
CHALLENGES IN MICROPLASTICS IDENTIFICATION IS ONE DETECTION METHOD ENOUGH?

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Motivation:

- MPs: how big is that problem?

Sampling methods

Sample preparations

Analysis

- Need to compare the results

Harmonization of the methods



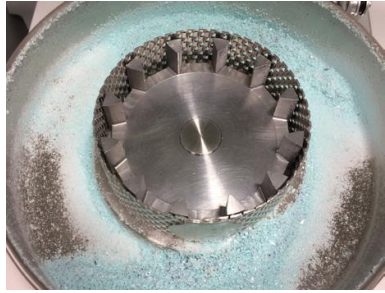
METHODS:

Optical Microscopy

Spectroscopic: (Raman, FTIR)

Chemical Extraction: TED-GC-MS, SEC

Reference material

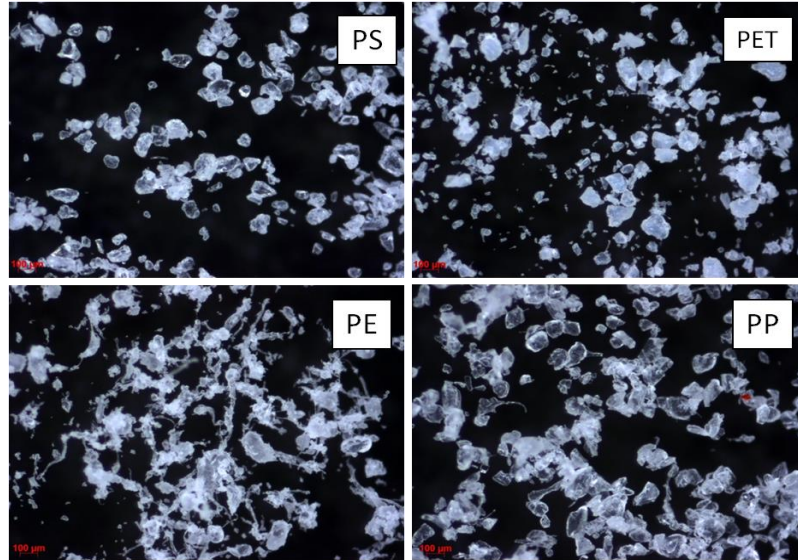


MP after cryo-milling

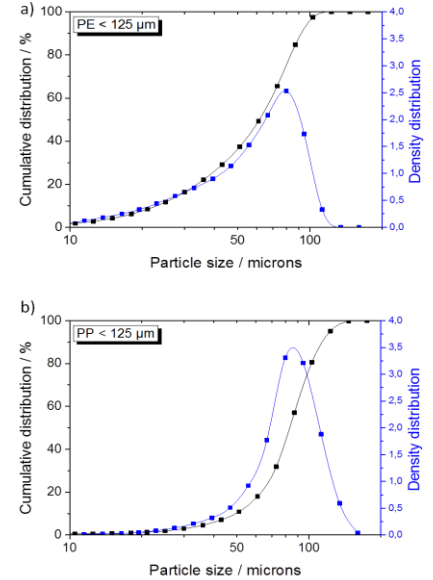


Soil reference material (<125 µm) contains of 1% PE,PP,PS,PET

OPTICAL MICROSCOPE



DLS



Courtesy of Dr. K. Altmann

Spectroscopic Methods: Confocal Raman Microscope



Witec Alpha 300

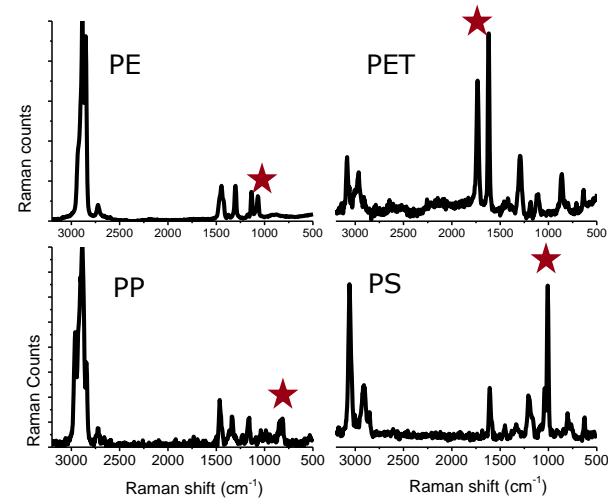
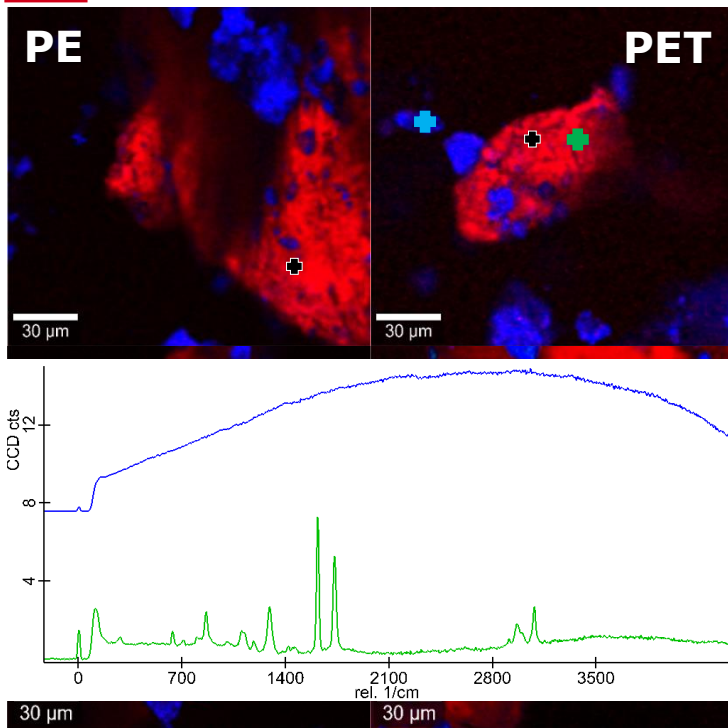
Basic Information

Spectra resolution $< 3\text{cm}^{-1}$, Pixel resolution $0,5\ \mu\text{m}$

Different wavelengths

Maximum scan size $1800\mu\text{m} \times 2000\mu\text{m}$ ($190 \times 200\mu\text{m}$)

Results: Raman imaging



- More detail information about chemistry on the surface of particles
- Time consuming (finding particles)
- Fluorescence

Spectroscopic Methods:

FTIR Microscope (Transmission Mode)



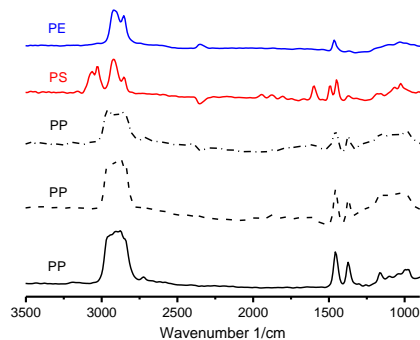
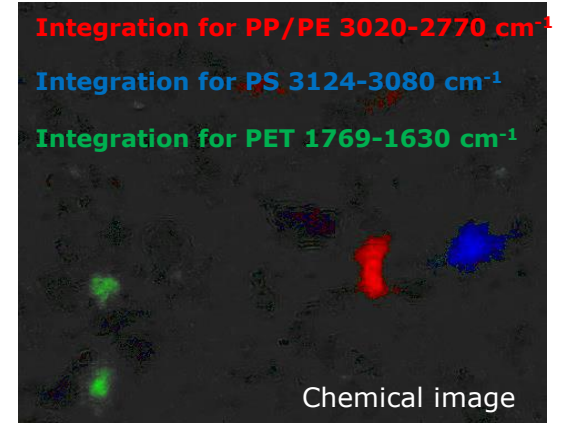
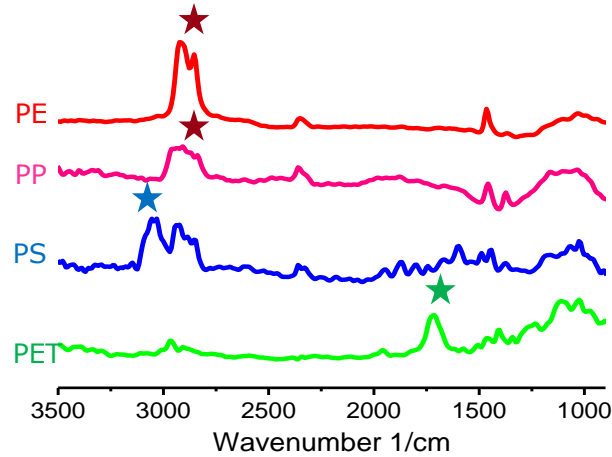
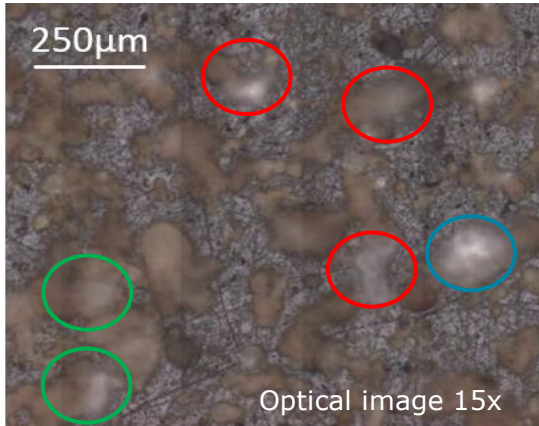
Basic Information

FPA detector: 4,096 spectra can be measured simultaneously covering sample areas $170 \times 170 \mu\text{m}$ (1 FPA field)

pixel resolution of $2.7 \mu\text{m}$.

Maximum scan size: 961 FPA fields $5.27 \times 5.27 \text{mm}$

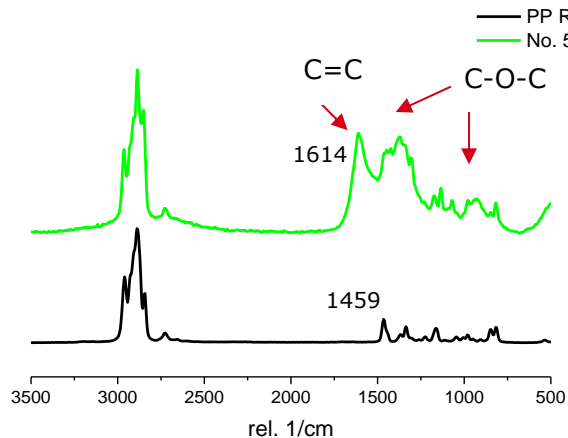
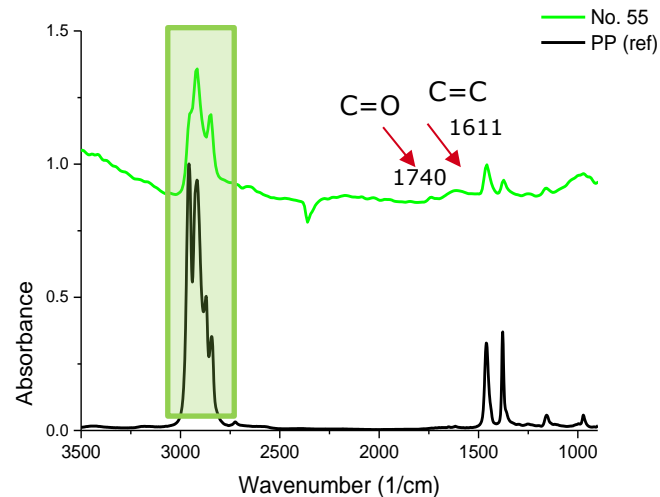
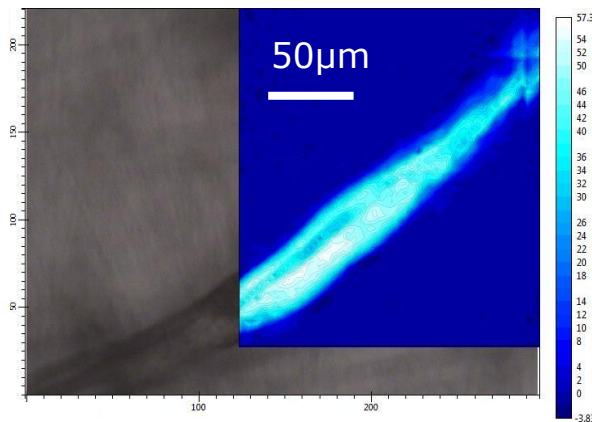
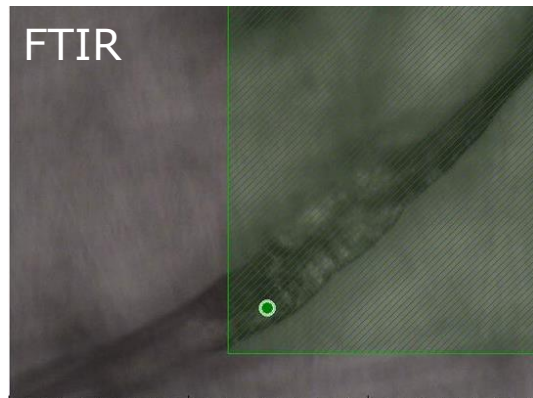
Results: FTIR imaging



- Big area up to 4x3 mm
- Poor signal to noise ratio
- Sample thickness less 100µm
- Long acquisition times (6h)
- Poor reproducibility

Comparison between RAMAN and FTIR in transmission

Wesch C., Elert A-M., Wörner M., Braun U., Klein R., Paulus M., Scientific Reports, 2017 accepted



- Raman more surface sensitive
- Aging status

Chemical Extraction Method TED-GC-MS



E. Dümichen et al., Assessment of a new method for the analysis of decomposition gases of polymers by a combining thermogravimetric solid-phase extraction and thermal desorption gas chromatography mass spectrometry, Journal of Chromatography A 1354 (2014) 117-128

Basic Information

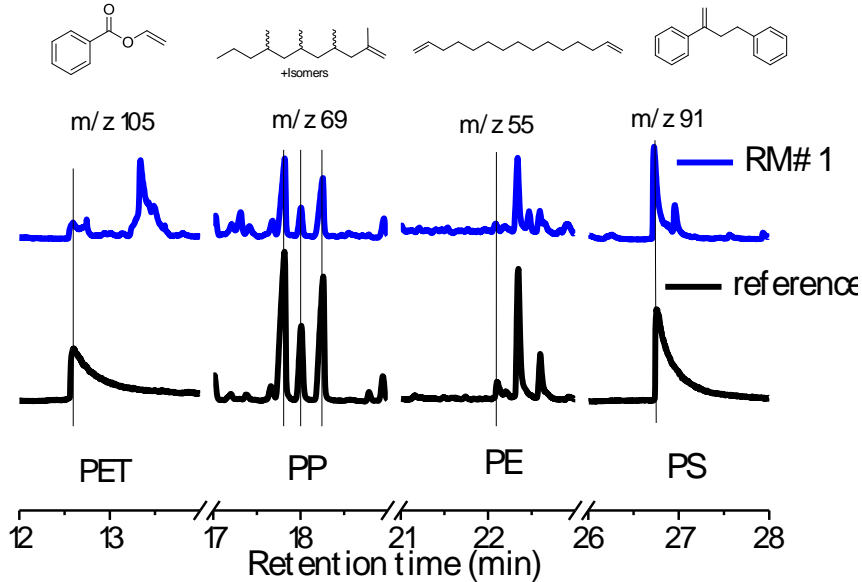
Identification via decomposition products

Sample amount: 20 - 50 mg, 300-600°C, 10 K/min, 30 mL/min N₂

Good results (PP, PE, PS)

Polymer	LOD in µg
PE	1,6
PP	0,44
PS	0,20
PET	0,68
PA6	0,52
PA6.6	2,8
PMMA	0,20
SBR	0,27

Results: TED-GC-MS



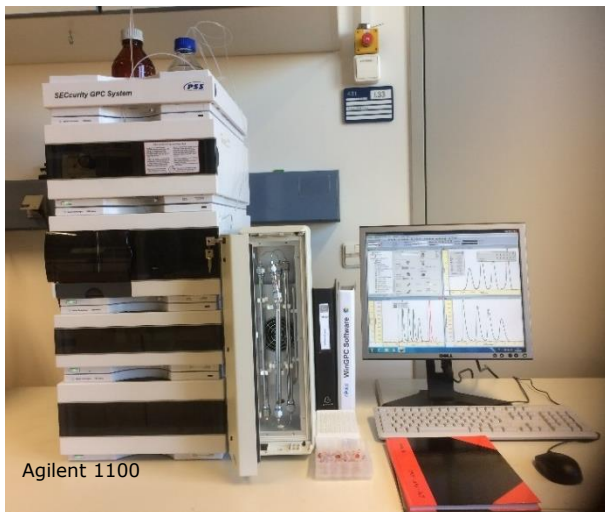
- Good reproducibility
- Fast analysis (YES/NO)
- No preparation needed

- No information about size or aging status of particles

- Possible quantification !

Chemical Extraction Methods

Size Exclusive Chromatography SEC



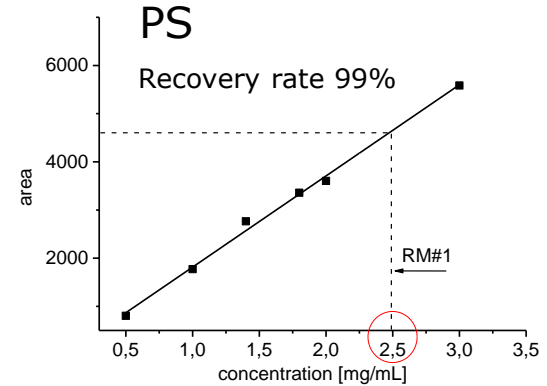
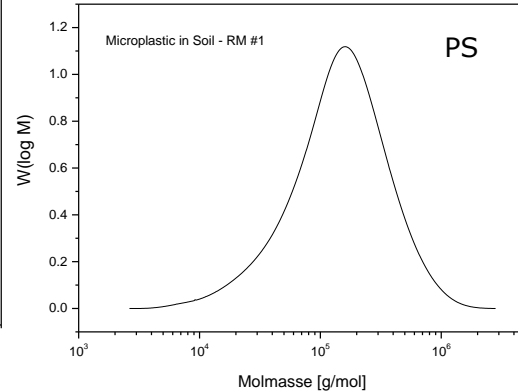
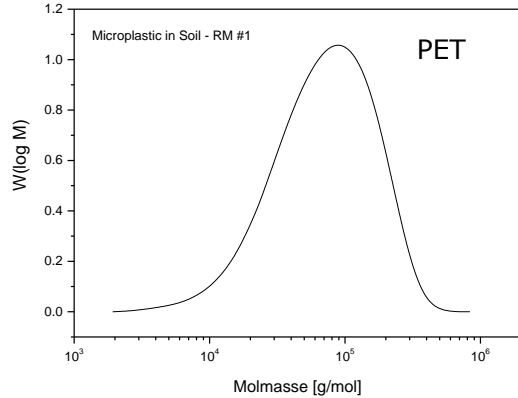
Basic Information

Solvents: THF for PS and HFIP for PET, PP and PE need high temp. SEC

Sample amount – 500mg

Detection of extract with IR ATR

Results: SEM and RP-LC (quantification)



[g mol ⁻¹]	PET Ref.	PET from RM	PS Ref.	PS from RM
M_w	92.5	93.0	208.7	205.7

- Big amount of sample
- Information about Mw distribution
- Quantification possible
- Only PET, PS (for PP, PE high temp. SEC)
- No information about size of particles
- Polymer has to be dissolved
- Information about degradation status

Assesment of the methods

Parameters	Raman scanning	FTIR imaging	TED-GC-MS	Chemical extraction
Sample amount	1 µg	1 mg	20 mg	500 mg
Sample preparation	Single particle	Single particle layer on IR transparent substrates	As received	As received
Measurement time for representative results	~ 40 min	3-6 h	3 h	2 h

Result information	Identification	PE, PP, PS, PET	PE, PP, PS, PET	PE, PP, PS, PET	PS, PET
	Quantification	No	No	Yes	Yes
	Particle size distribution	Yes	Yes	No	No
	Aging Status	Yes	Yes/No	No	Yes

Summary

- ✓ Scientific question “what information need to be known” – before analysis
 - ✓ TED-GC-MS, SEC – fast and quantitative analysis
 - ✓ Spectroscopy (FTIR, RAMAN)– particle size, more detail analysis
 - ✓ Combination of several parallel approaches - broader understanding
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Acknowledgement:

BAM, TF Microplastics Team

THANK YOU FOR YOUR ATTENTION!
