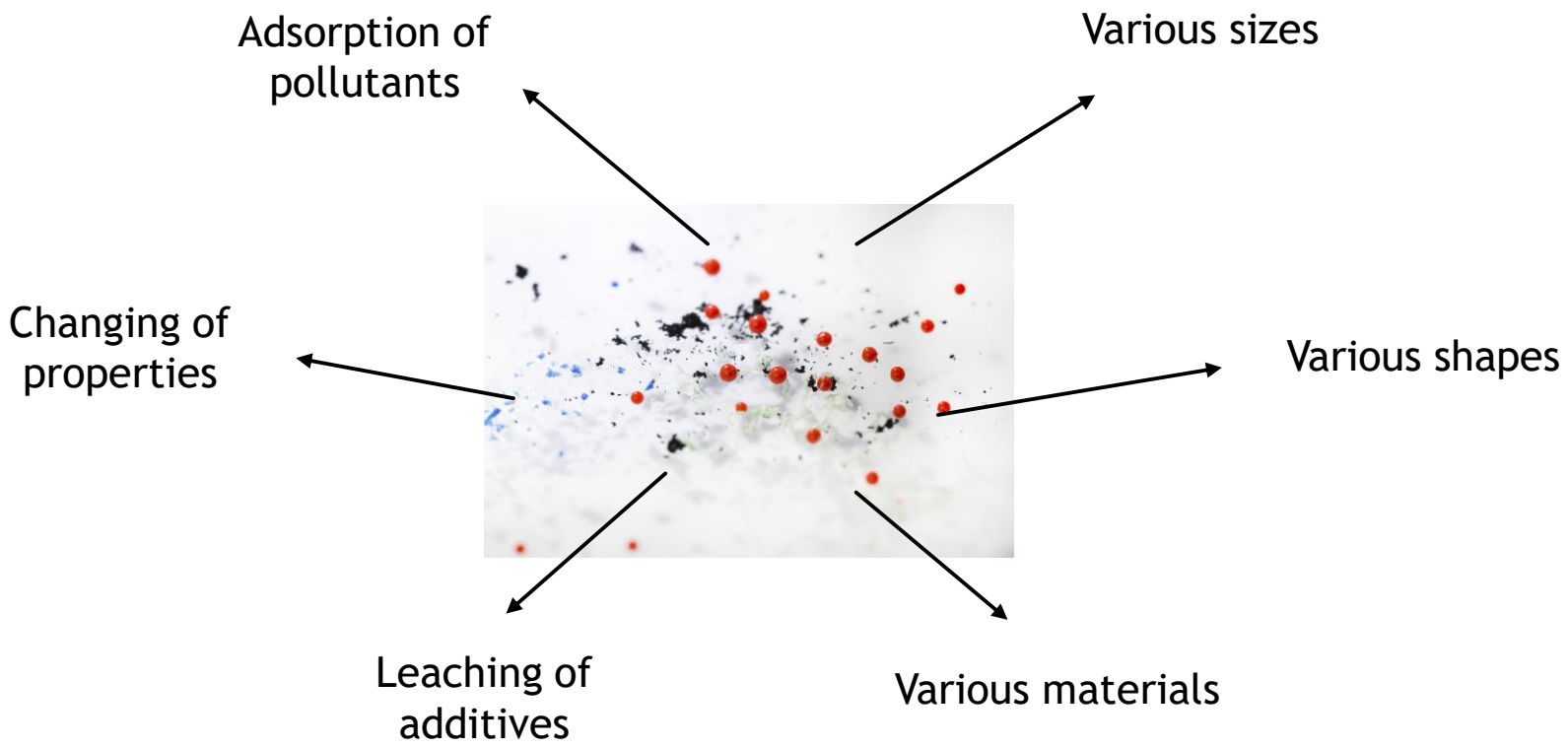
Microplastics



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Microplastics

20 µm

A microplastic particle…

800 µm

… also a microplastic particle

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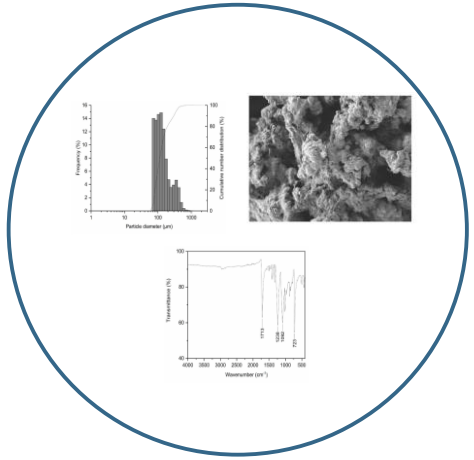
Microplastics for laboratory research

Preparation



Characterization

Aging/weathering

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Microplastics for laboratory research

Preparation

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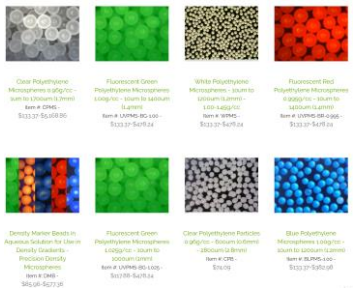
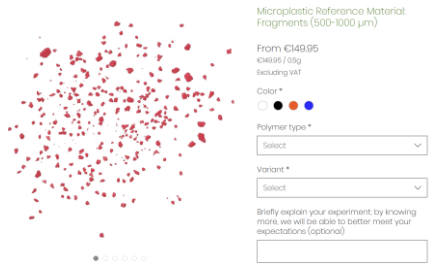
1. Microplastics preparation

- **Purchase**
  - Industrial microplastics – specific size and chemical composition
    - Spheres → low environmental relevance
    - Fragments





- Products that contain microplastics (e.g. cosmetic and personal care products) – spherical and fragments, environmentally relevant, characterization needed

https://youtu.be/RjXNXNHGQys

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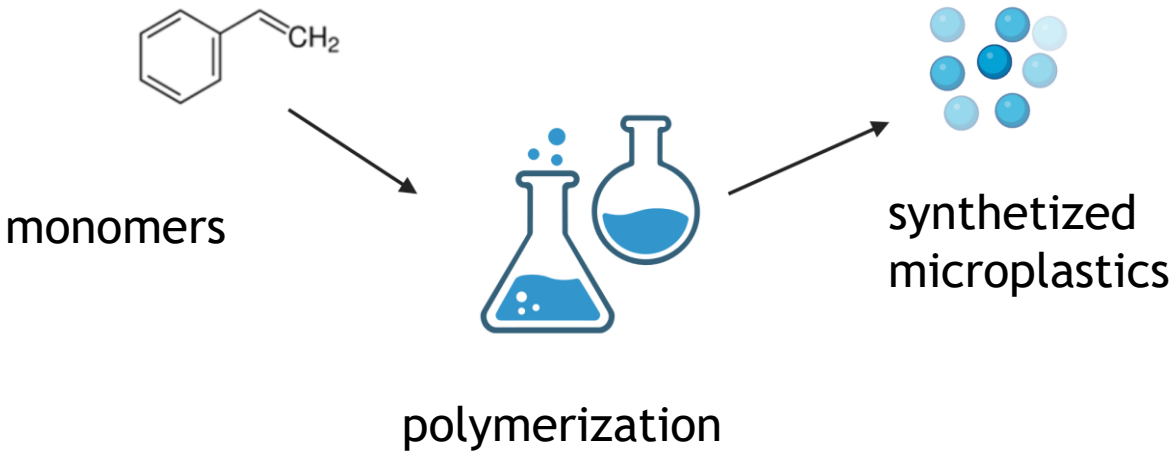
1. Microplastics preparation

- **Preparation**
  - Bottom-up (i.e., chemical synthesis) – various shapes, sizes (characterization is needed), environmentally relevant?



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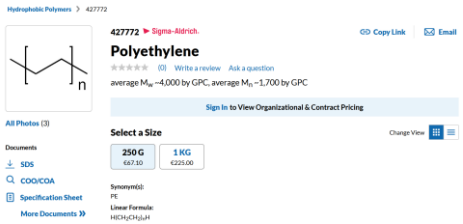
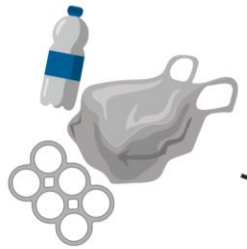
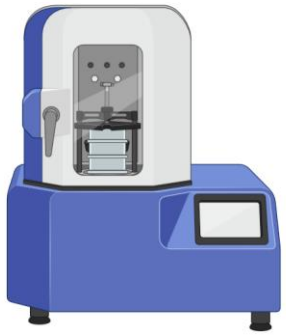
1. Microplastics preparation

- **Preparation**
  - Top-down (i.e., fragmentation) – various shapes, sizes (characterization is needed), environmentally relevant





Plastic items
Pellets



Fragmentation

Microplastic fragments

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1. Microplastics preparation

- **Preparation**
  - Top-down: Fragmentation

Centrifugal mill

Ball mill

Blender/mill

- Liquid nitrogen
- Dry ice

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1. Microplastics preparation

- **Preparation**
  - Top-down: Fragmentation

Example: Centrifugal mill

https://www.youtube.com/watch?v=fR01N7NF4N0&feature=emb_title

Example: Ball mill

https://www.youtube.com/watch?v=VJDEuF8CEWI

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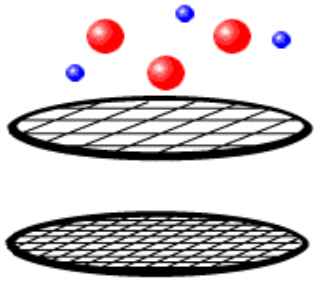
1. Microplastics preparation

- **Preparation**
  - Top-down: Fragmentation
    - Obtaining particles of specific size? → **Sieving**



https://www.youtube.com/watch?v=5ommkOS1Fek&list=PLAUyTgal3dz80NvJrpj4DvClpsO2wAxdR&index=7

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Microplastics for laboratory research

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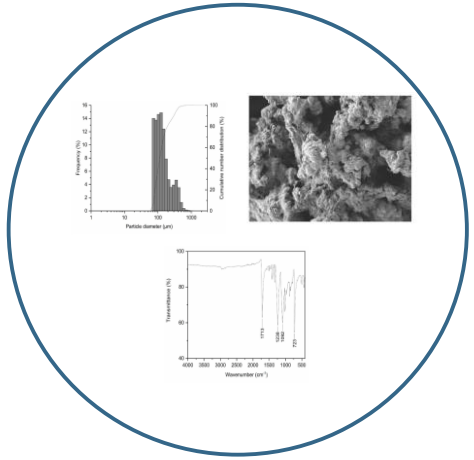

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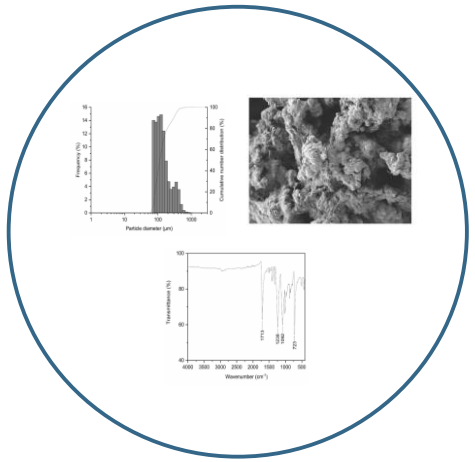
Microplastics for laboratory research



Characterization

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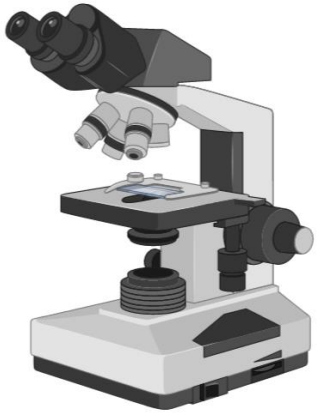
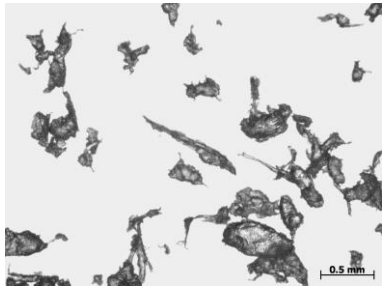
2. Microplastics characterization

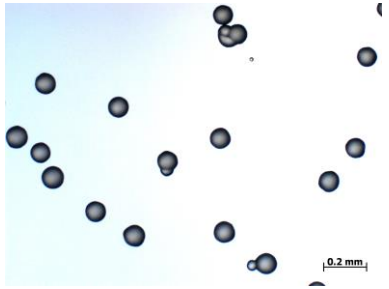
- **Shape**

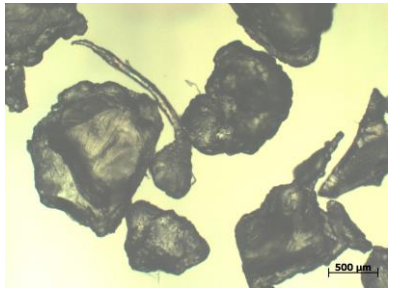
Light microscope

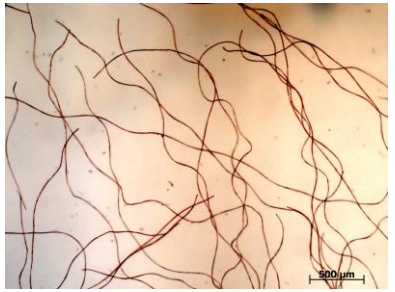


Stereomicroscope









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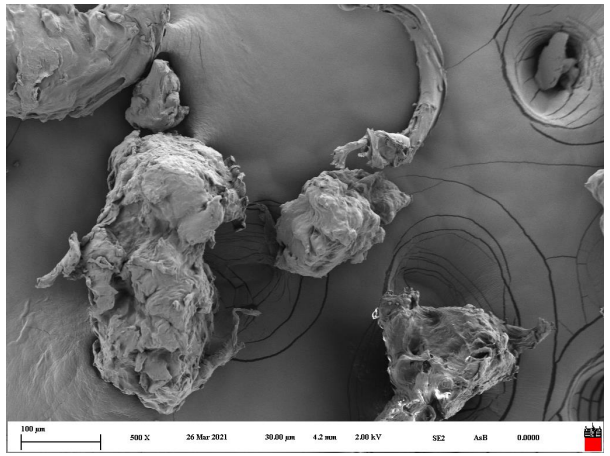
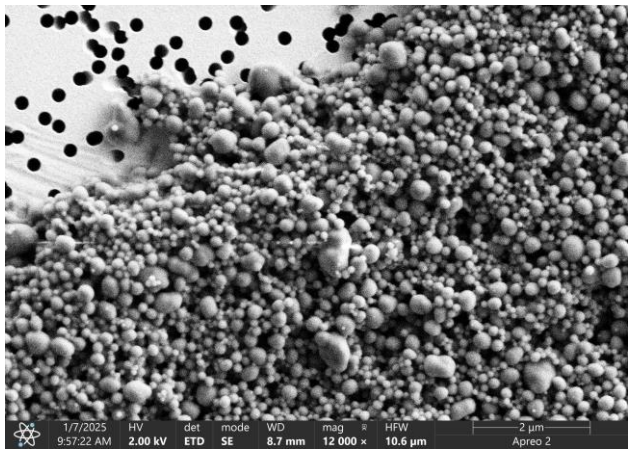
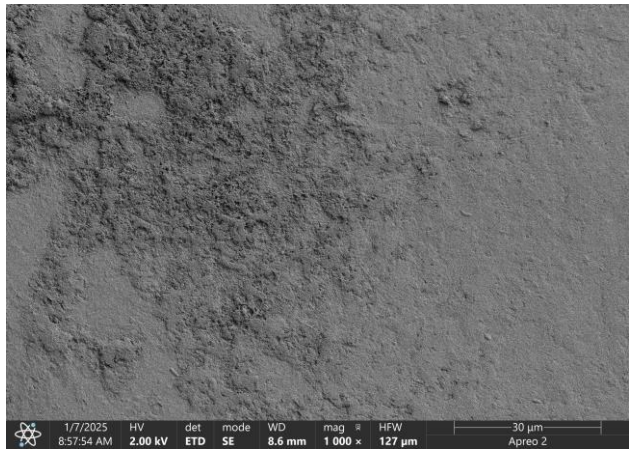
2. Microplastics characterization

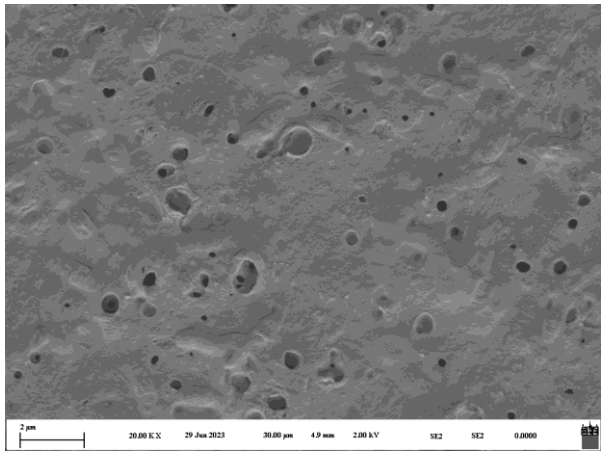
- **Shape/Surface morphology**

Scanning electron microscopy (SEM)
Transmission electron microscopy (TEM)









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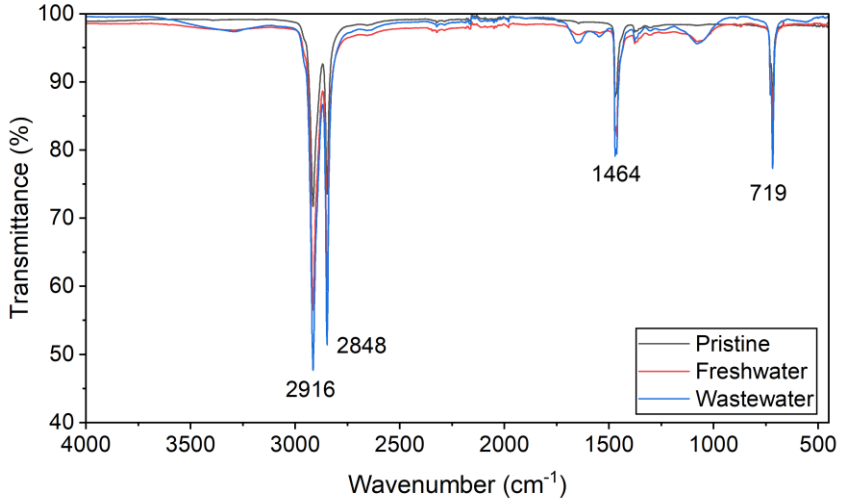
2. Microplastics characterization

- **Chemical composition**
  - Fourier-transform infrared spectroscopy (FTIR)
  - Raman spectroscopy
  - Thermal analysis and mass spectrometry (PY-GC–MS)
  - Laser based methods (LIBS, LA-ICP-MS…)
  - Scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM-EDX)
  - …

2. Microplastics characterization

- **Chemical composition**
  - Fourier-transform infrared spectroscopy (FTIR)



2916 - CH_2 - asymmetric C-H streching
2848 - CH_2 - symmetric C-H streching
1464 - CH_2 - bending
719 - CH_2 rocking

- Comparisons with spectral libraries and articles
- FTIR free libraries:
  - Spectra Chem: https://spectra.chem.ut.ee/
  - Wiley: https://spectrabase.com/
  - Cameo material database: https://cameo.mfa.org/wiki/Category:Materials_database
  - https://webbook.nist.gov/chemistry/

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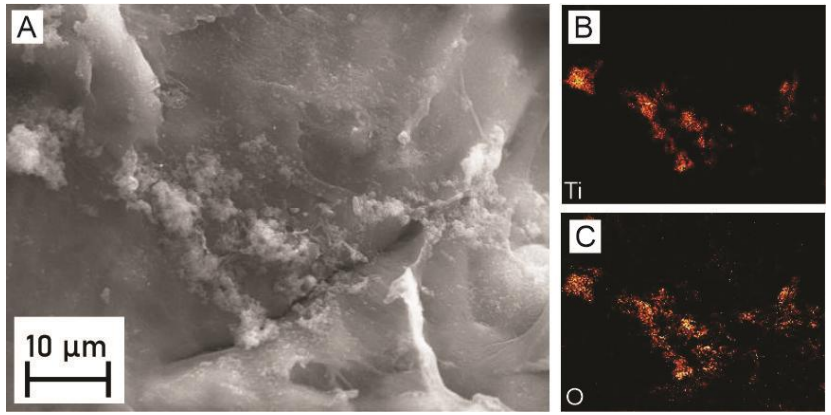
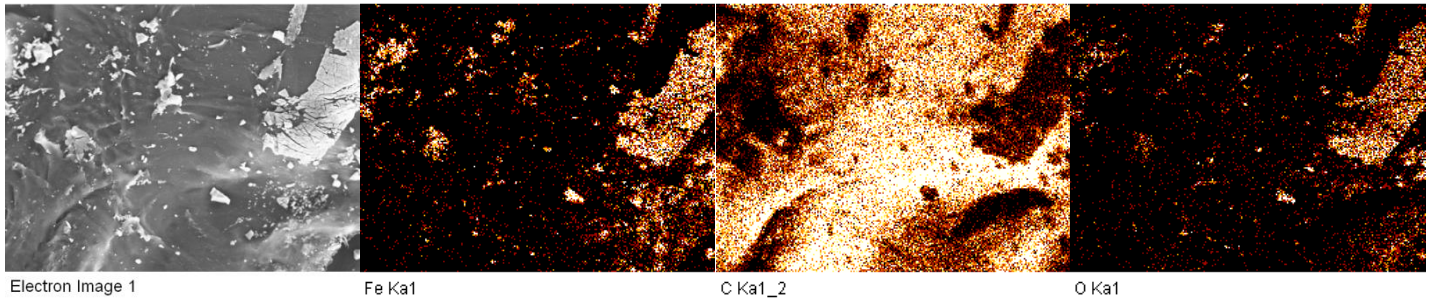
2. Microplastics characterization

- **Chemical composition → elemental composition**
  - Scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM-EDX)



PE with adsorbed $nTiO_2$

PE modified with magnetic fluids



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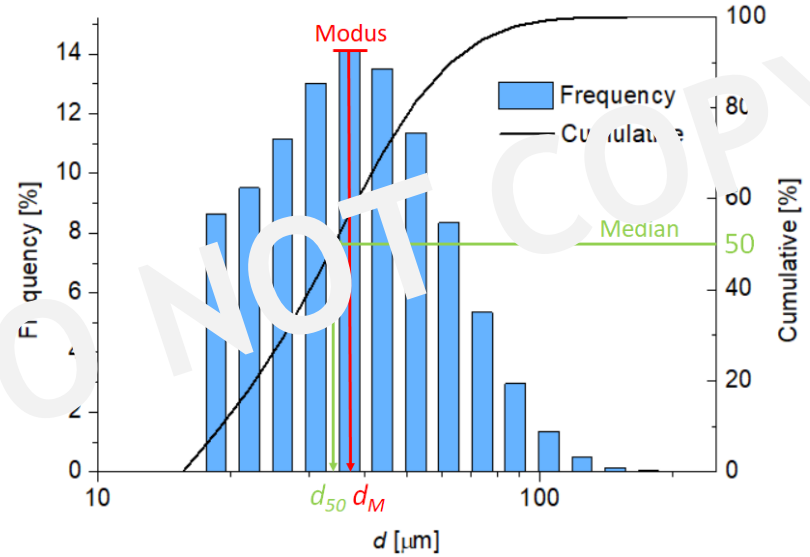
2. Microplastics characterization

- **Size analysis**
  - Sieving analysis
  - Optical/stereo microscope
  - Laser diffraction analysis
  - …

- Mean ± SD
- Min. – Max.
- **Particle size distribution**



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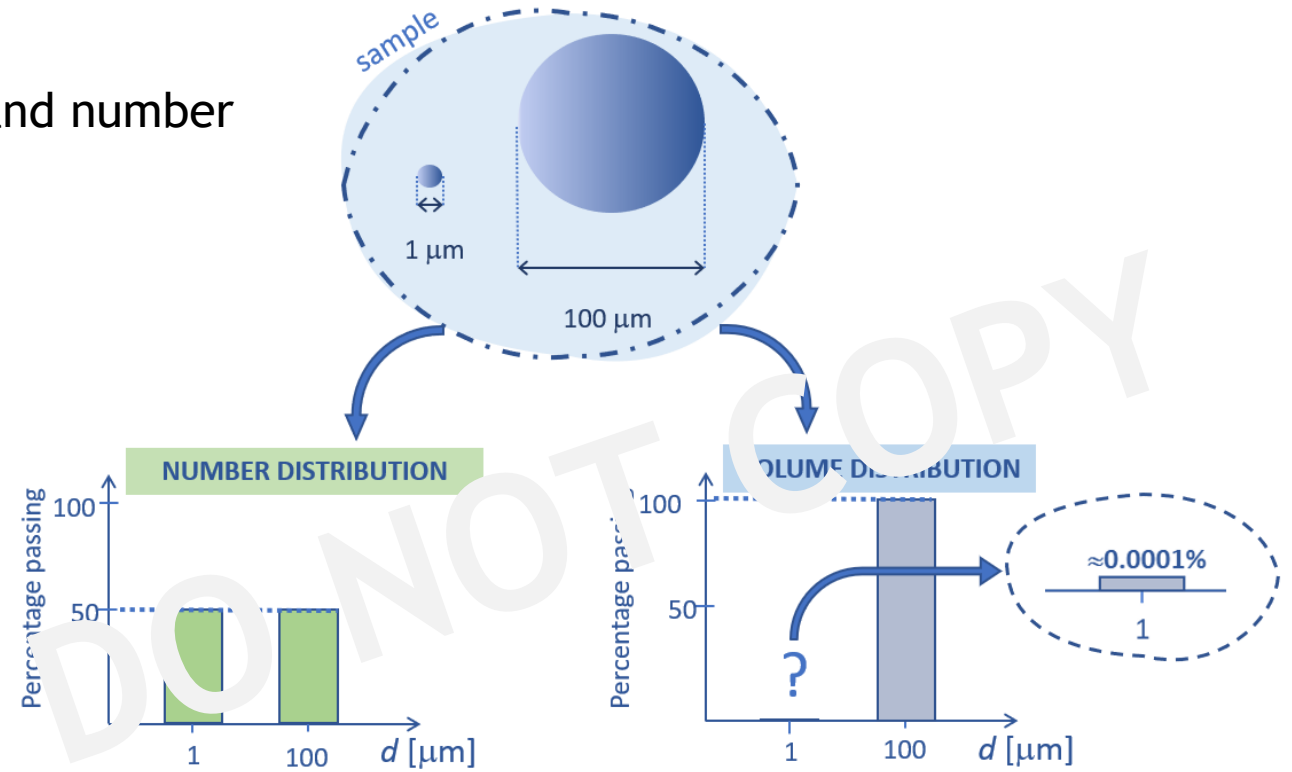
2. Microplastics characterization

- **Size analysis**
  - Particle size distribution – volume and number



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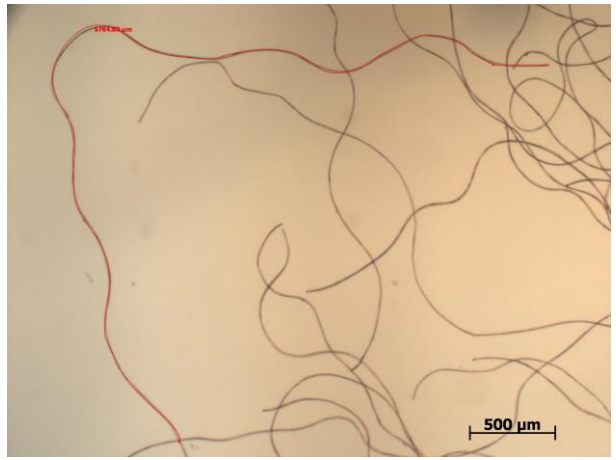
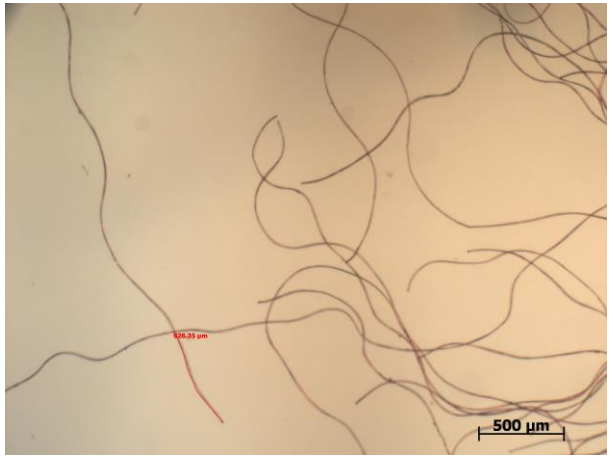
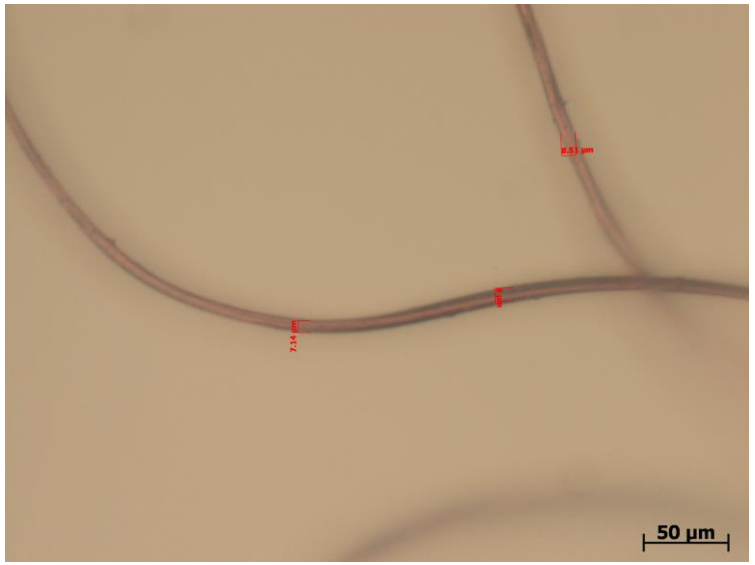
2. Microplastics characterization

- **Size analysis**
  - Optical microscope







5765 µm + 626 µm = 6391 µm

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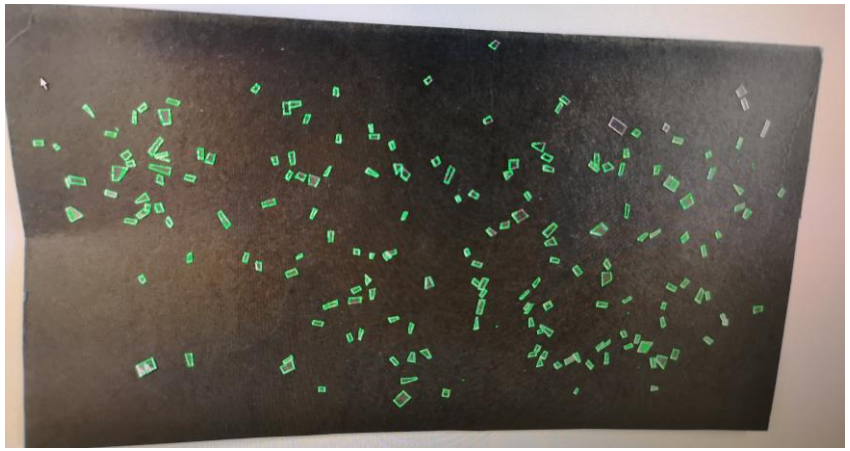
2. Microplastics characterization

- **Size analysis**
  - Photography (digital camera) and processing

Larger particles (>2 mm)



AxioVision program

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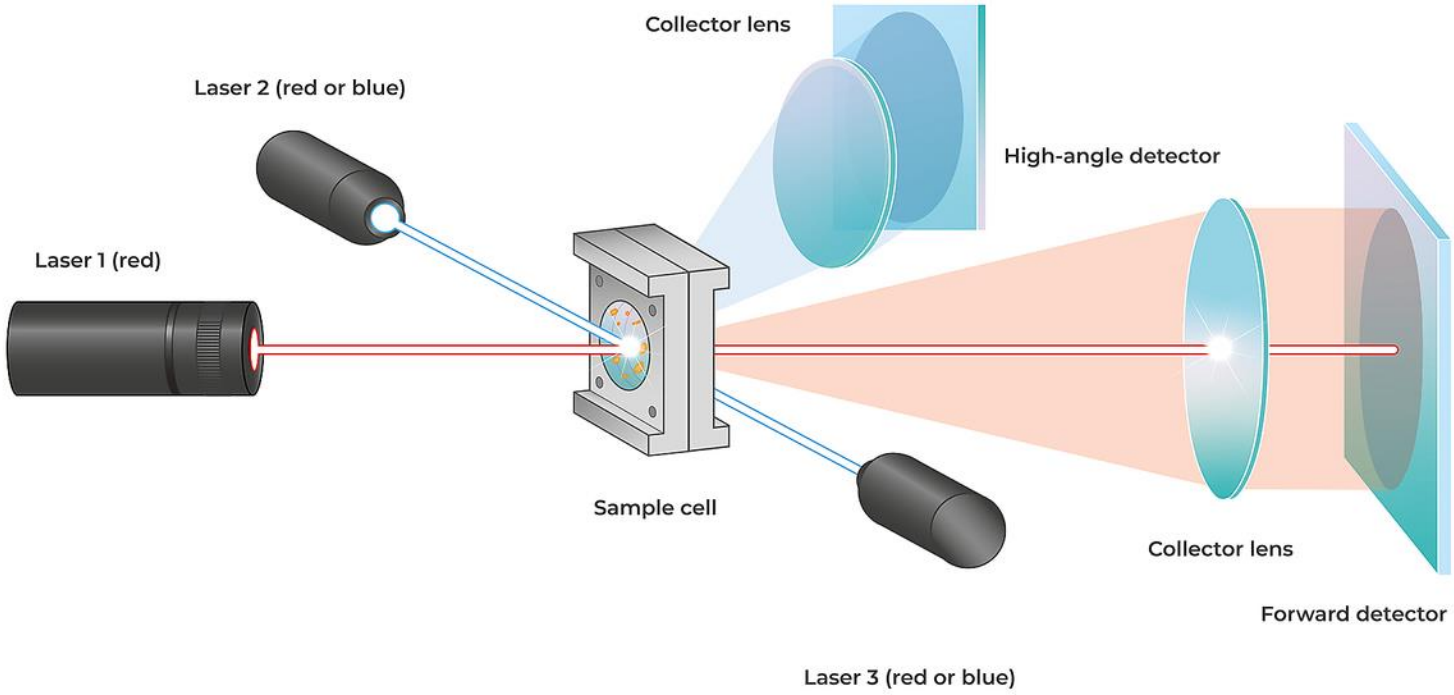
2. Microplastics characterization

- **Size analysis**
  - Laser diffraction analyser



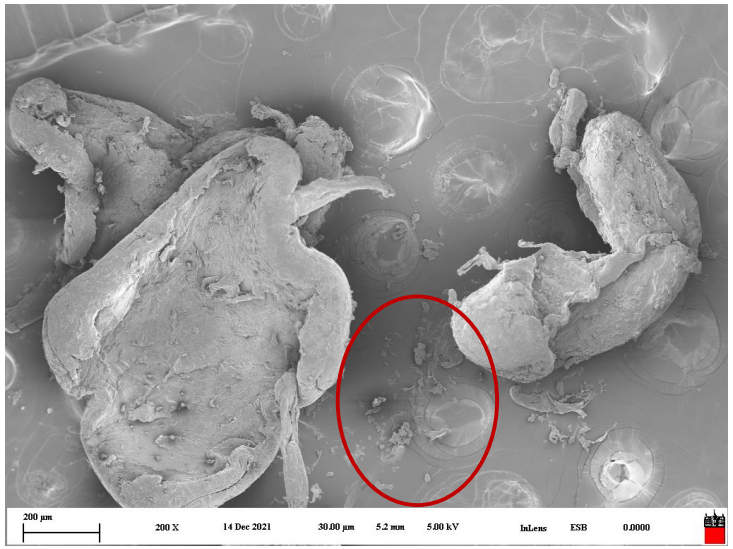
https://youtu.be/ZQqlOrEPl3I

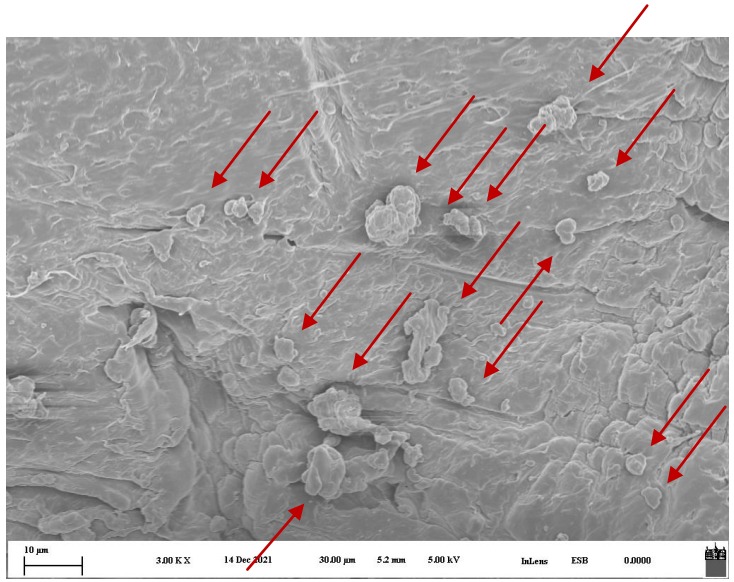
2. Microplastics characterization

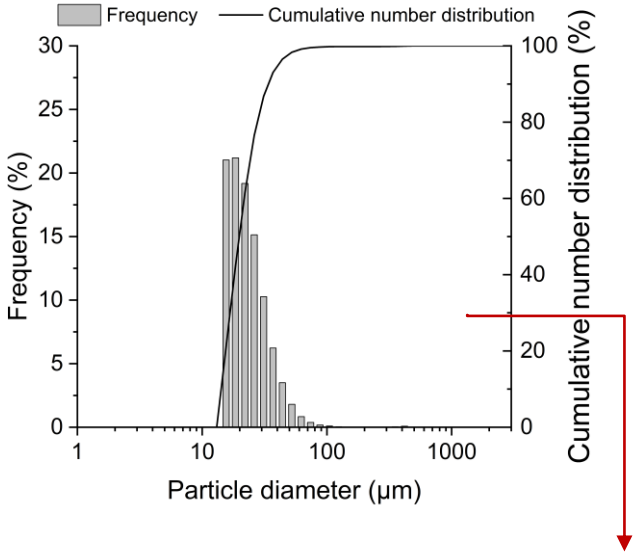
- **Are results always accurate?**

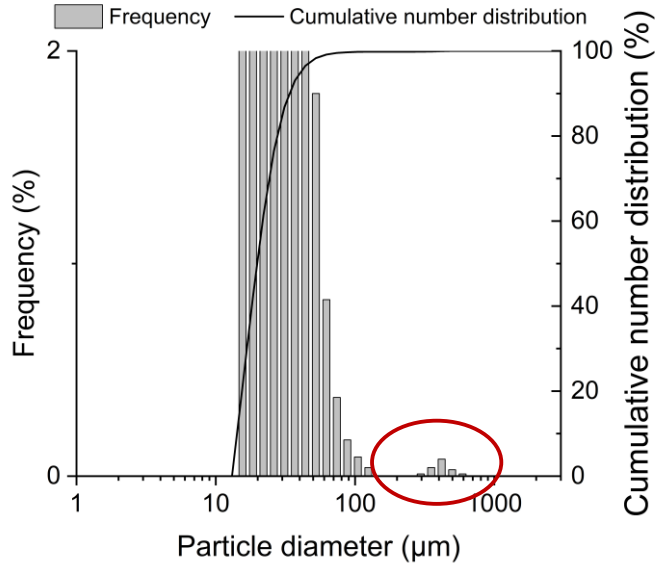
  Small/large particles

Mean ± SD
Numerical: 23 ± 7 µm
Volume: 351 ± 179 µm









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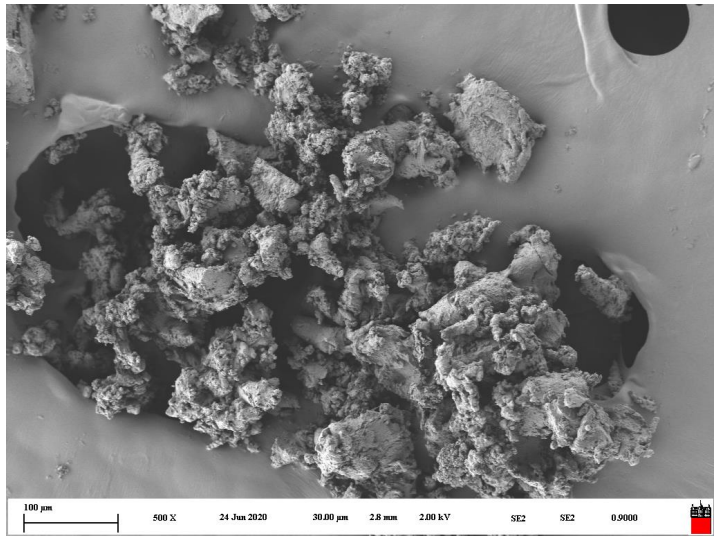
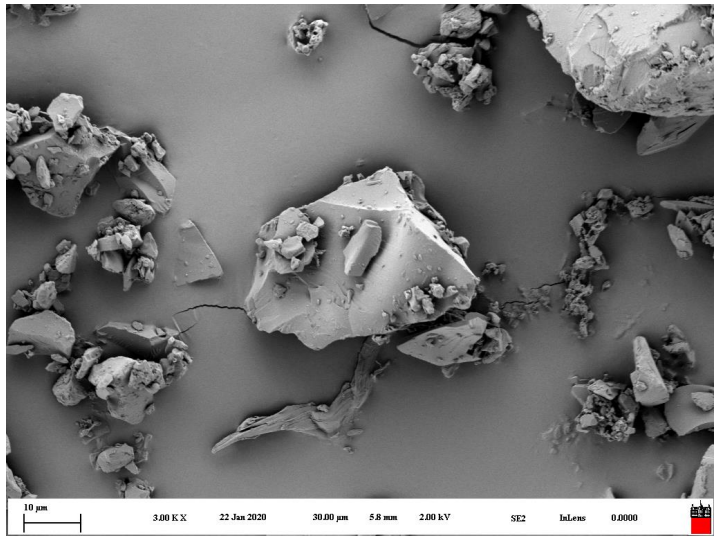
2. Microplastics characterization

- **Are results always accurate?**

Agglomeration



Size measurement with addition of surfactant



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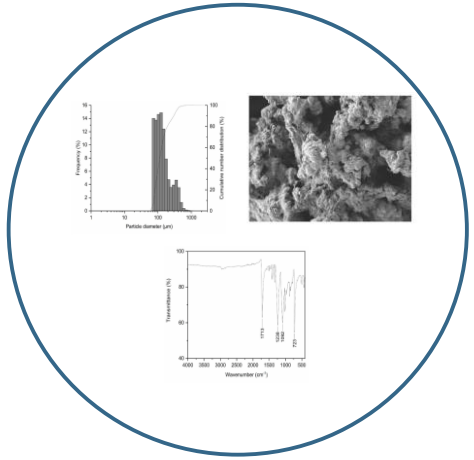
2. Microplastics characterization

- **Other characteristics**
  - Number of particles per mass
  - Specific surface area (BET method)
  - Zeta potential, point of zero charge (DLS – dynamic light scattering, potentiometric titration)
  - Crystallinity (DSC – differential scanning calorimetry, XRD – X-ray diffratography
  - Wettability (contact angle)
  - Molar mass (chromatography)
  - Density (pycnometer)
  - Presence of additives (GC/LC-MS)
  - …

Microplastics for laboratory research

Preparation



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Aging/weathering

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Microplastics for laboratory research

Aging/weathering

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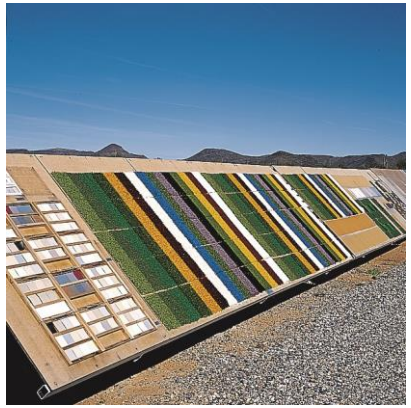
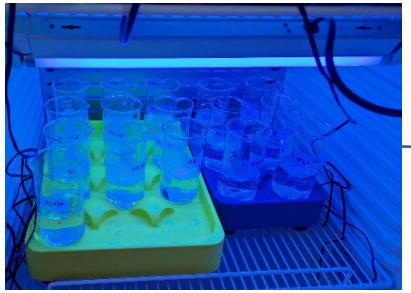
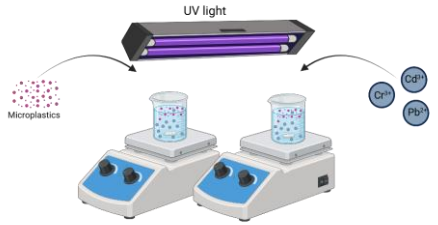
3. Microplastics aging

- **Abiotic aging**
  - Accelerated weathering, e.g. Suntest, chemical oxidation
  - Laboratory approach (simulating natural conditions)
  - Outdoor approach







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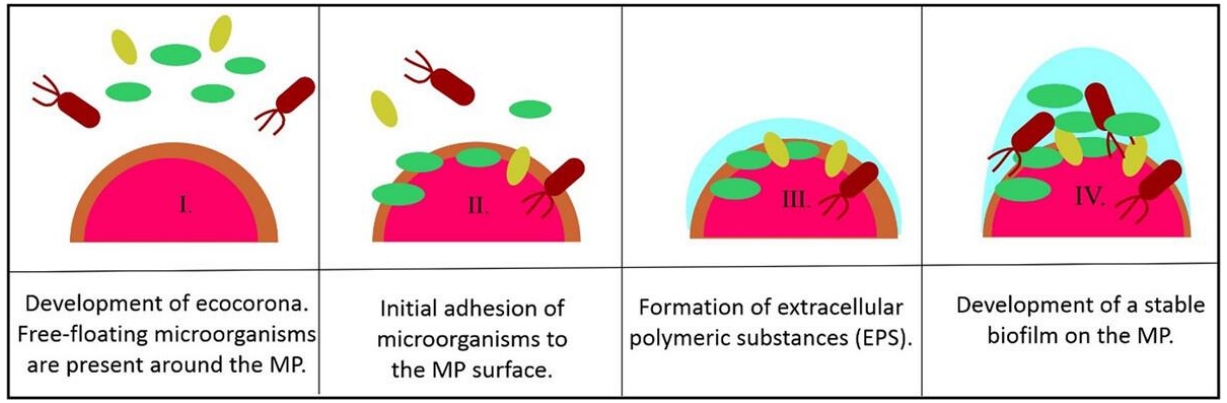
PLANT ERASTICS

FKKT UNIVERSITY OF LJUBLJANA Faculty of Chemistry and Chemical Technology

3. Microplastics aging

- **Biotic aging/biofouling**



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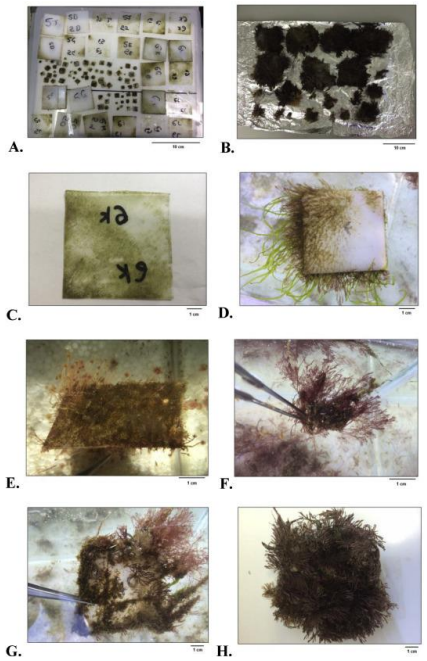
3. Microplastics aging

- **Biotic aging/biofouling**
  - **Natural aging** in the environment (sea, lake…): environmentally relevant but difficult for microplastics (aged in mesh)
  - **In laboratory**
    - Specific monocultures – low environmental relevance
    - Combined approach: natural stream water, incubation in laboratory, increased environmental relevance



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3. Microplastics aging

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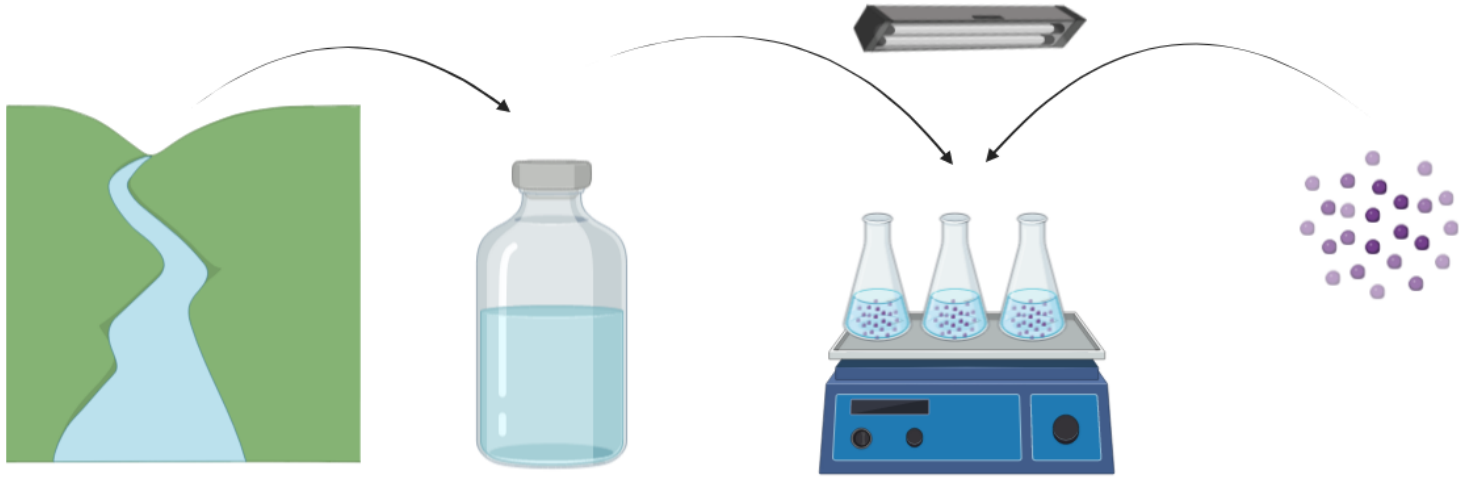

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3. Micro

- **Biotic ag**

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Microplastics

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Conclusions

- Different needs in different fields:
  - Development of analytical methods
  - Ecotoxicology
  - Interaction with other contaminants
  - Fate and behaviour in the environment
  - Degradation
  - …

But always characterize your particles!

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Slovenian Research and Innovation Agency

Thank you for your attention!

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