

Harmonized Analysis of Microplastics by FTIR Spectroscopy and Imaging

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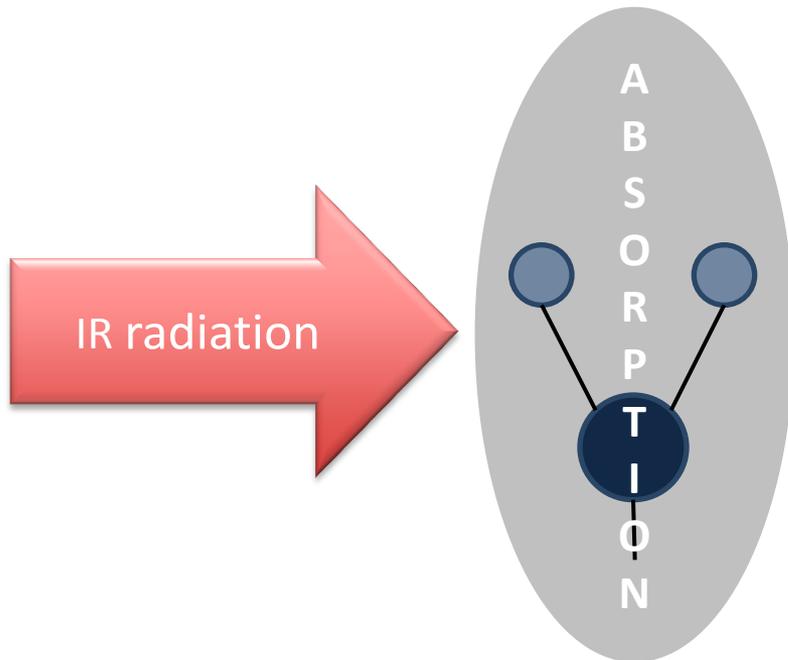
FTIR spectroscopy

Fourier transform infrared (FTIR)



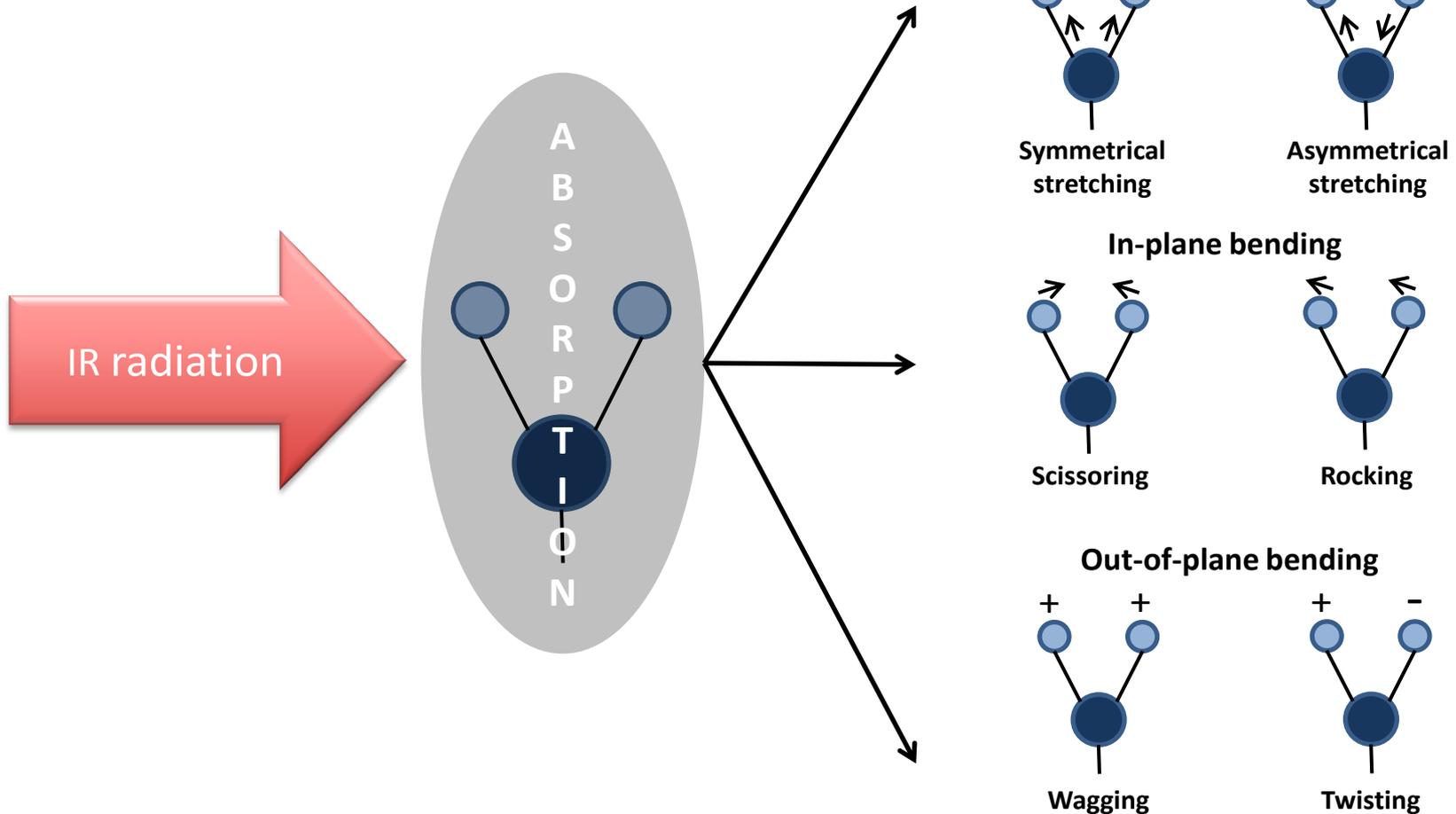
FTIR spectroscopy

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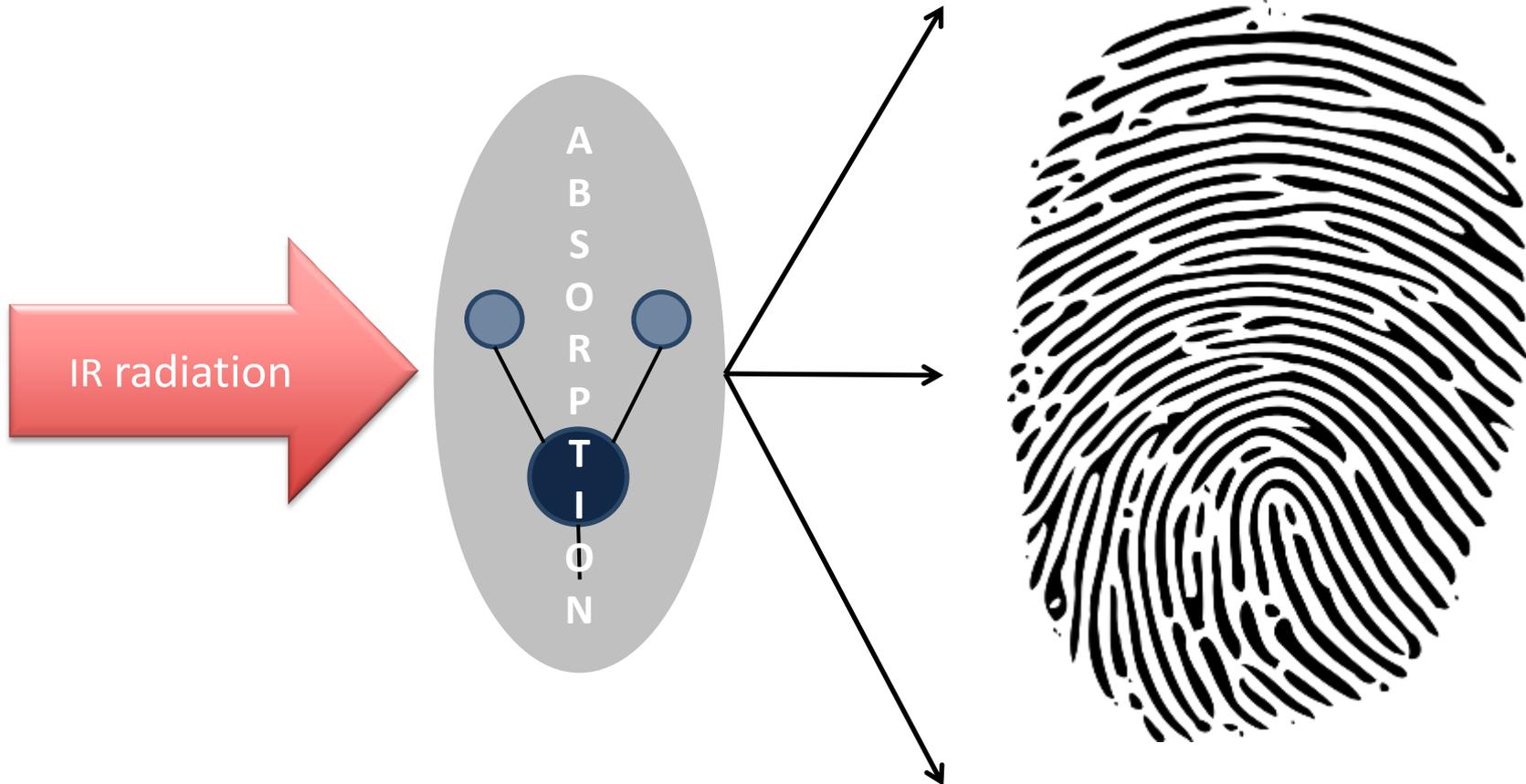
FTIR spectroscopy

Fourier transform infrared (FTIR)



FTIR spectroscopy

Fourier transform infrared (FTIR)



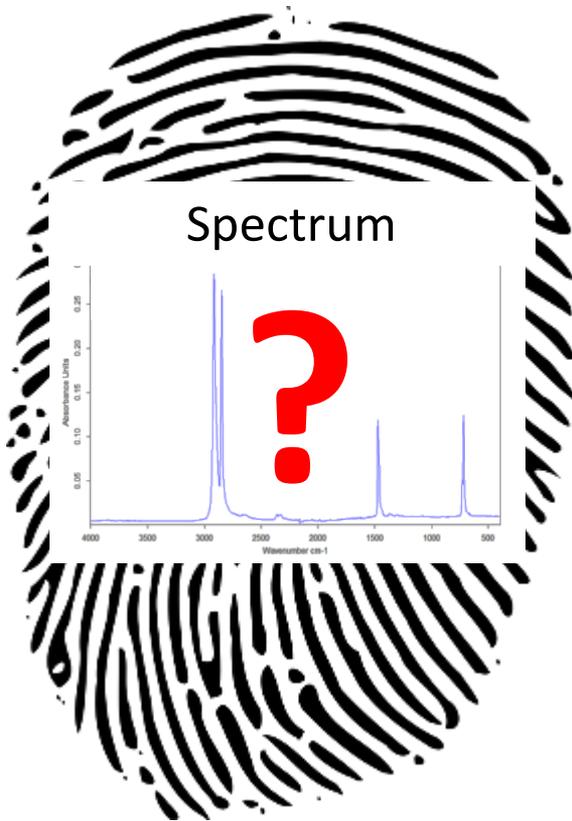
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Analysis via Library Search

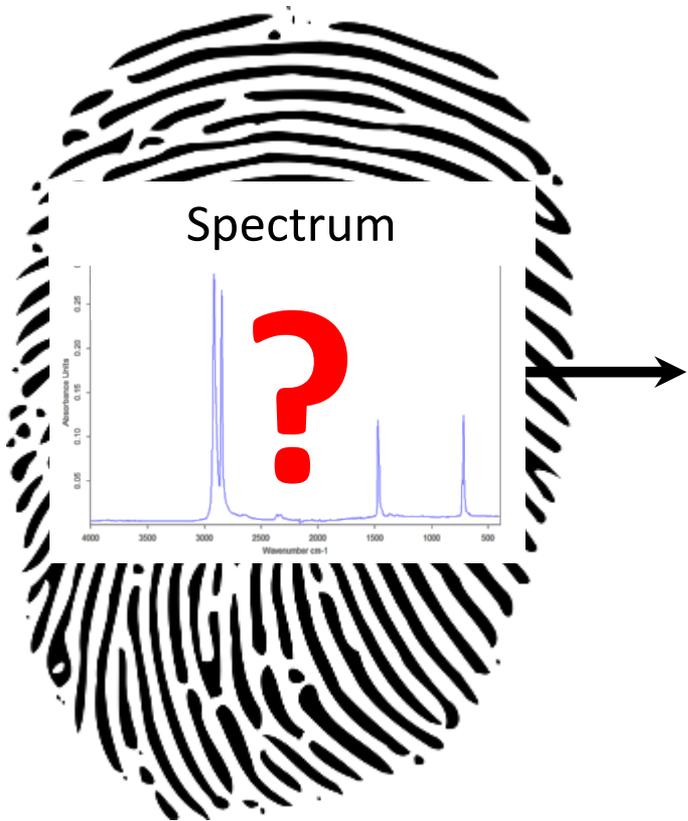


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Analysis via Library Search



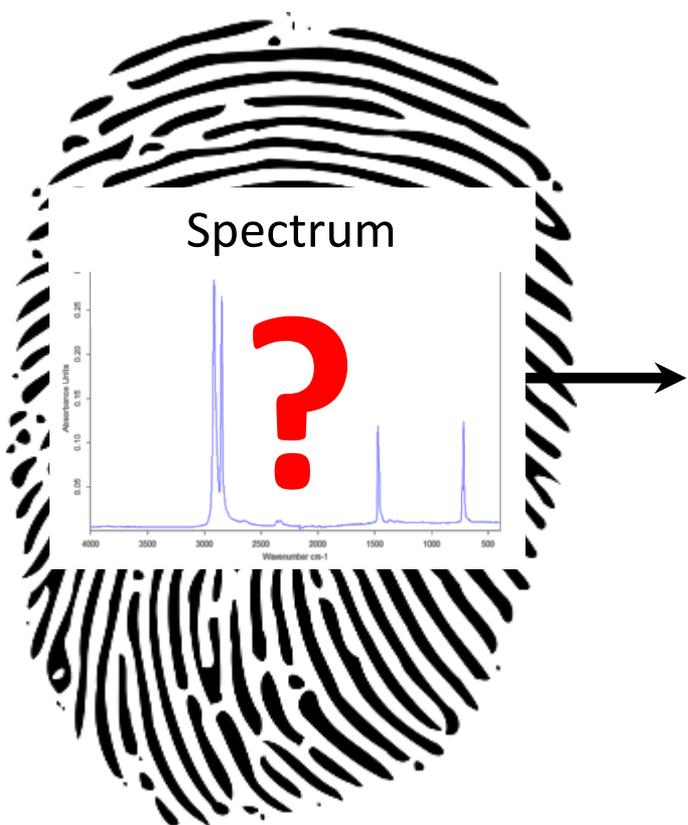
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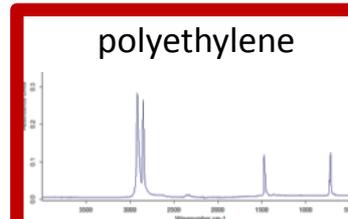
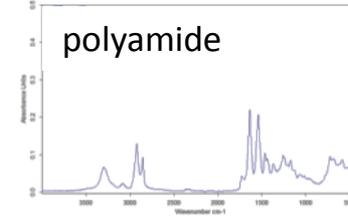
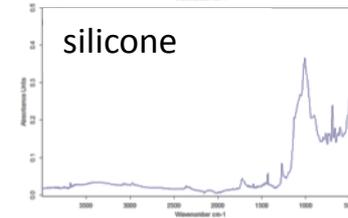
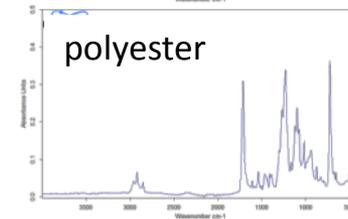
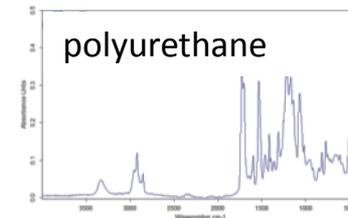
Spectral database



Analysis via Library Search

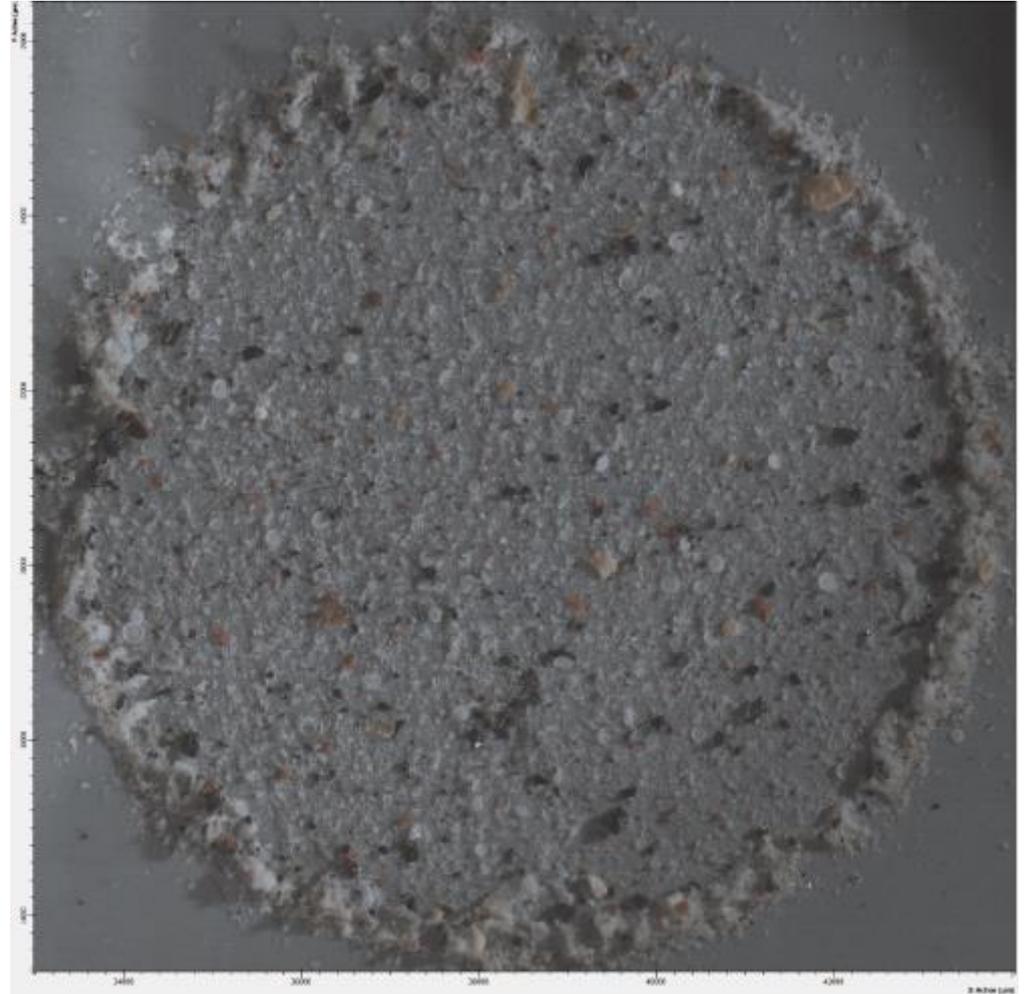


Spectral database



Why FTIR for MP analysis?

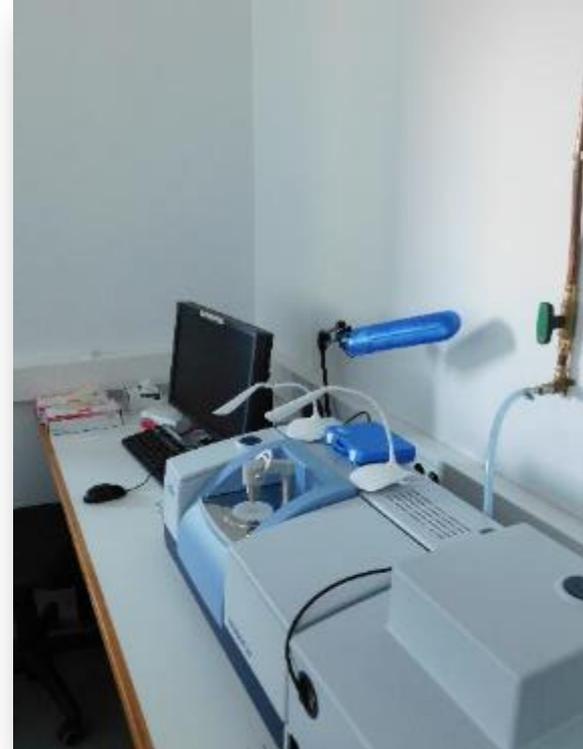
- Chemical imaging via a non destructive method.
- Sample preparation compared to RAMAN microscopy is less demanding.
- Complete mapping of membrane filters is possible.



Particles $> 500 \mu\text{m}$

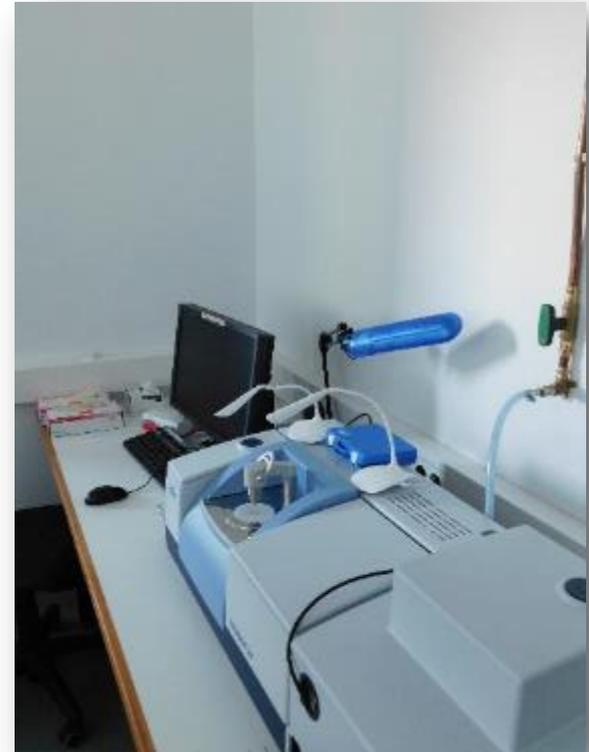
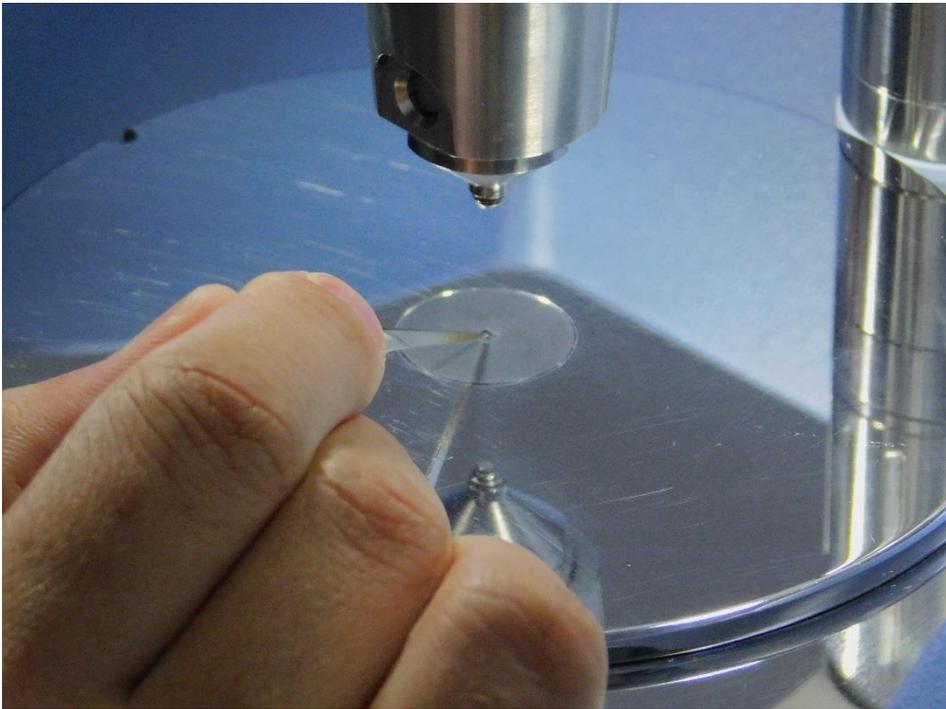
If the particles can be sorted by hand:

- Filtration onto filter meshes with $500 \mu\text{m}$ pore size
- Optical sorting of the particles
- Attenuated total reflection (ATR)-FTIR- measurement



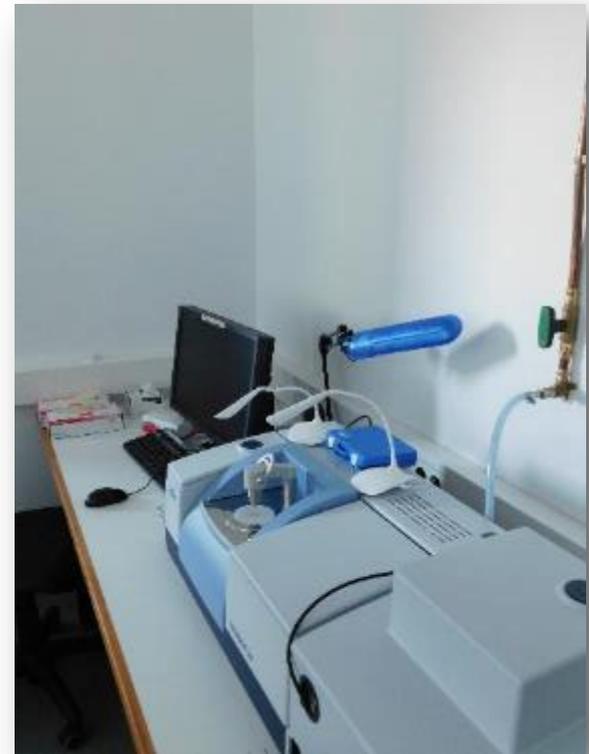
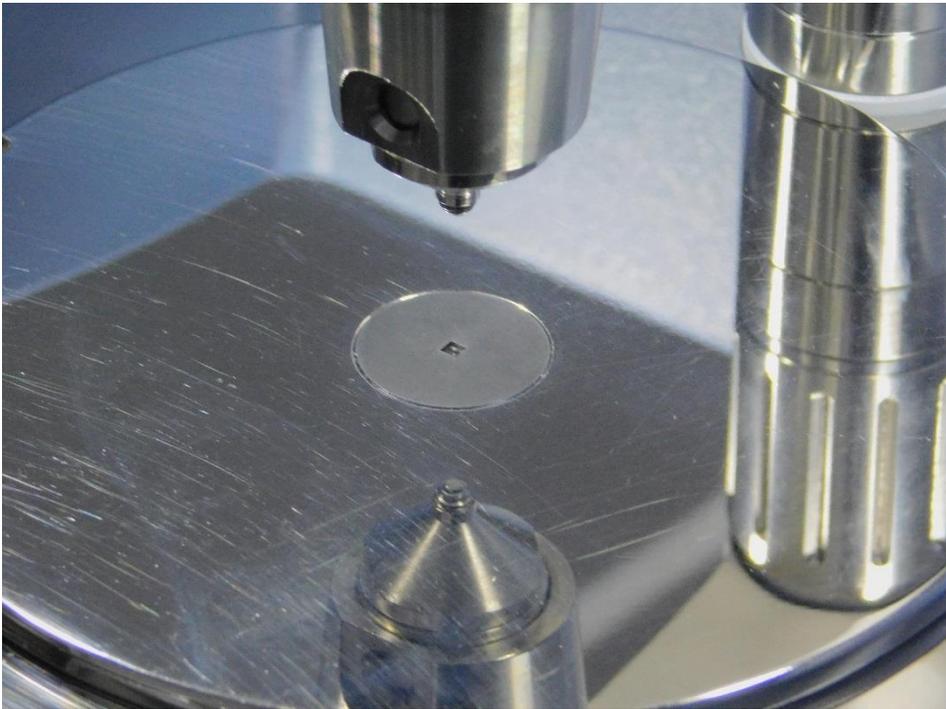
Particles $> 500 \mu\text{m}$

If the particles can be sorted by hand:

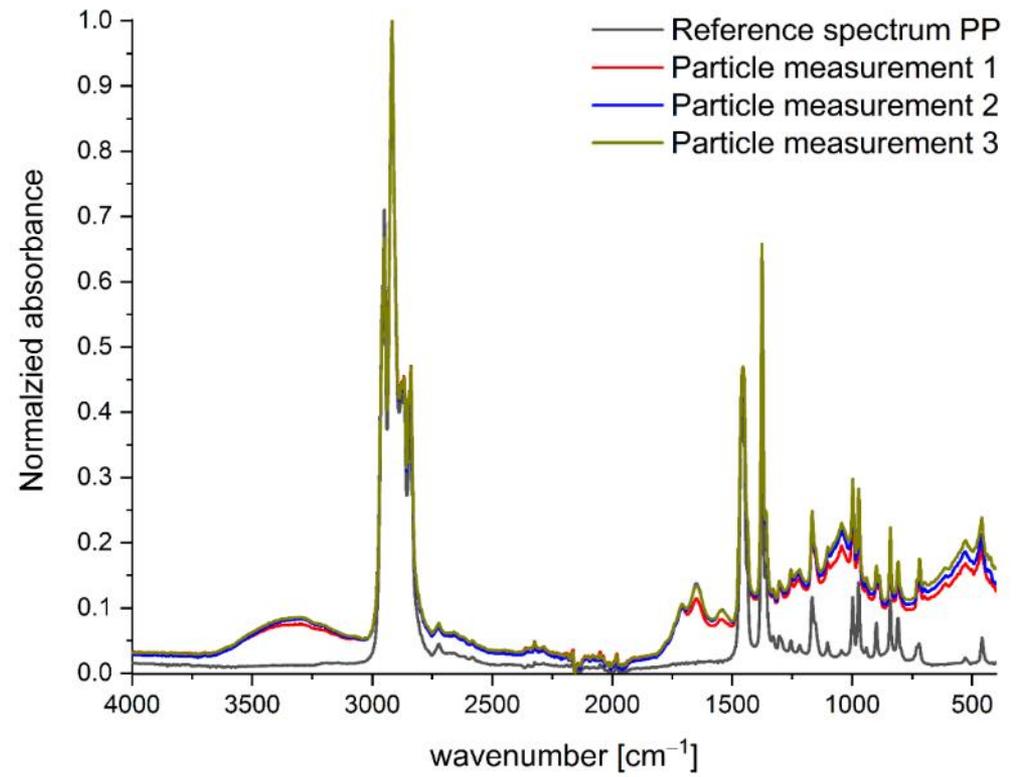
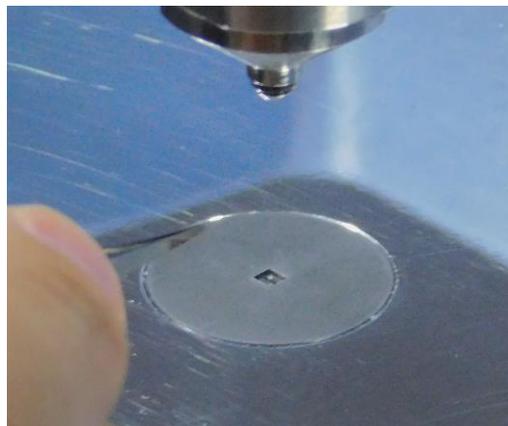


Particles $> 500 \mu\text{m}$

If the particles can be sorted by hand:



Example – ATR-FTIR-spectroscopy

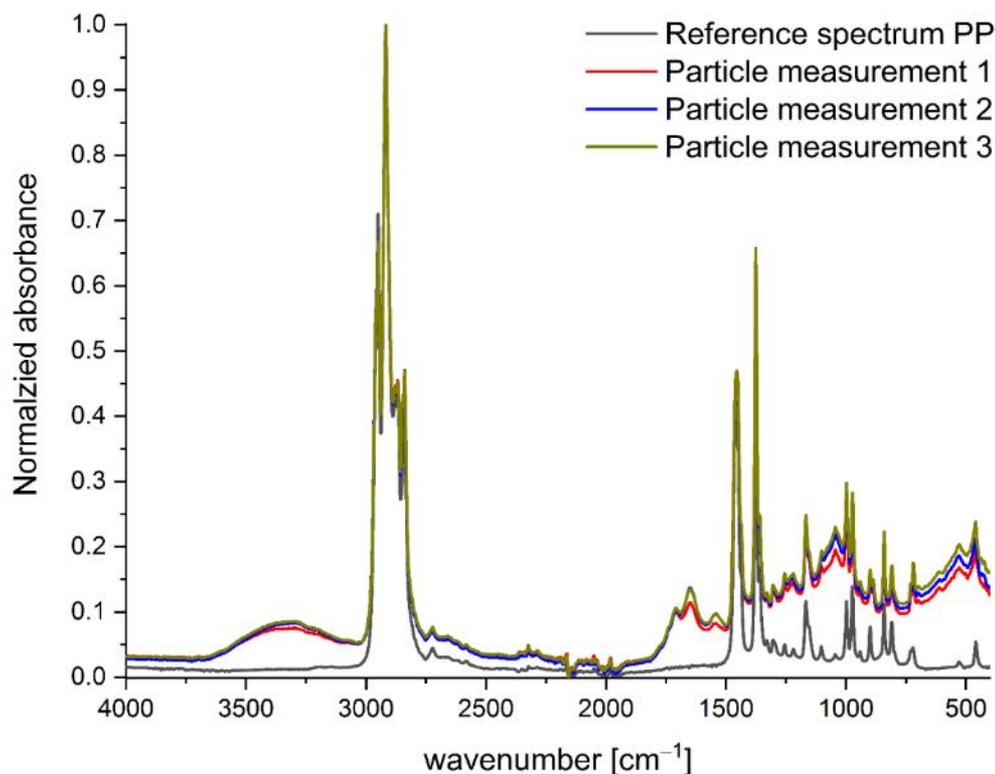


Towards Harmonization of Analysis



Single particles via attenuated total reflection (ATR) – FTIR:

Reference database available via Open Access in Primpke, S. et. al., Analytical and Bioanalytical Chemistry 2018, 410, (21), 5131-5141



1. Primpke, S. et. al., Analytical and Bioanalytical Chemistry 2018, 410, (21), 5131-5141

FTIR Imaging

Using the common Fourier-transform infrared (FTIR) spectroscopy

Allows the analysis of large filters (diameter usually 10 - 13 mm)

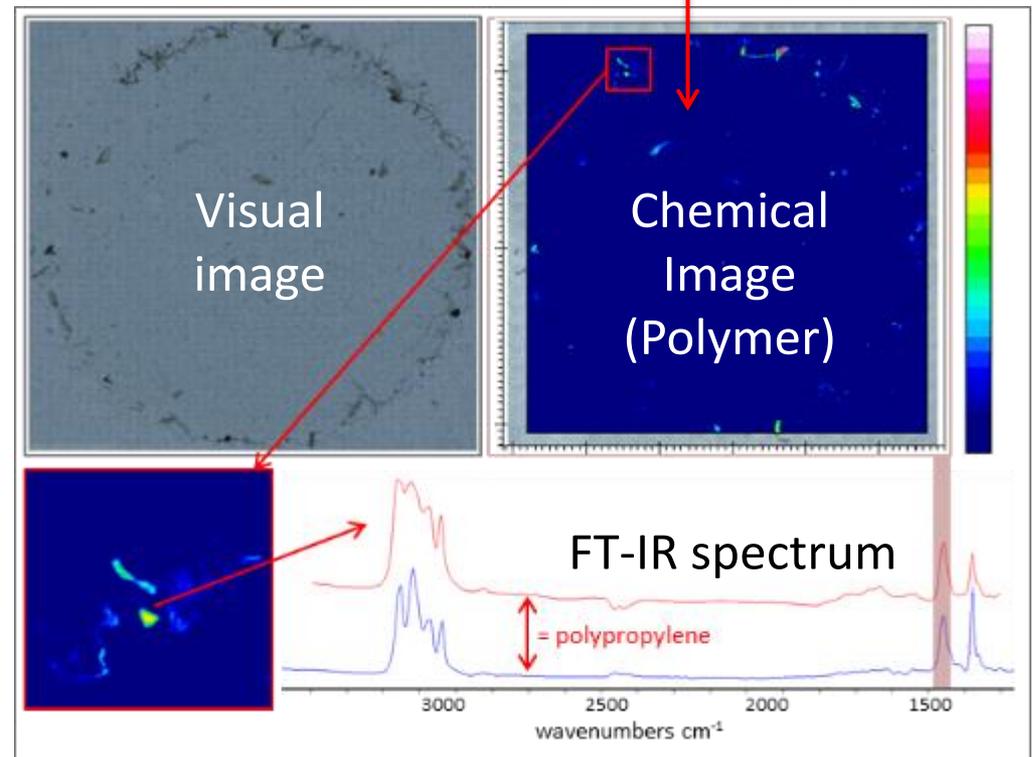
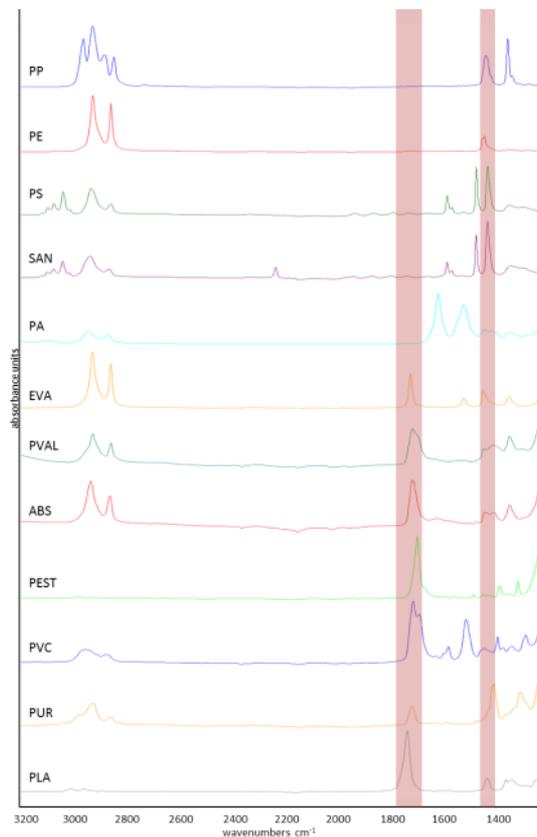
Applicable in transmission and reflection mode

Example: Sediment sample



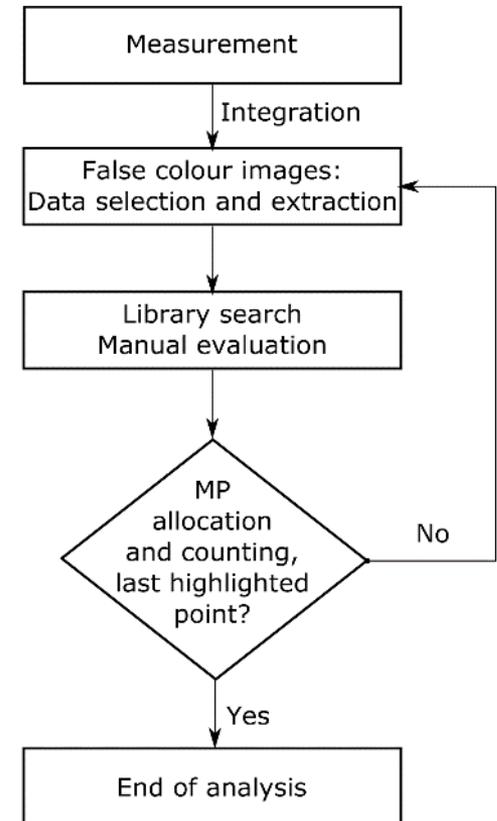
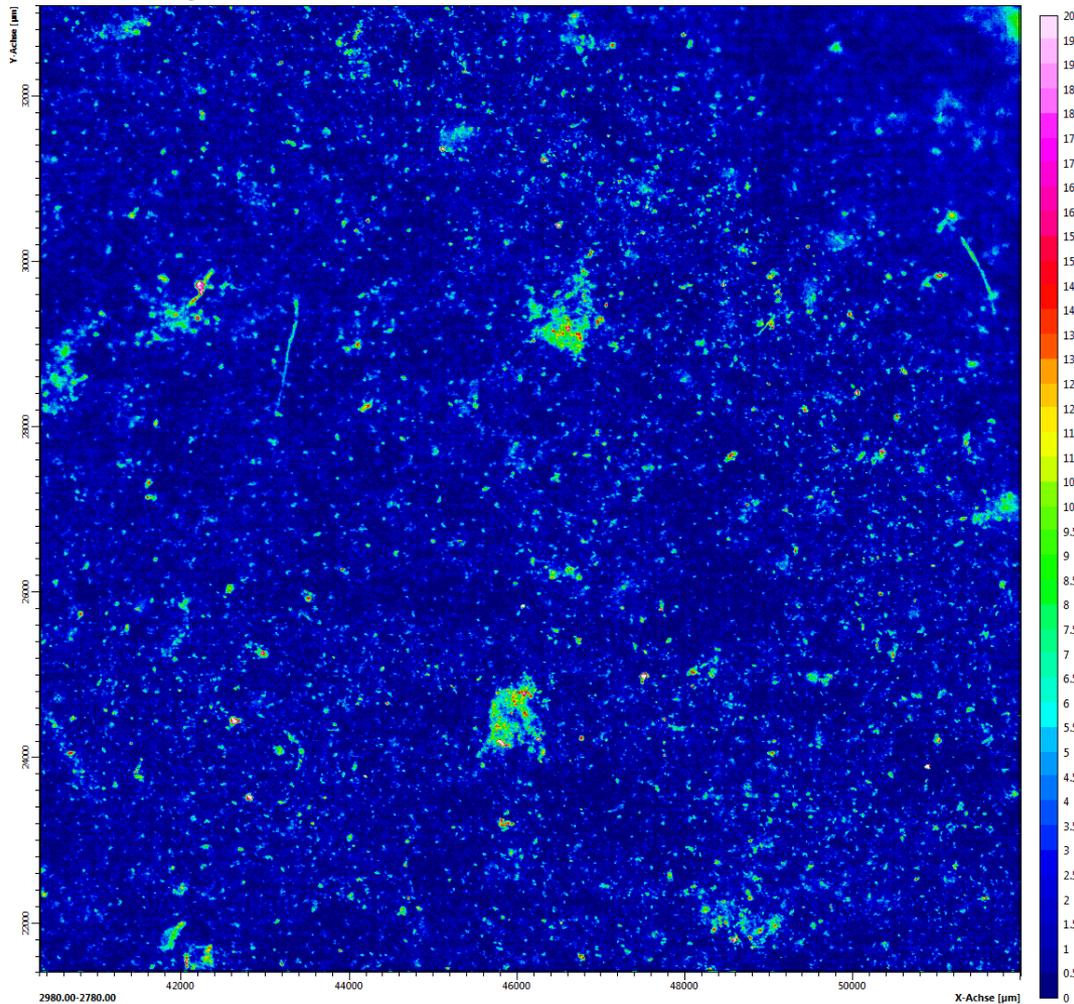
Manual analysis based on false color images

Polymer signatures



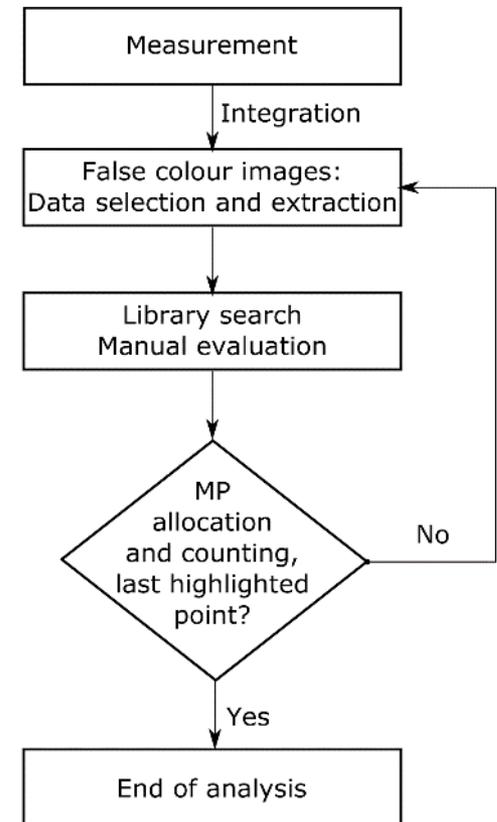
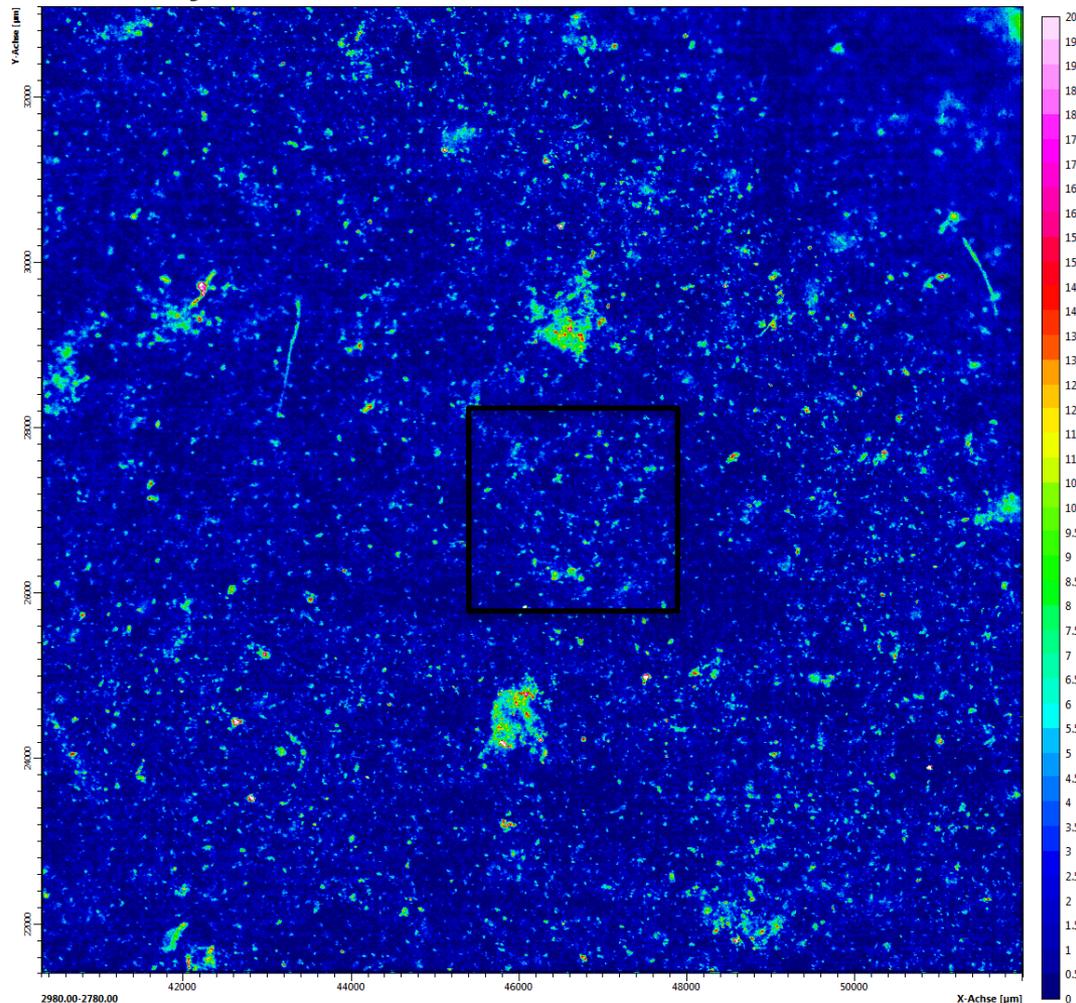
Manual analysis

➤ An easy task?



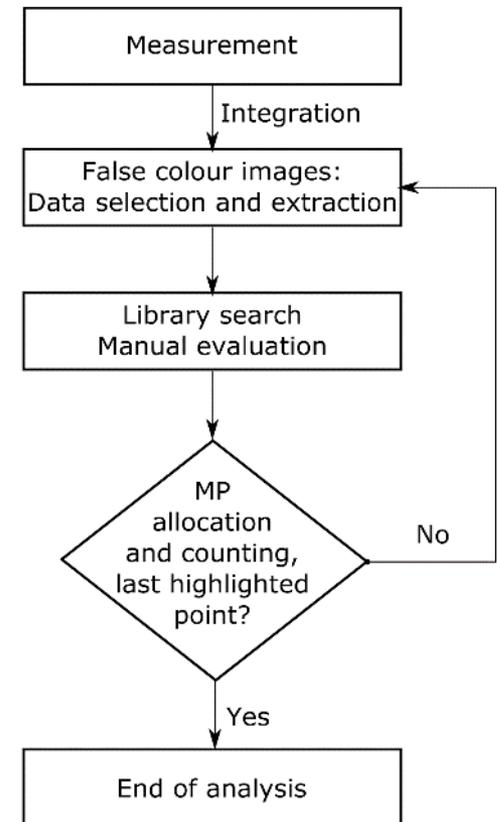
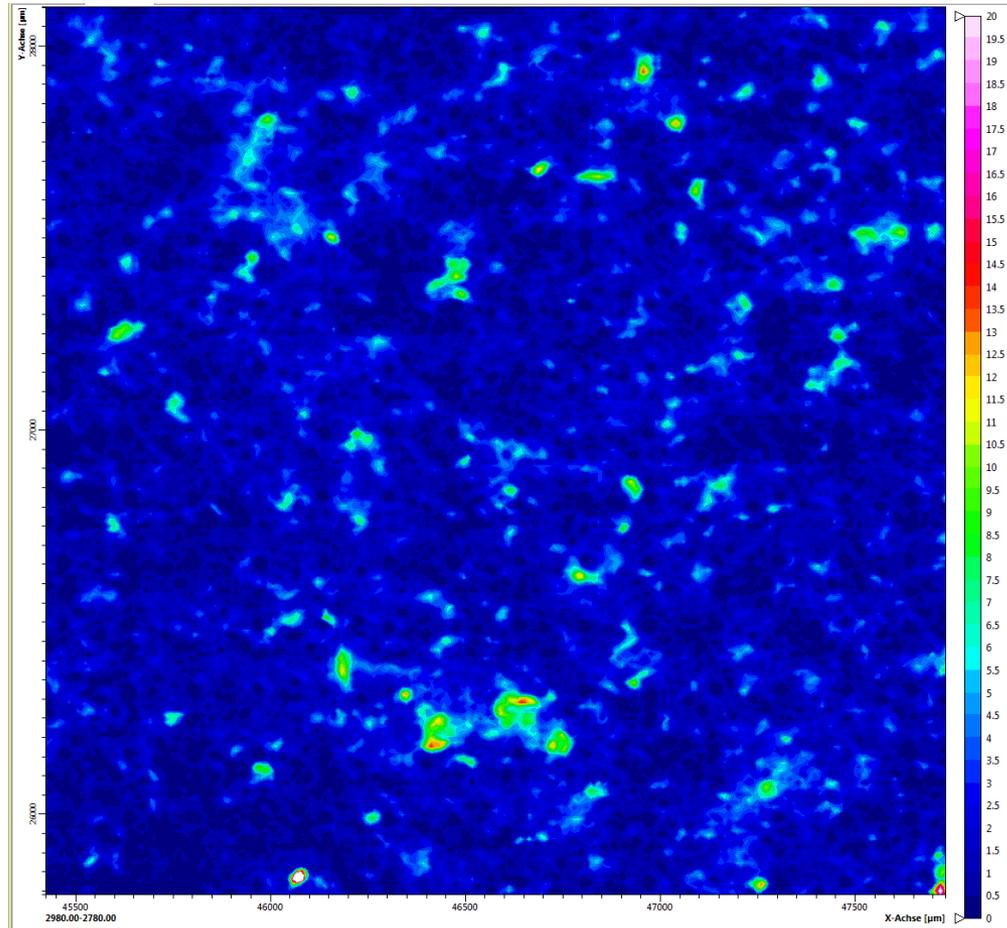
Manual analysis

➤ An easy task?



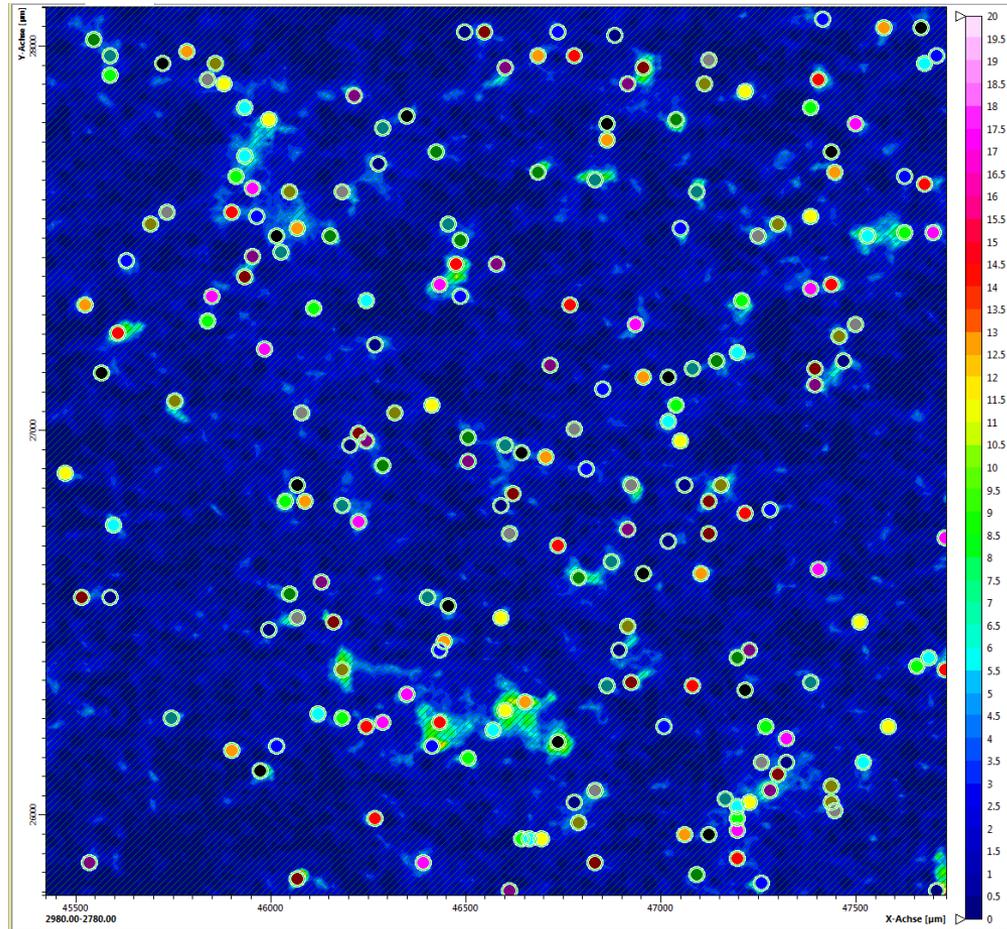
Manual analysis

➤ An easy task?



Manual analysis

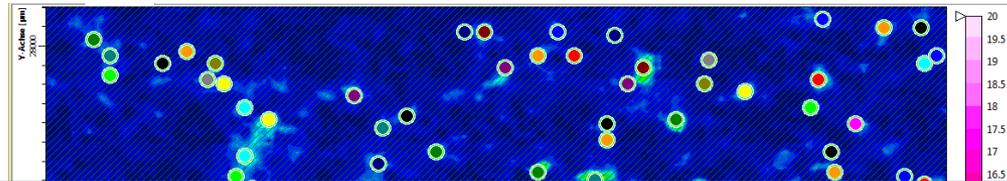
➤ An easy task?



- circa 60 seconds per particle
- Around 300 spectra per day
- Experienced operators necessary
- Worst case 2-6 weeks per sample

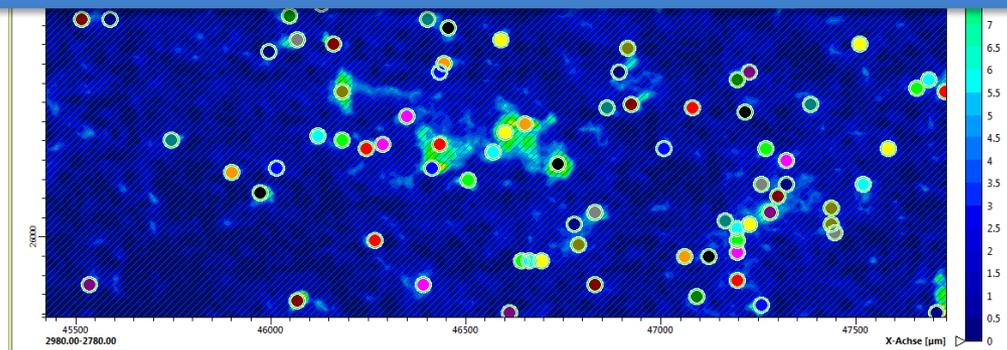
Manual analysis

➤ An easy task?



➤ circa 60 seconds per particle

No!



➤ Worst case 2-6 weeks per sample

High expenditure of time:

- Manual selection of possible particles
- Manual library search
- Size determination of the particles limited

Overall the process is prone to human bias!

Additionally a high demand of personnel requirements

High expenditure of time:

- Manual selection of possible particles

Unsuitable for standardization
of microplastic analysis

Additionally a high demand of personnel requirements

Requirements for harmonization



- Low expenditure of time
- Impartial analysis
- A minimum of personnel requirements
- Fast and reliable measurements

Requirements for harmonization



- Low expenditure of time
- Impartial analysis
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Harmonization by automation of microplastic analysis
based on FTIR imaging

Automated Analysis

Combination of two library searches with different data handling

- Correlation of the original spectrum with vector normalization
- Correlation with the 1st derivative of the original spectrum with vector normalization

Successfully automated data generation with a 3% error value

Transformation into images possible

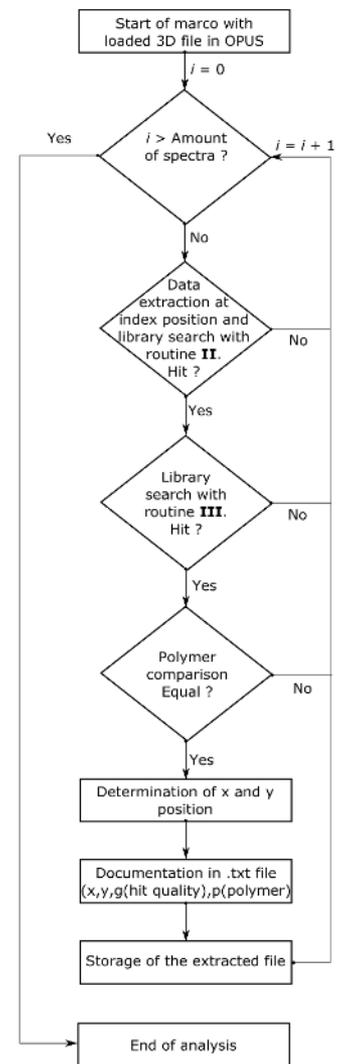


Image Analysis

Implementation of an analytical program based on Python and SimpleITK

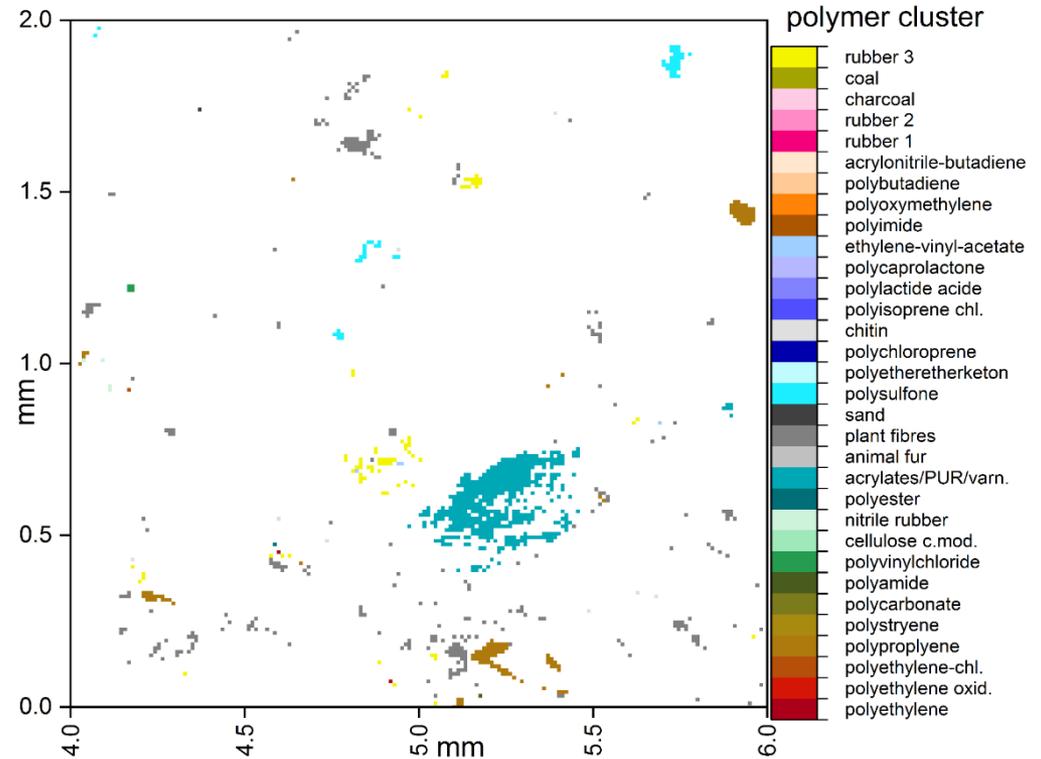


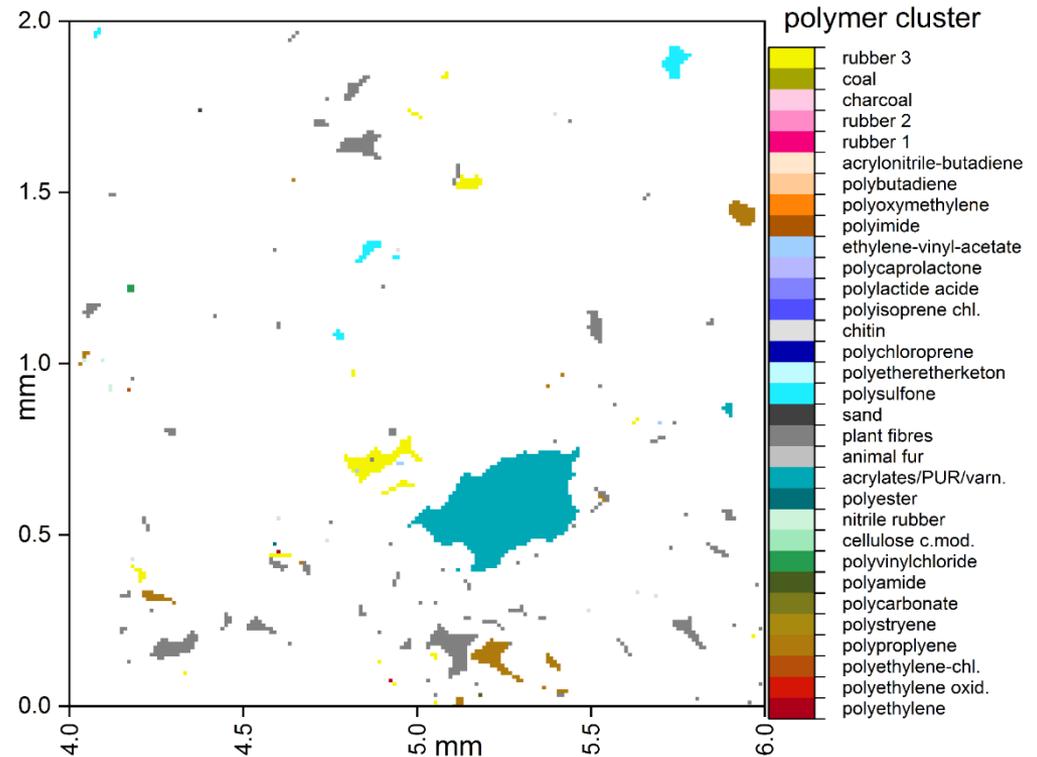
Image Analysis

Implementation of an analytical program based on Python and SimpleITK

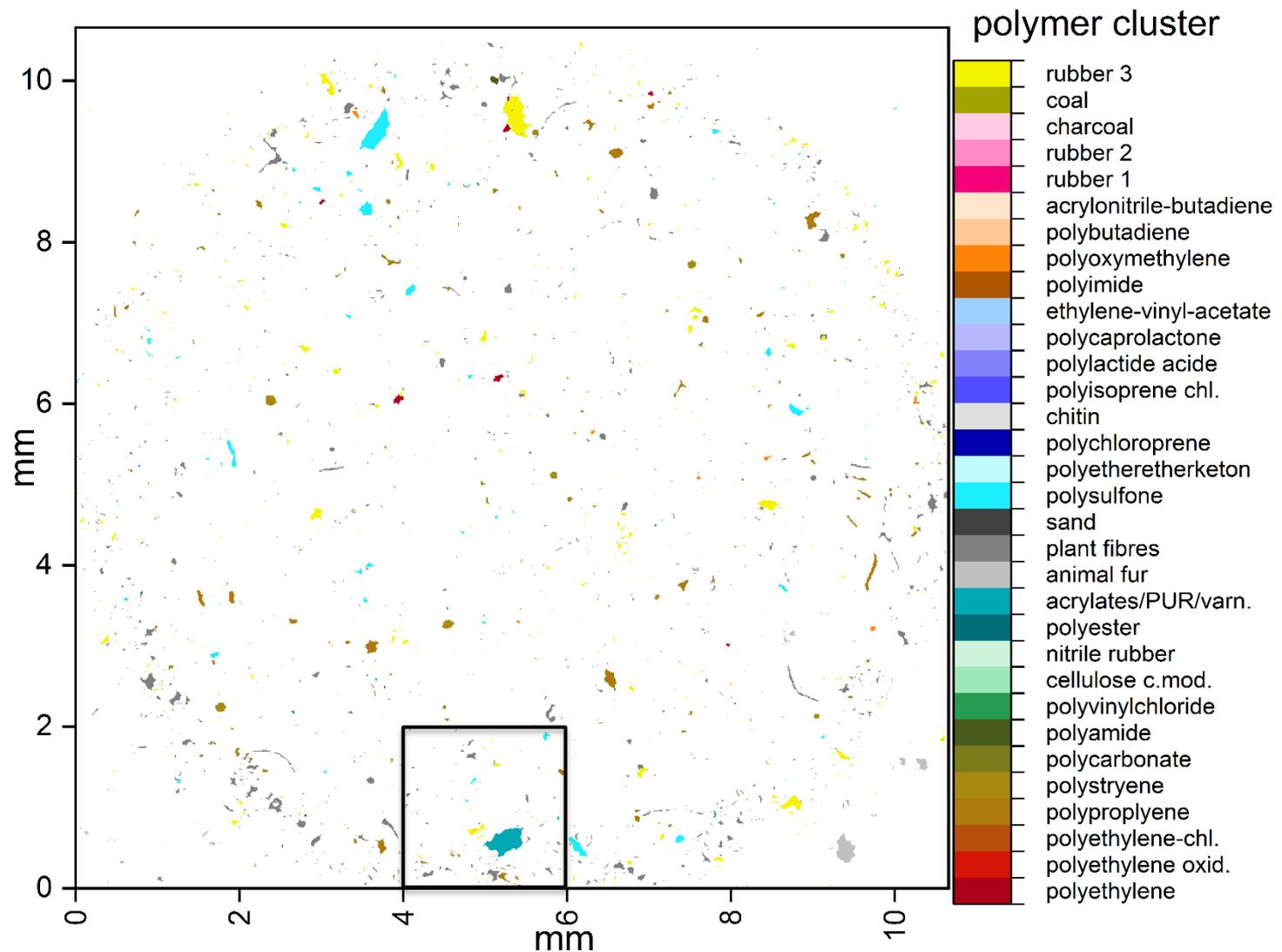
Allows determination of particle sizes

Resulting in high quality data within a short time

Sediment sample as example

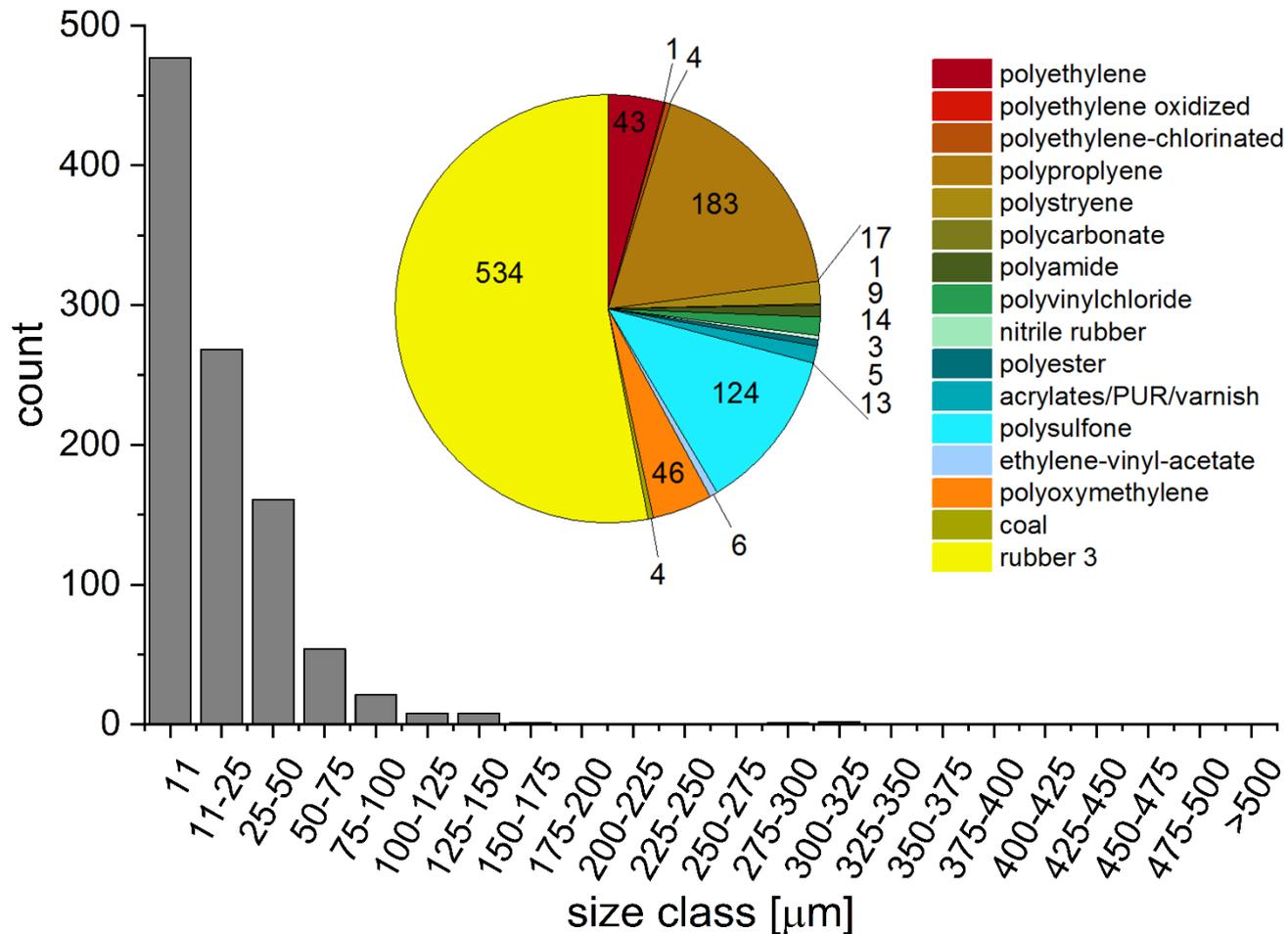


And on larger scale



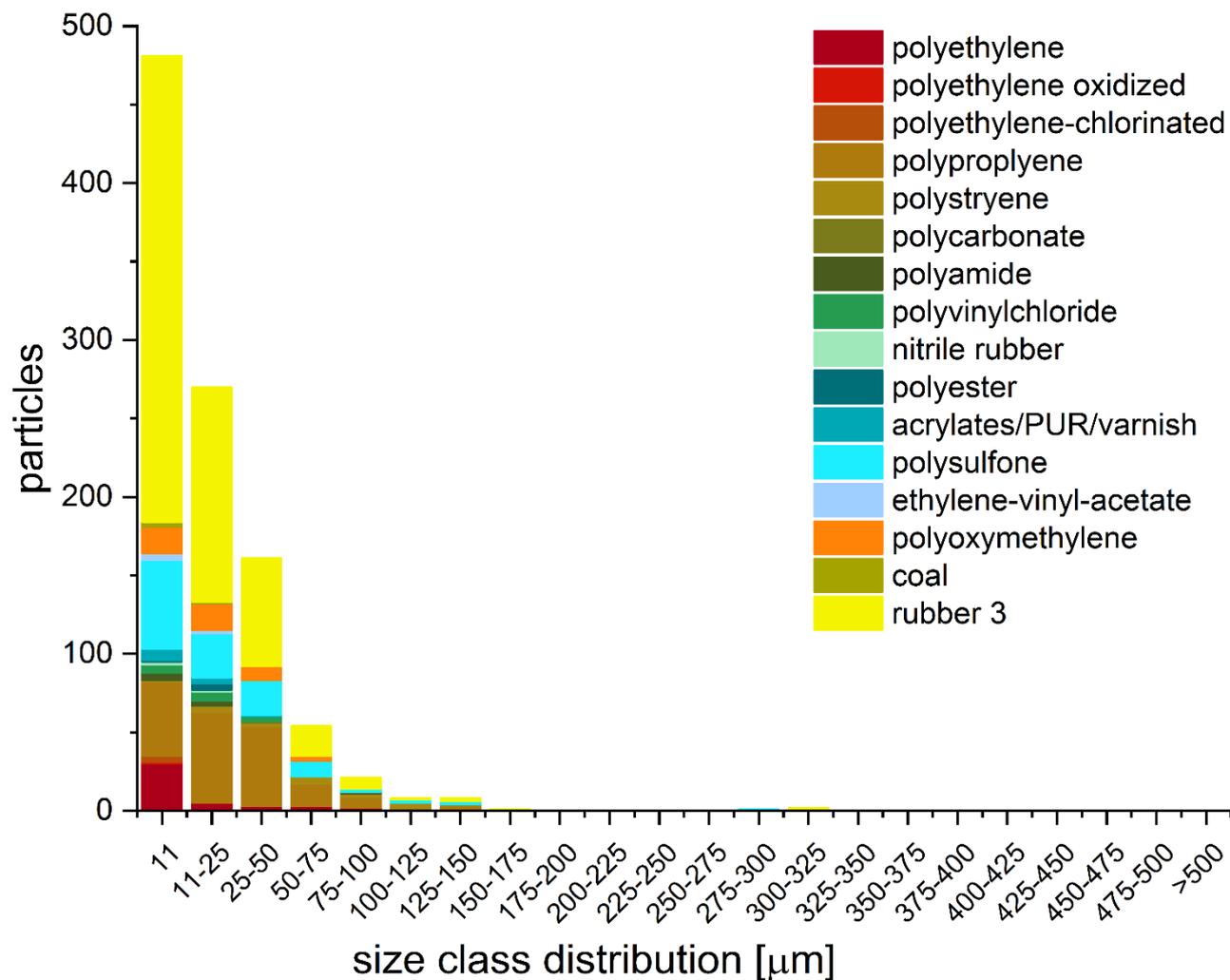
After Particle Analysis

Size distribution and polymer numbers accessible



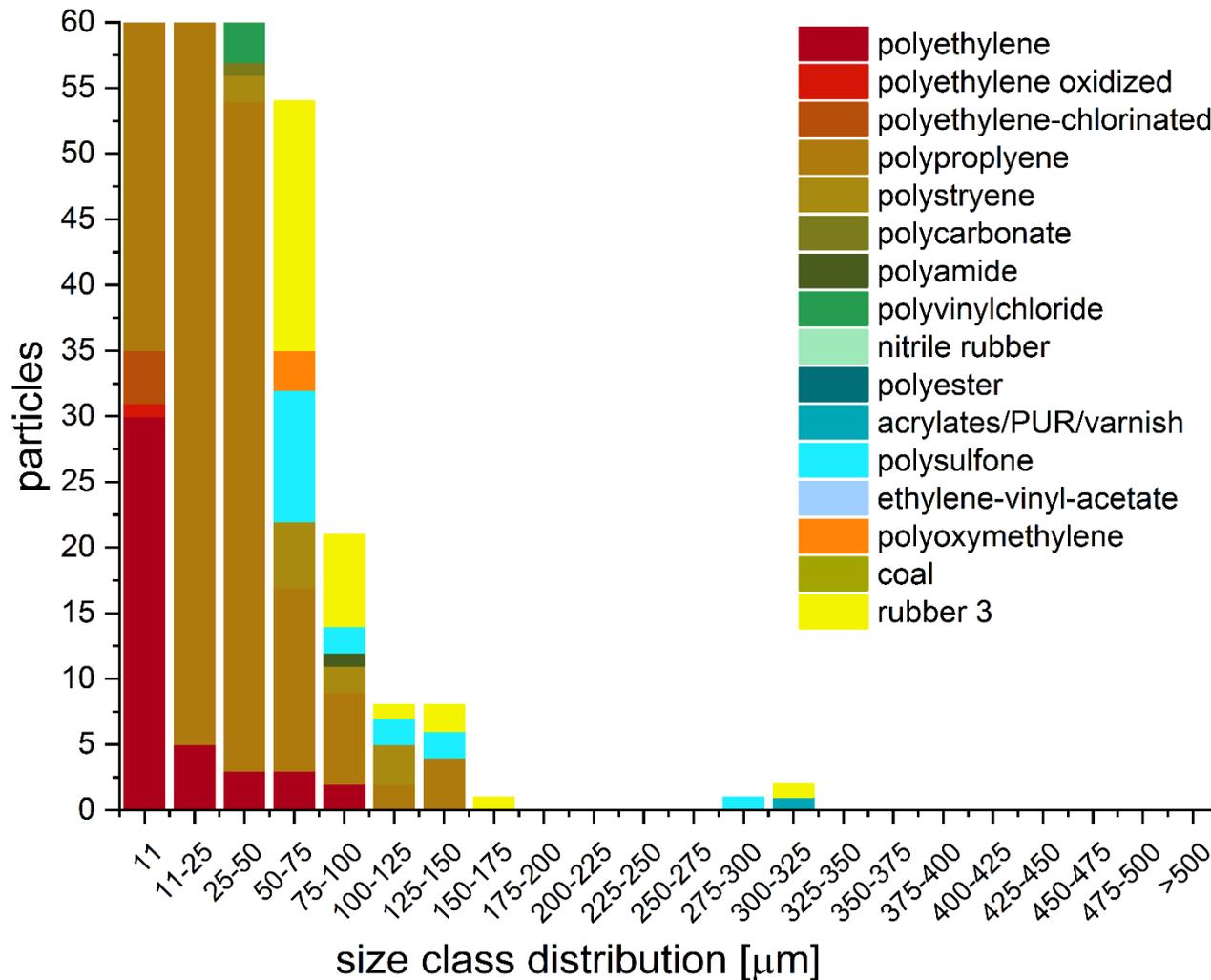
After Particle Analysis

Or even combined:



After Particle Analysis

And as zoom in:

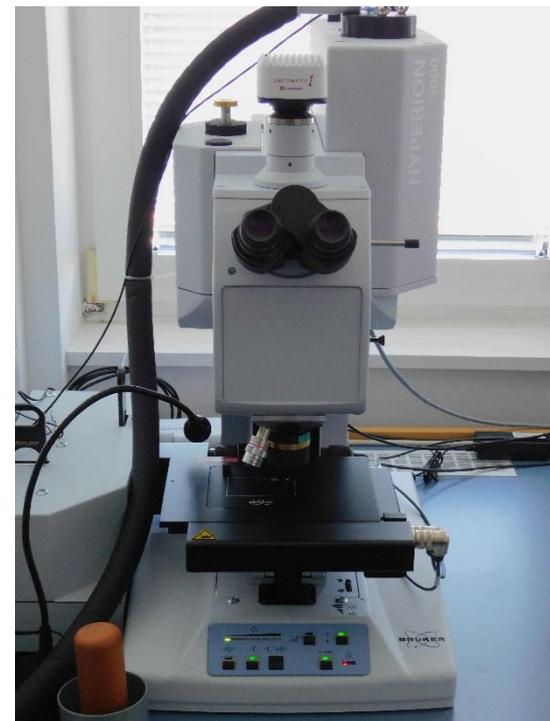
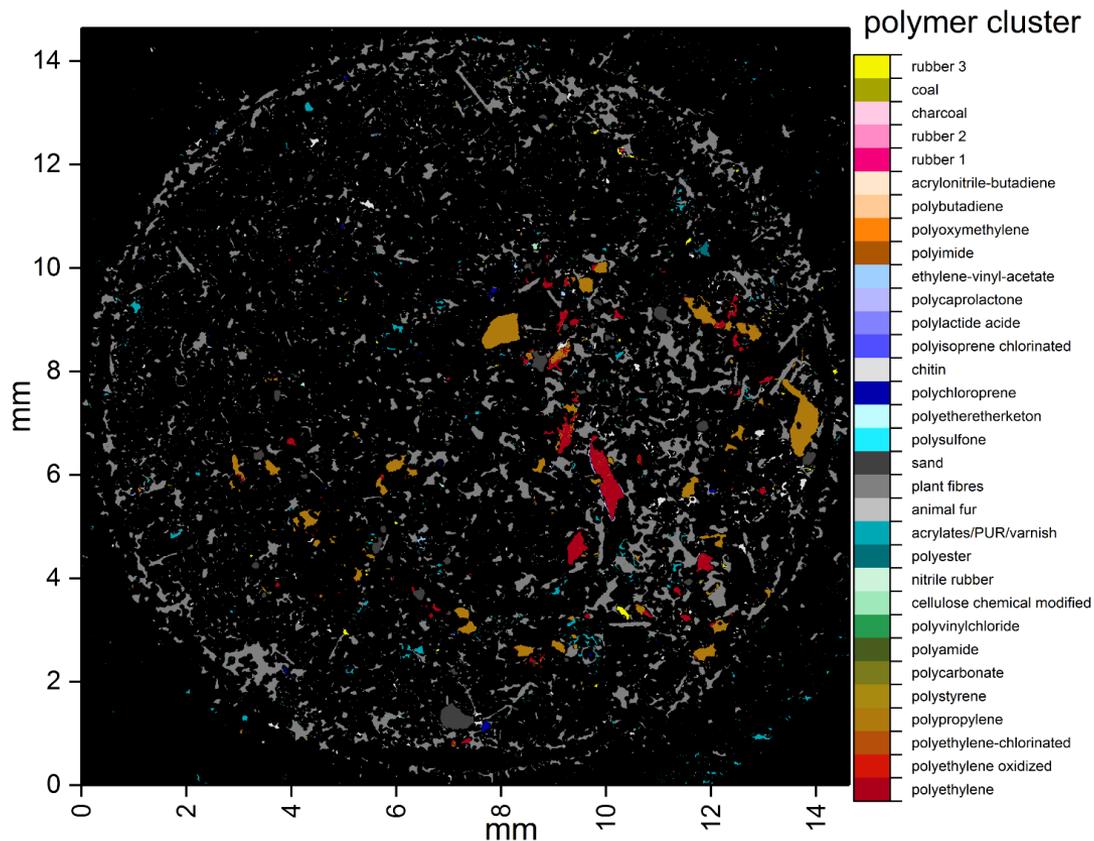


Towards Harmonization of Analysis



FTIR microscopy and imaging:

Automated analysis and reference database published via Open Access

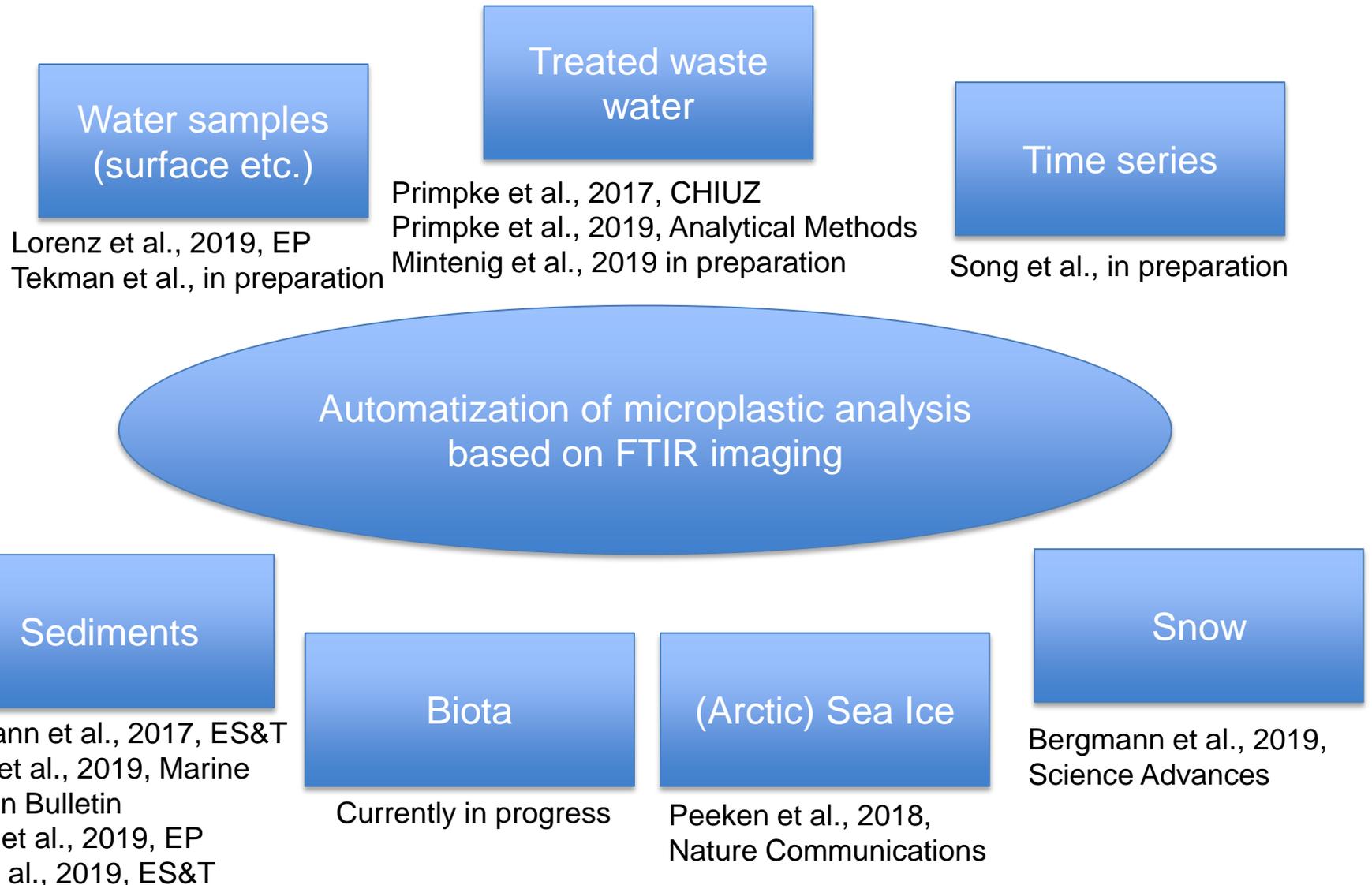


1. Primpke, S. et. al., Analytical and Bioanalytical Chemistry 2018, 410, (21), 5131-5141

Automatization of microplastic analysis
based on FTIR imaging

- Data analysis independent from human bias via automated analysis
- Identification and Quantification of MP already within this process
- Time saving due to parallelization
- High comparability of results!

Harmonization!



Standardization!

Treated waste
water

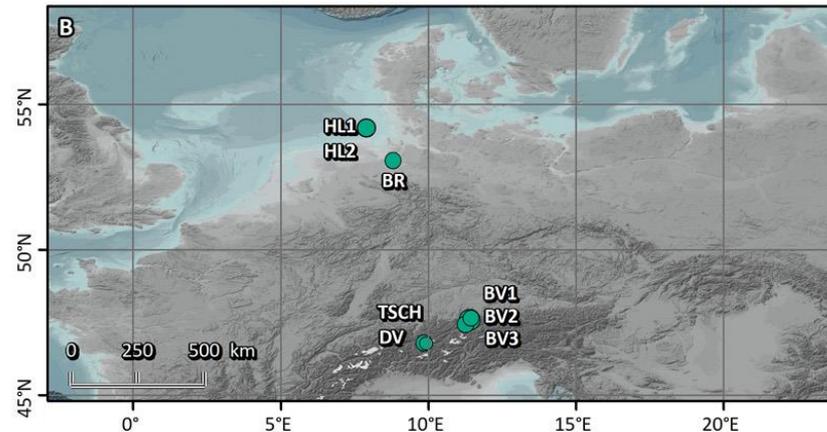
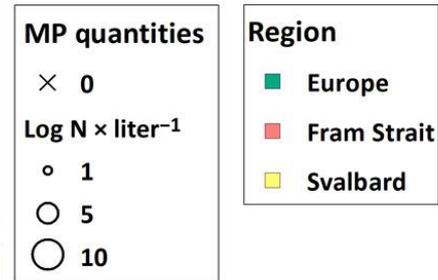
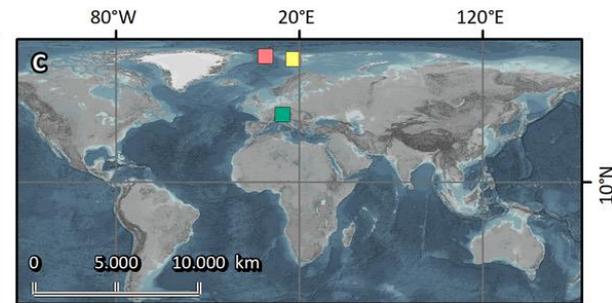
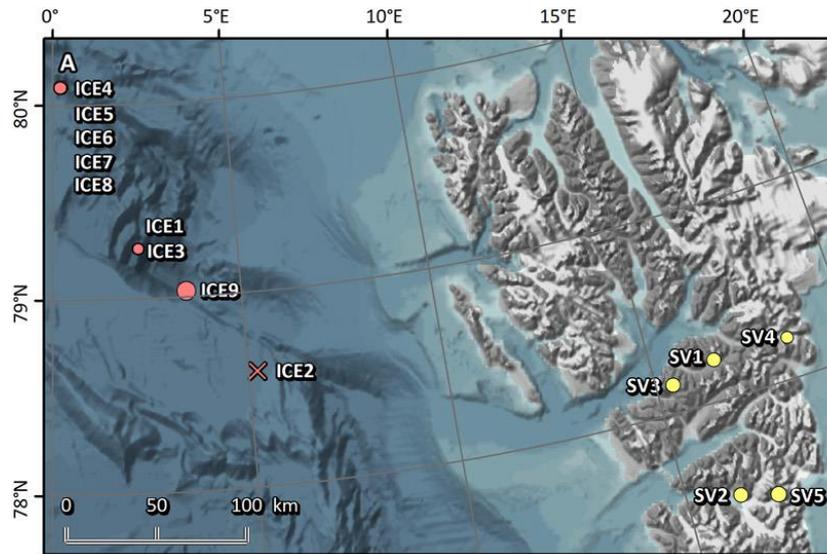
Primpke et al., 2019, Analytical Methods

Automatization of microplastic analysis
based on FTIR imaging

Snow

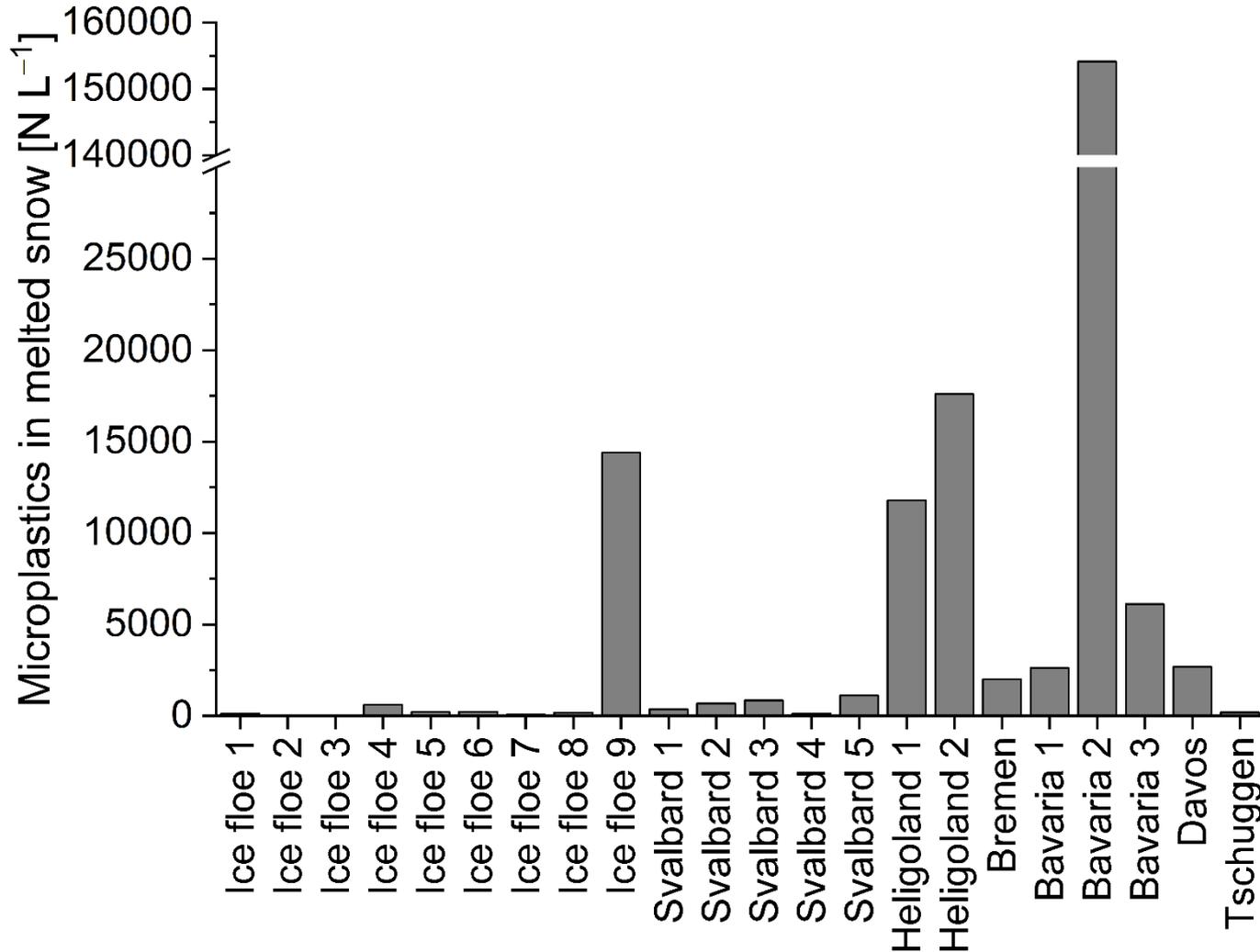
Bergmann et al., 2019,
Science Advances

Microplastics in Snow



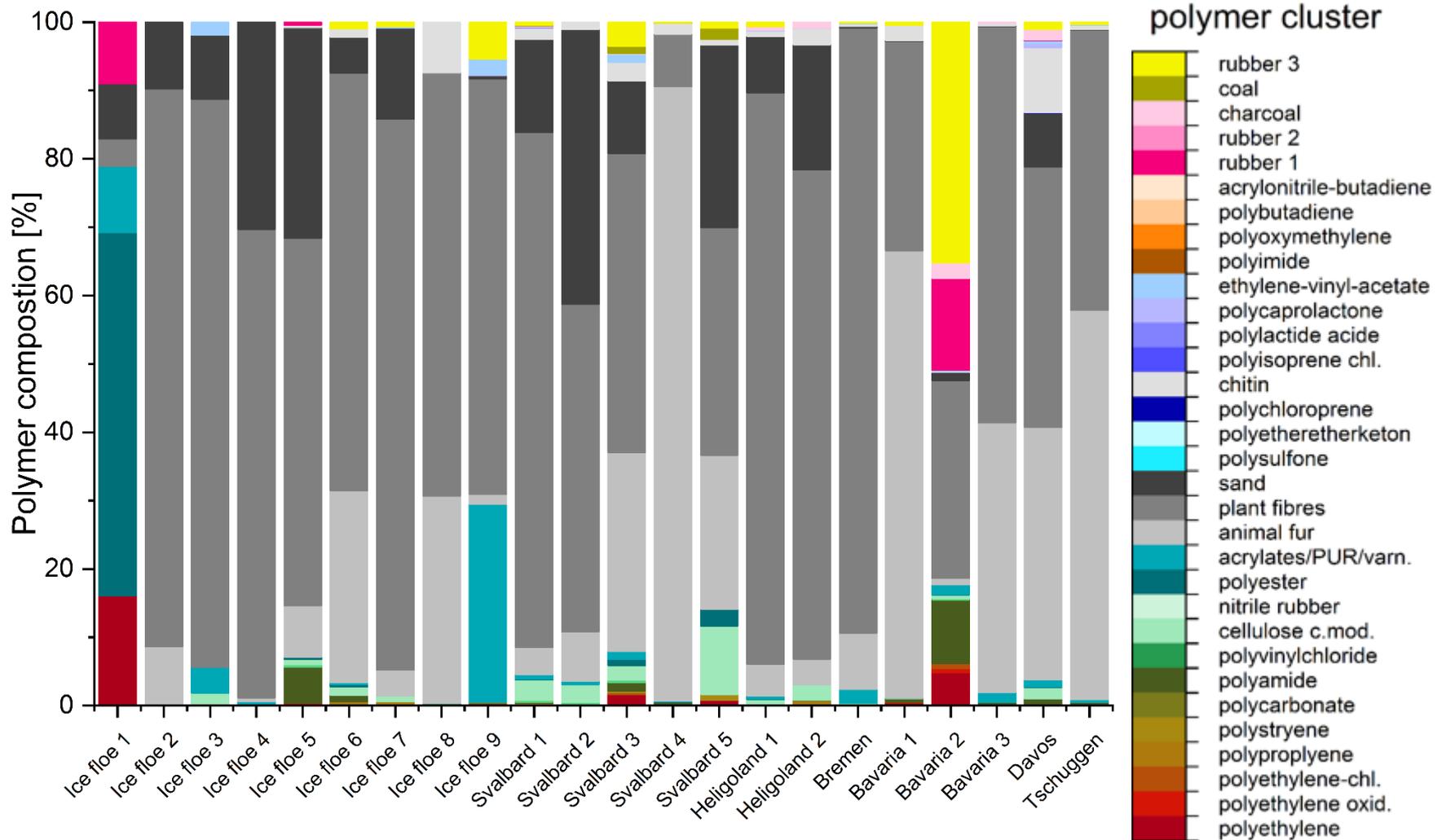
1. Bergmann, M., Mützel, S. et al., Science Advances 5, eaax1157.

Microplastics in Snow



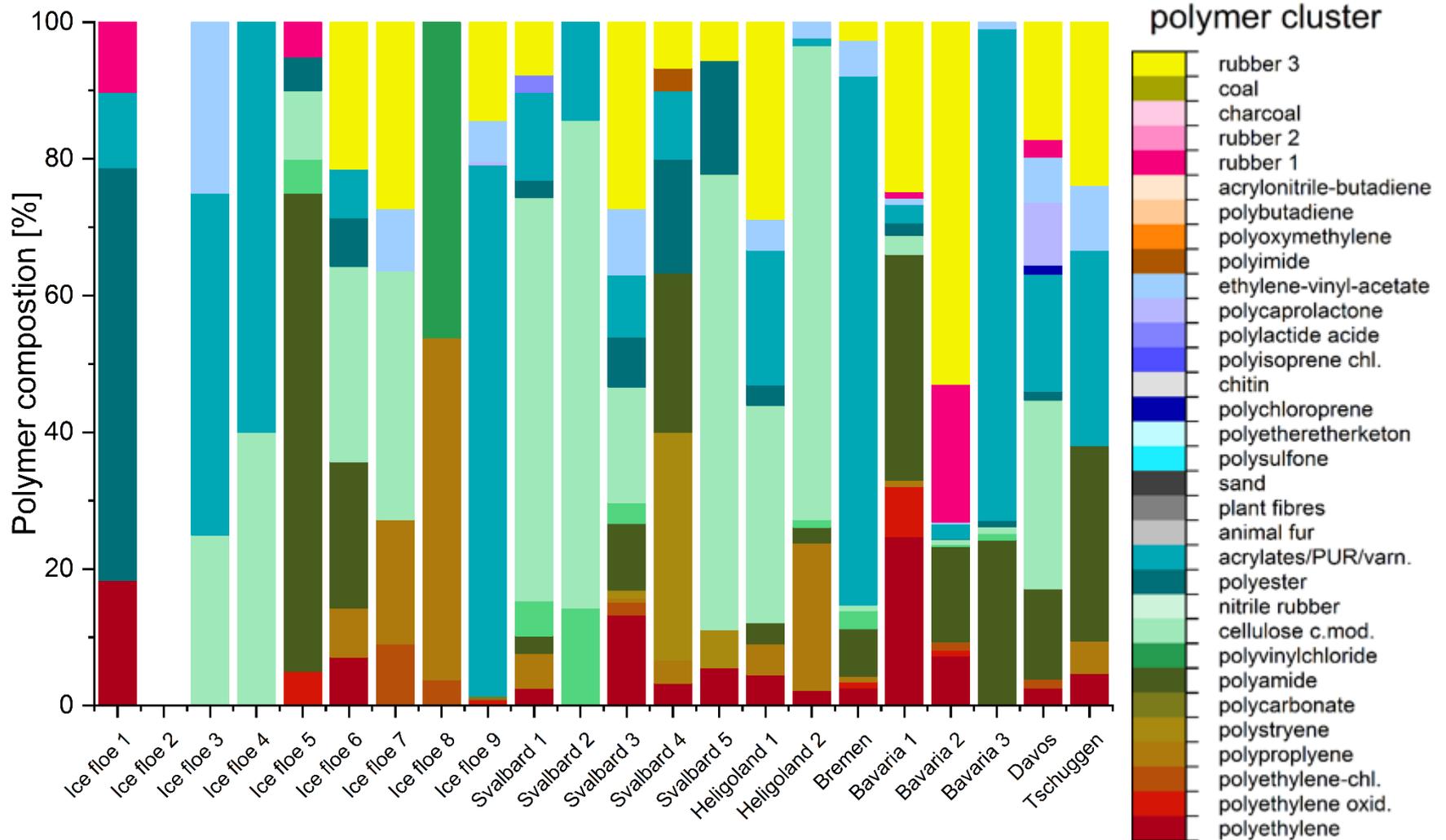
1. Bergmann, M., Mützel, S. et al., Science Advances 5, eaax1157.

Identified Particles in Snow



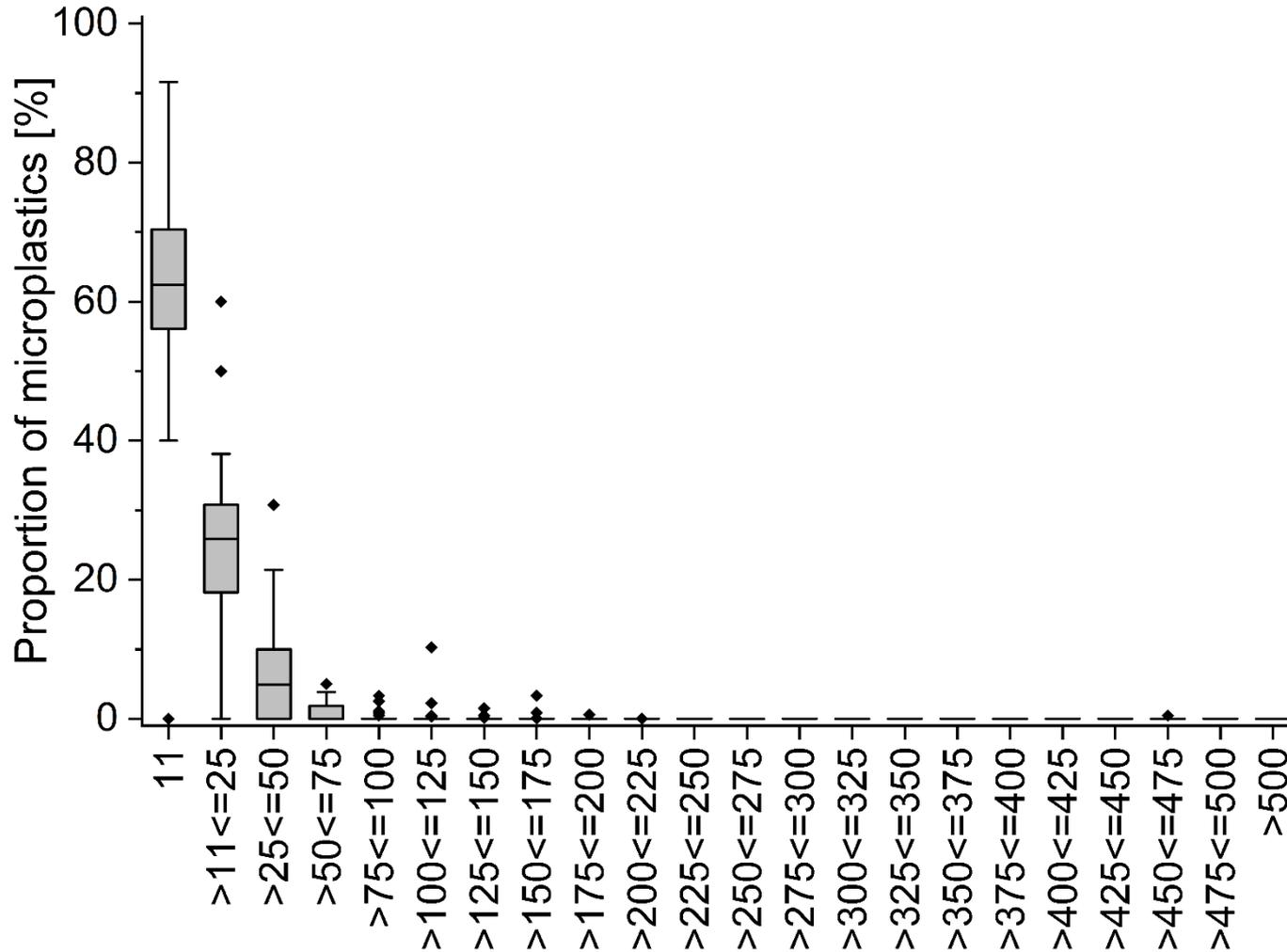
1. Bergmann, M., Mützel, S. et al., Science Advances 5, eaax1157.

Microplastics in Snow



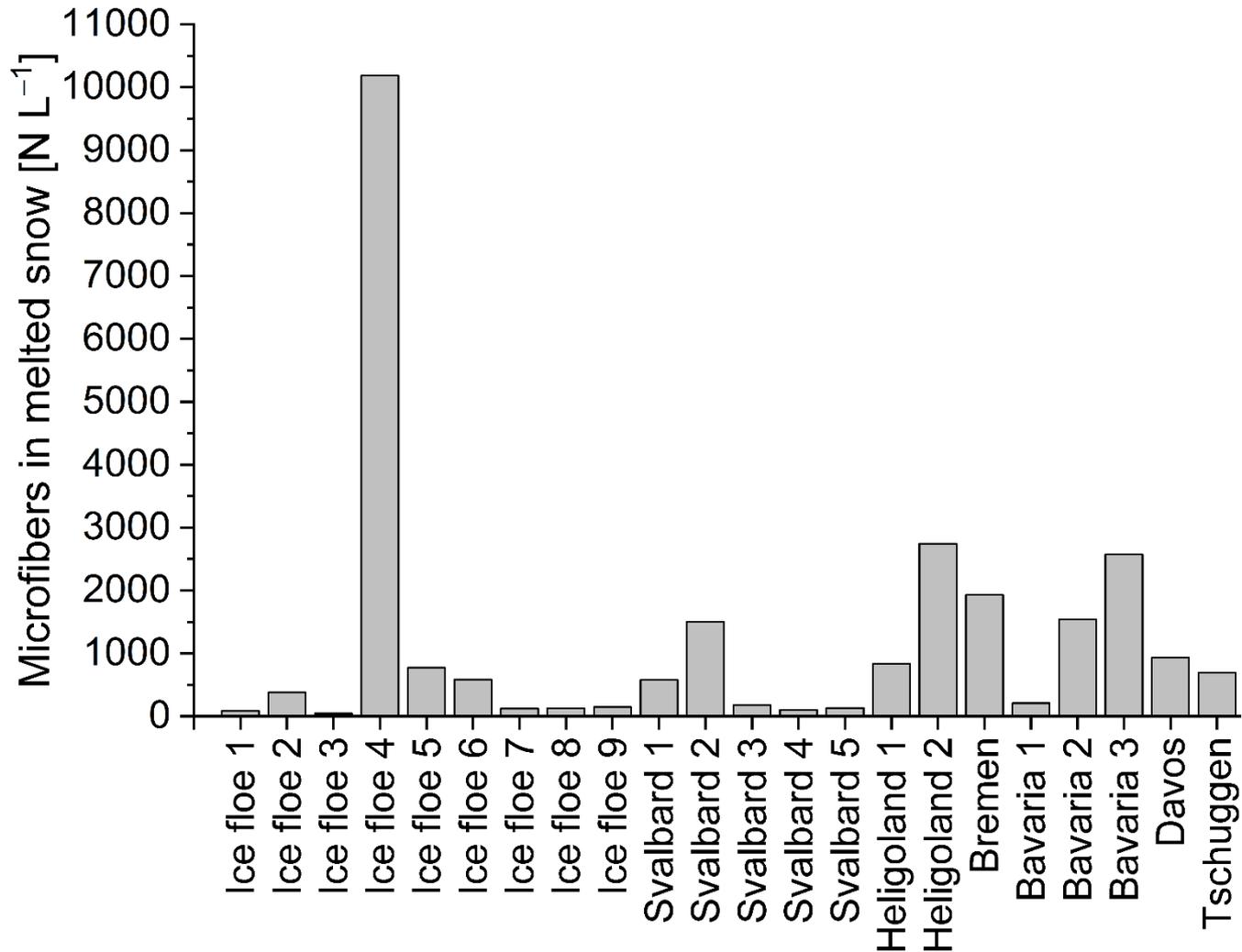
1. Bergmann, M., Mützel, S. et al., Science Advances 5, eaax1157.

Sizes of Microplastics in Snow



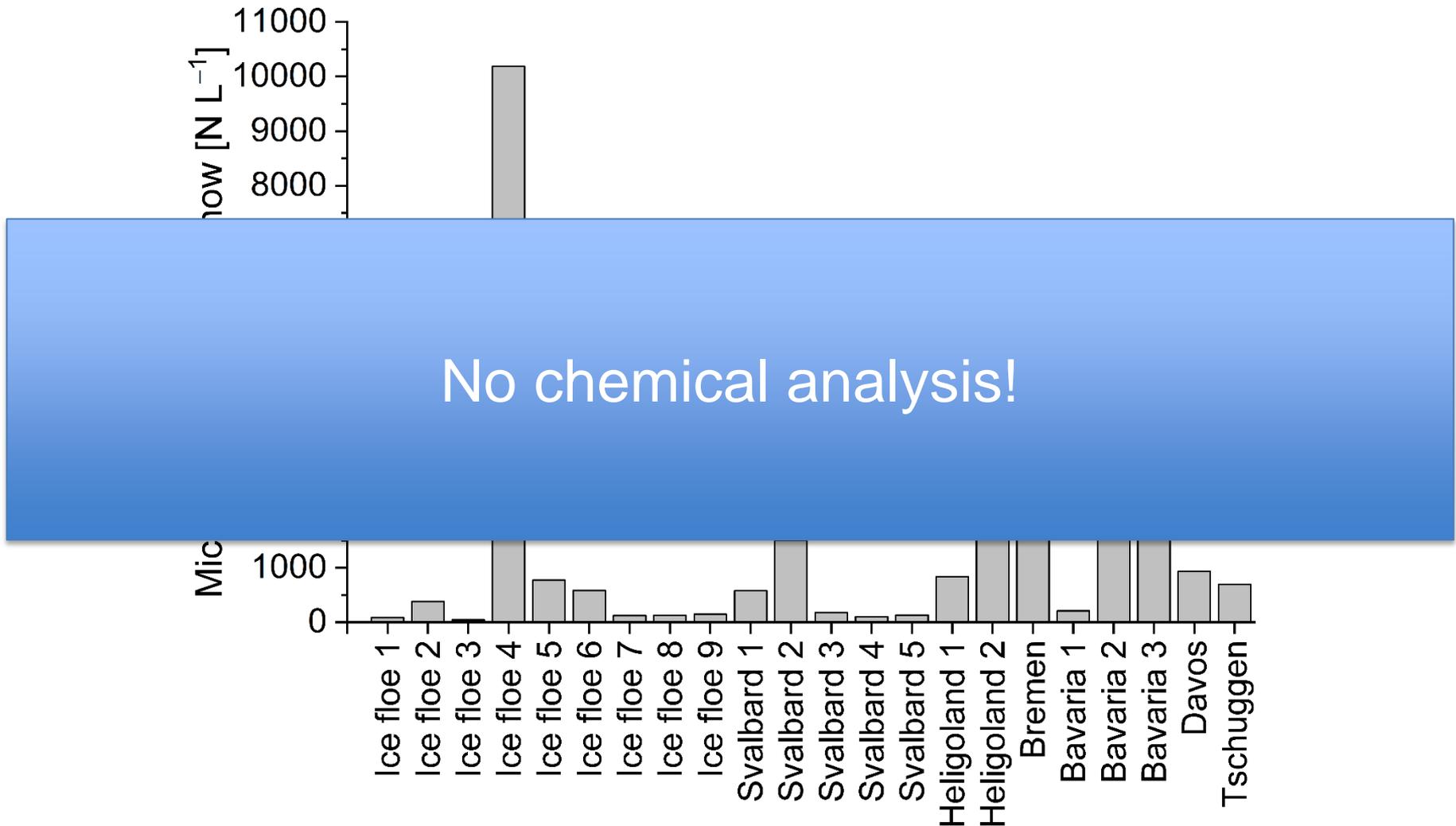
1. Bergmann, M., Mützel, S. et al., Science Advances 5, eaax1157.

Microfibers in Snow



1. Bergmann, M., Mützel, S. et al., Science Advances 5, eaax1157.

Microfibers in Snow

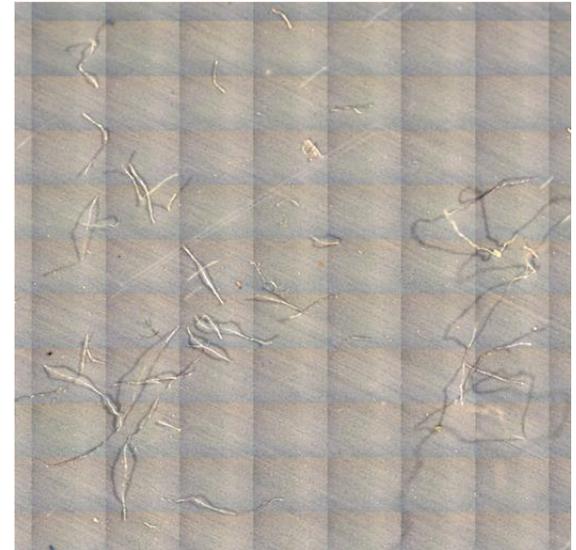


1. Bergmann, M., Mützel, S. et al., Science Advances 5, eaax1157.

Chemical analysis for fibers

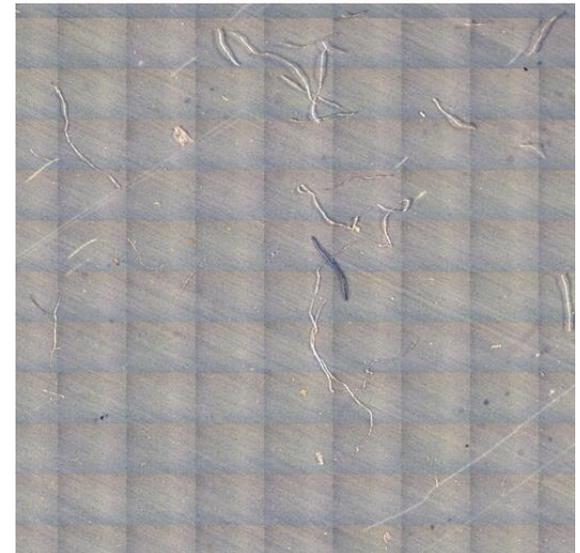
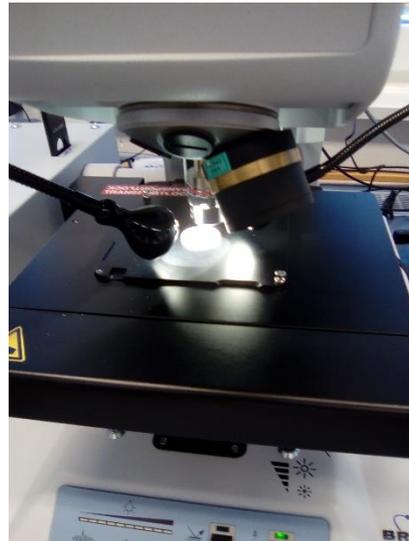
➤ Problem:

Fibers are nearby
never completely in
the focal plane

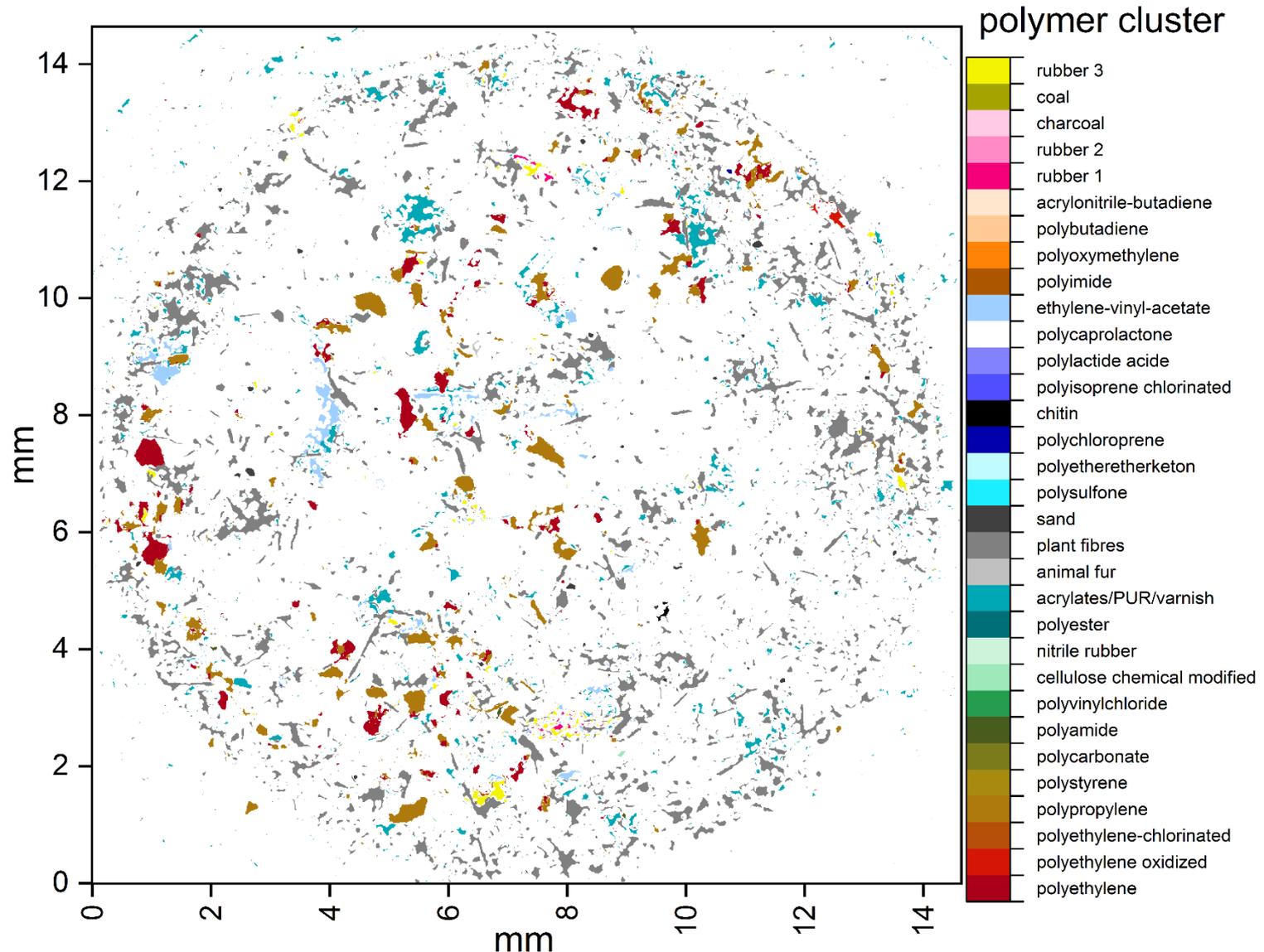


➤ Solution:

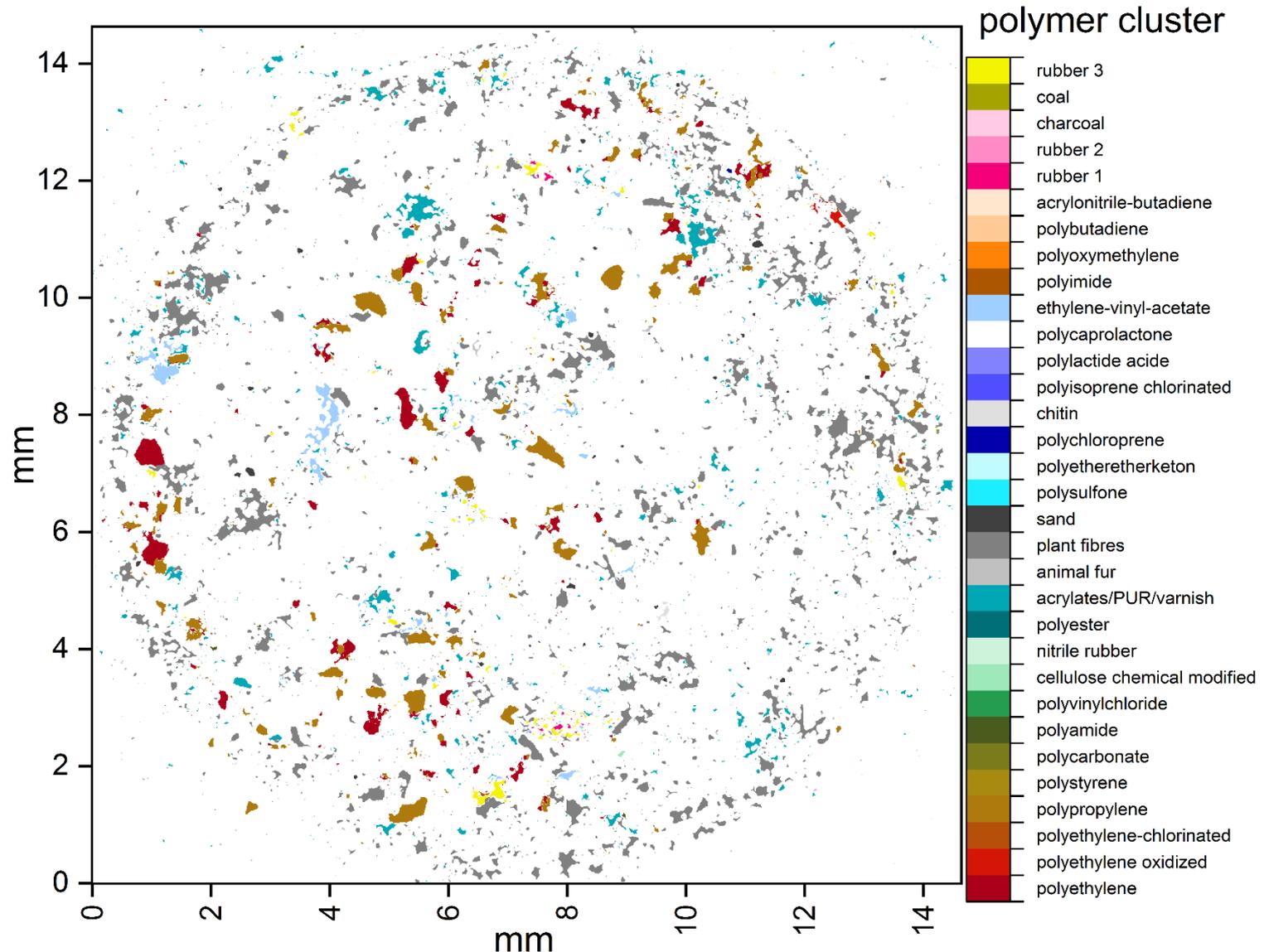
Covering the
sample with an IR
suitable window



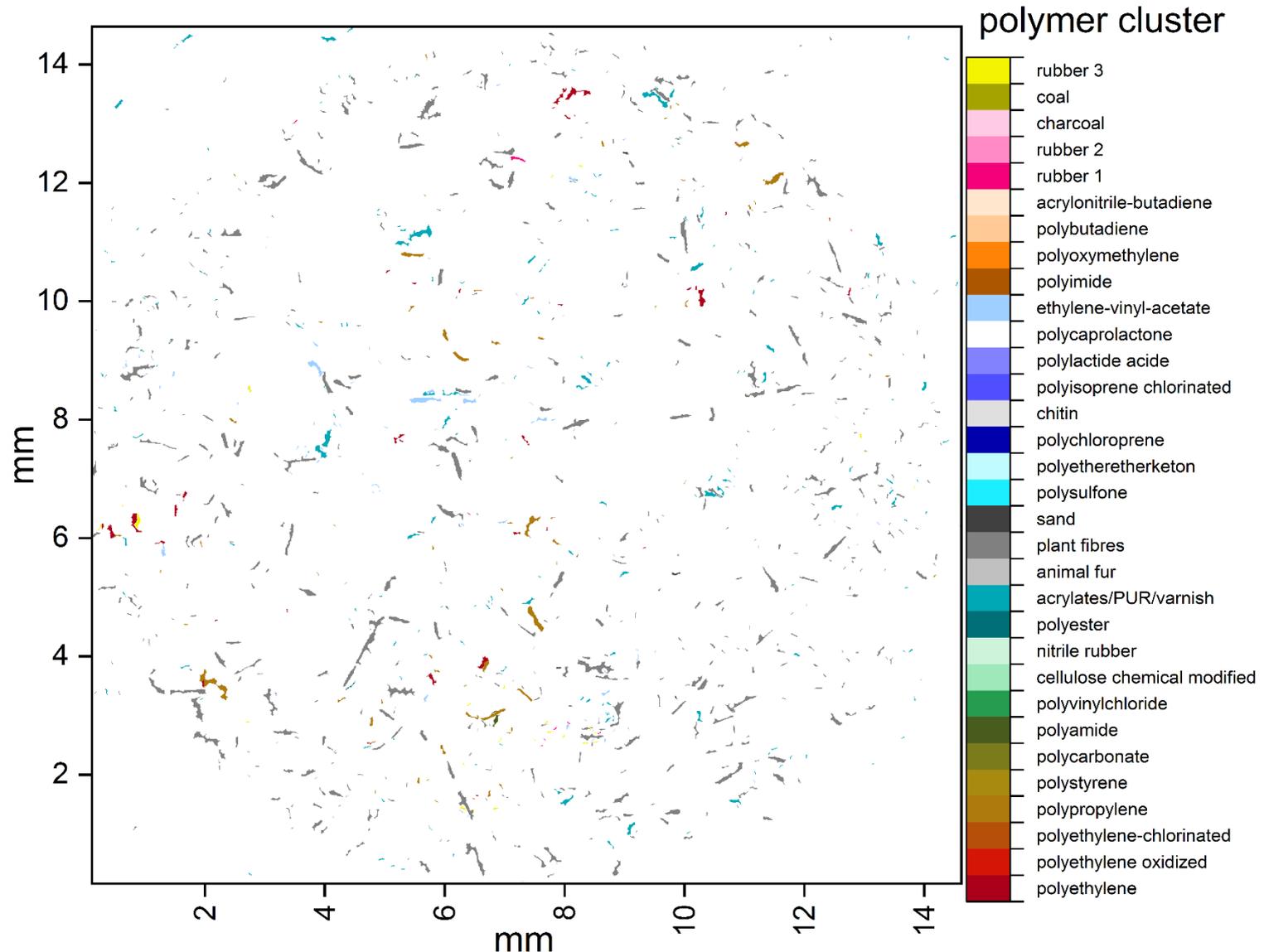
Chemical analysis for fibers



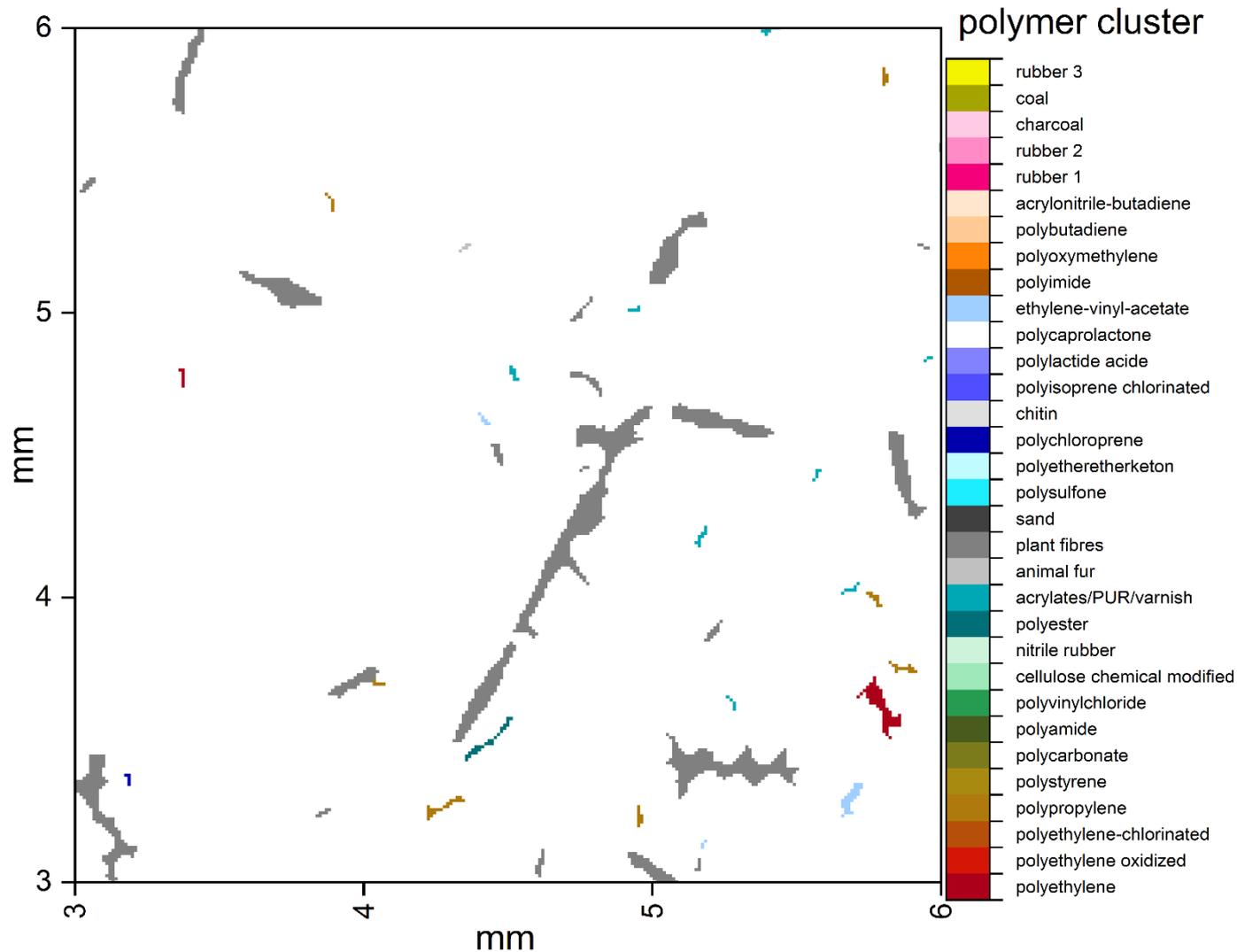
Particles



Fibers

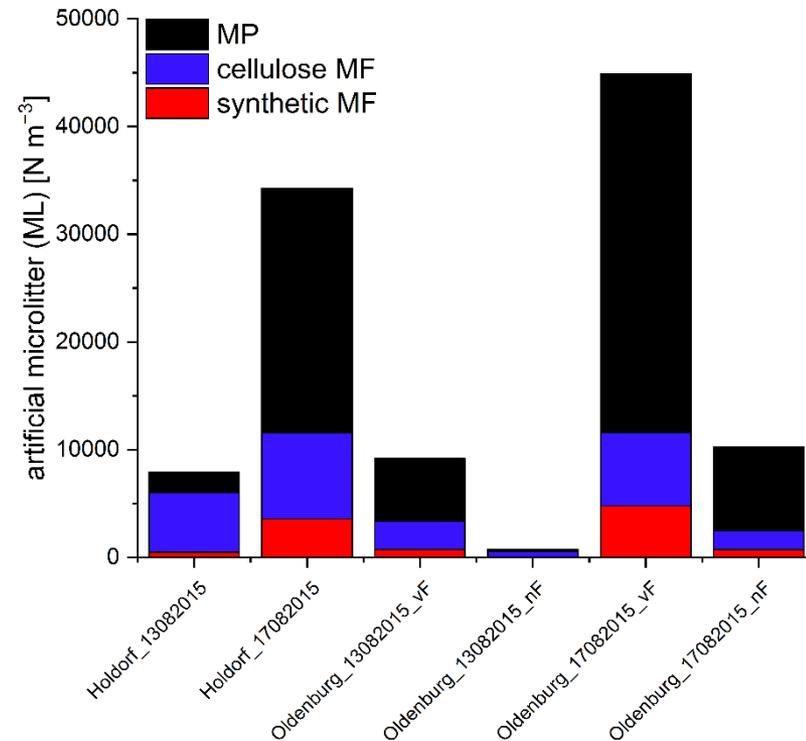


Fibers in details



Fibers

- MP dominated most of the samples
- Cellulosic fibers dominated
- MP was removed by ~ 86% during filtration
- Synthetic MF was removed by ~ 89% during filtration
- Cellulosic MF was removed by ~ 78% during filtration



Towards Harmonization of Analysis



siMPle: Standardized Identification of MicroPLastics in the Environment

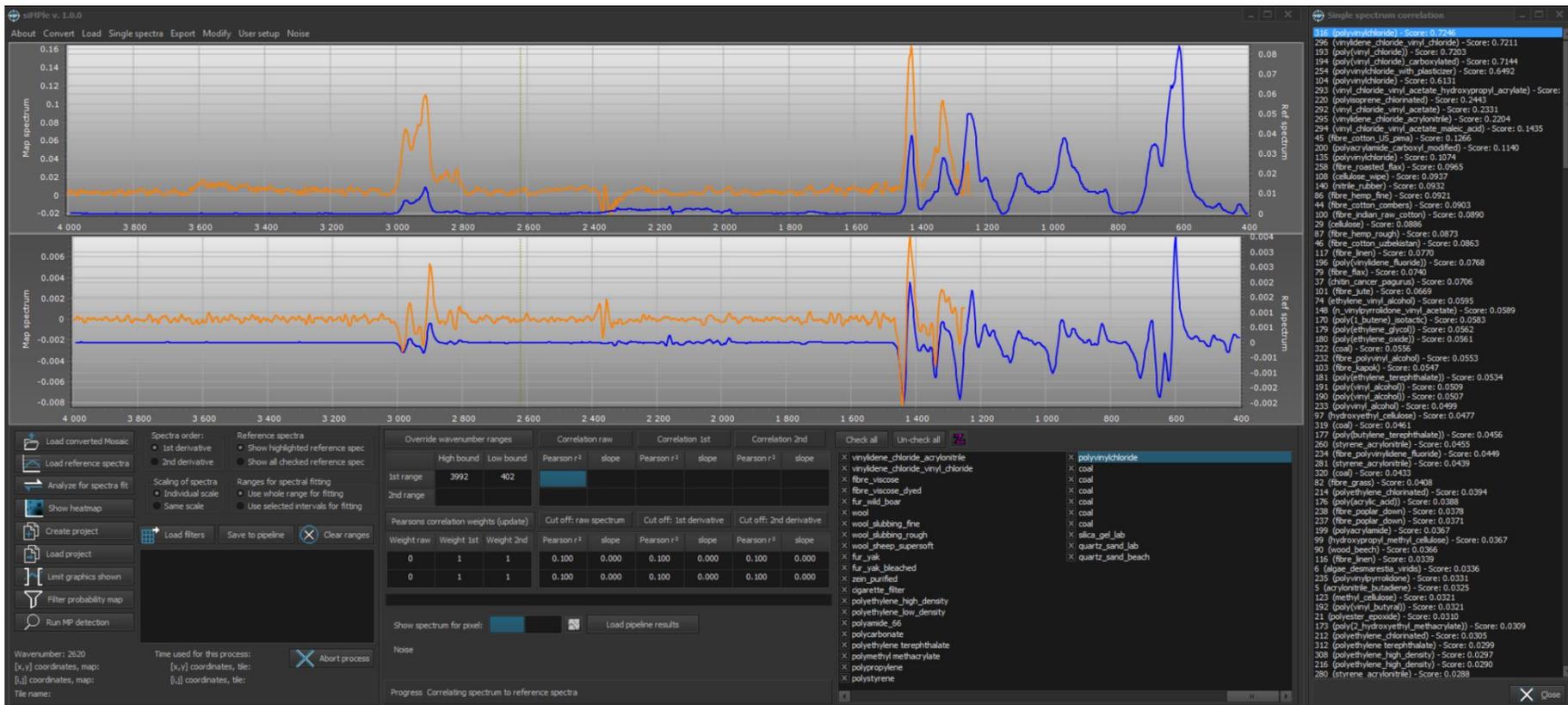


**Systematic Identification of
MicroPLastics in the Environment**

Developed by Aalborg University, Denmark
and Alfred Wegener Institute, Germany

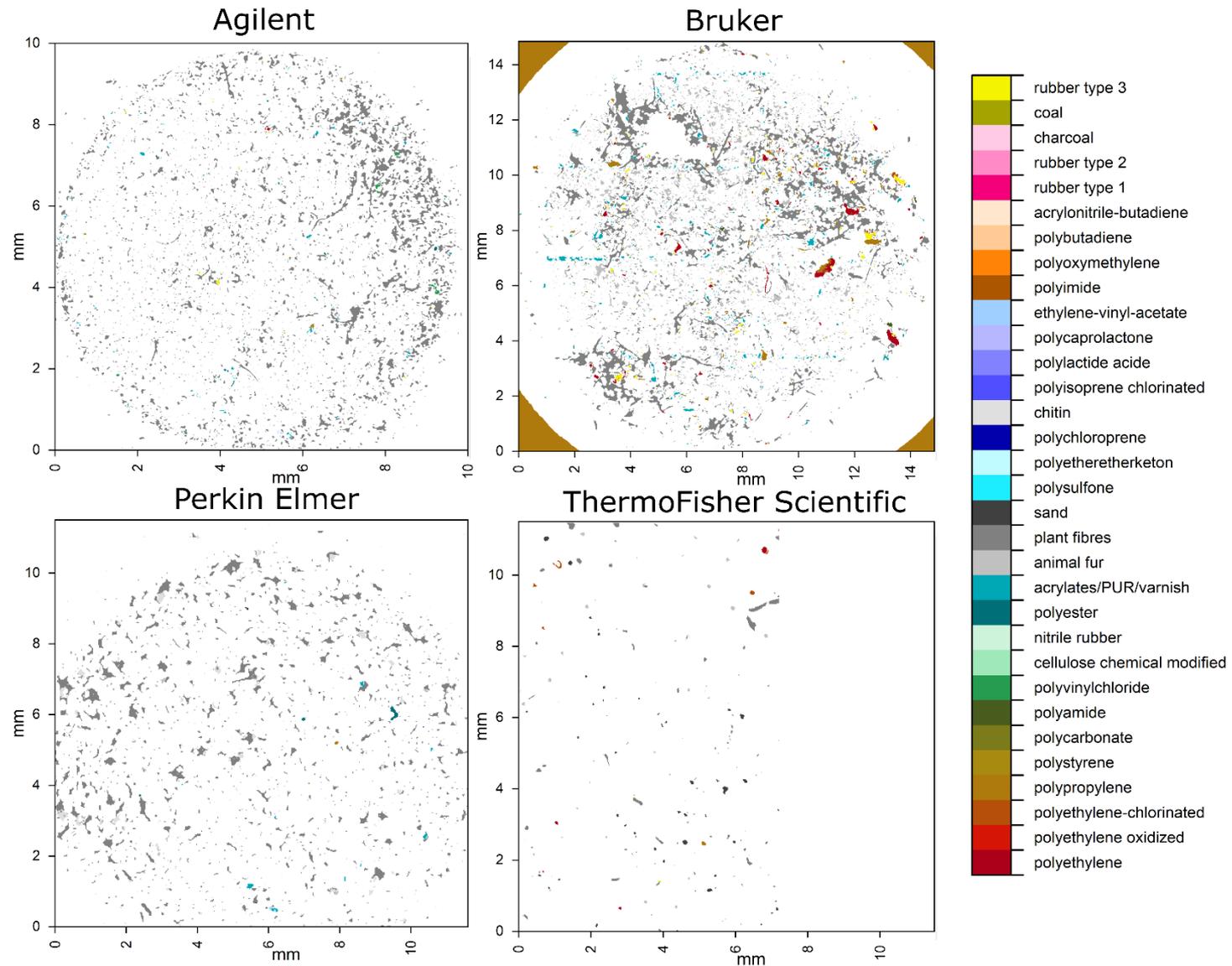
Available on www.simple-plastics.eu

Towards Harmonization of Analysis



Available on www.simple-plastics.eu

Example: Analysis of Water Samples



- **Low expenditure of time:**
 - Data analysis time currently reduced from 24 hours to 6 hours by siMPLe for the automated analysis.
 - Depending on FTIR system 1 hours or less of manual labor per sample

- **Impartial analysis**
 - Evaluation within a fixed confidential interval

- **Minimum of personnel requirements**
 - One person can perform and analyze several samples in parallel
 - Data analysis can be parallelized

- **Fast and reliable measurements**
 - Measurement time 4 hours to 18 hours for the same region (14 × 14 mm) depending on lenses used.

Harmonization by automation of microplastic analysis based on FTIR imaging

- Low expenditure of time
- Impartial analysis
- Minimum of personnel requirements
- Fast and reliable measurements

Harmonization by automation of microplastic analysis based on FTIR imaging

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Nick Mackay-Roberts
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Current Master students: Laura Stutzinger, Lorenz Reisel
and Annika Fehres
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Wirzberger, Julia Guetermann, Birte Beyer, Mathilde
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Questions ?

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