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Microplastic in Soils - Berlin

Current methods for the detection of microplastics in soils – An Overview

Carmen Wolf, Mike Wenzel, Jochen Tuerk

An-Institut der
UNIVERSITÄT DUISBURG ESSEN

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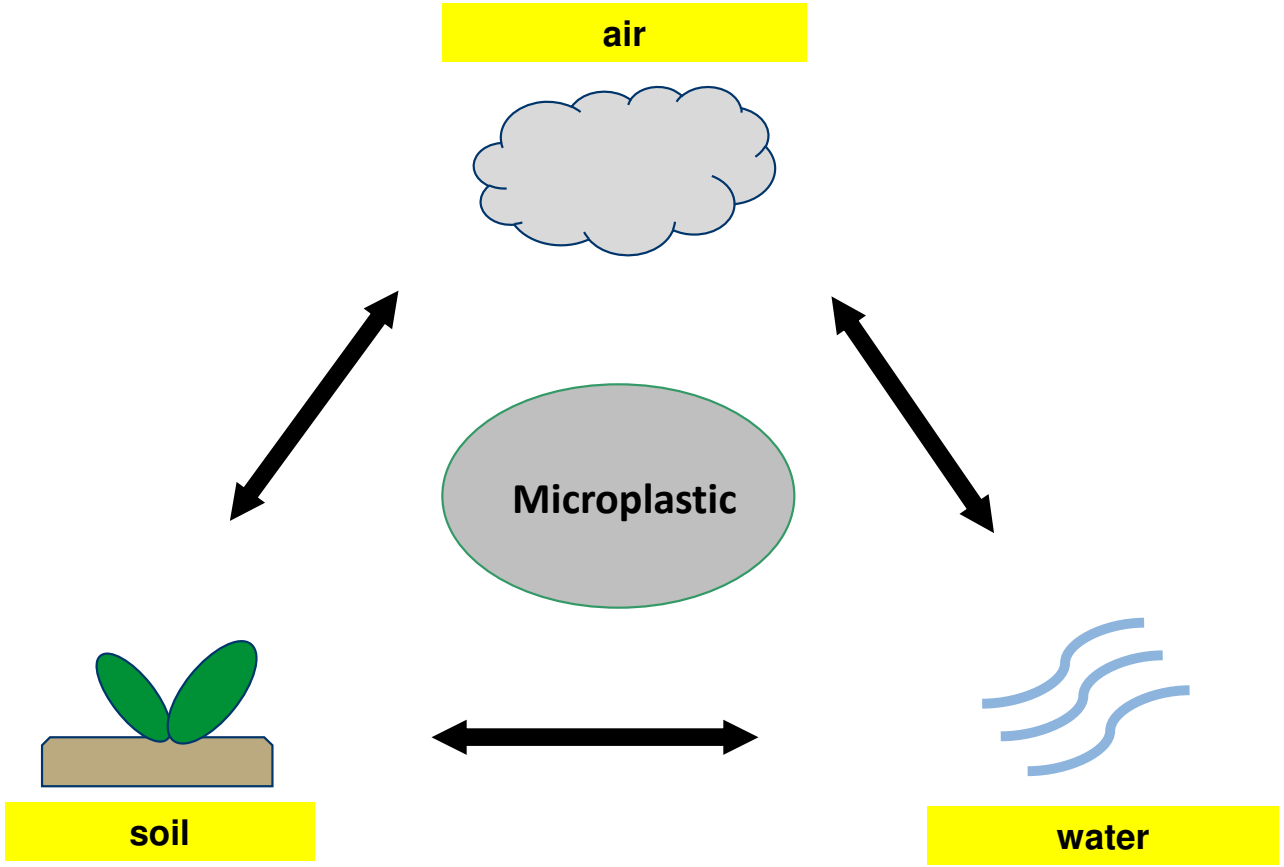
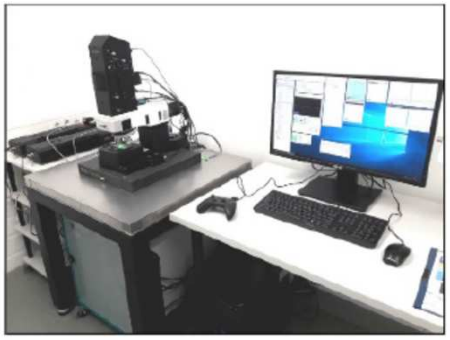
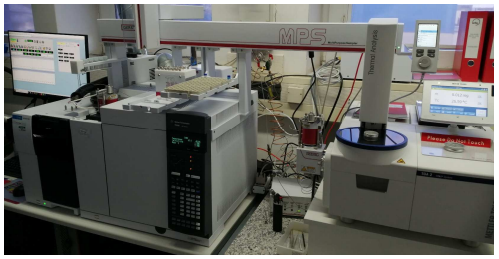
Emission



Microplastic in Soils

Sampling → Sample Preparation → Detection





Sampling

- Avoidance of plastic equipment
- Avoidance of plastic clothing → Note in the protocol
- Note in protocol if plastic fragments are seen on the soil

→ At this stage it is not yet clear which form is most suitable
→ More research needed

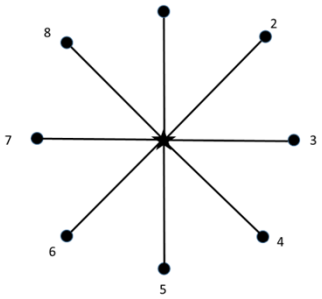
- Representative sampling
  - no homogeneous distribution → Hot spots
  - existing sampling methods suitable?
  - can different results be compared if different methods were used?

Sampling

Examples

- Sampling iMulch Project
  - Pürkhauer Sampling, diagonal across the field
  - Based on the sampling for the determination of soil parameters such as nutrient content

- Sampling UBA Projekt FKZ: 3720 72 288 0
  - Split tube sampler (4-5 kg - 0-30 cm – field, 0-10 cm grassland)
  - Satellite sampling
  - Based on: Richtlinie zur Probenahme und Probenbearbeitung – Umweltprobenbank 2012



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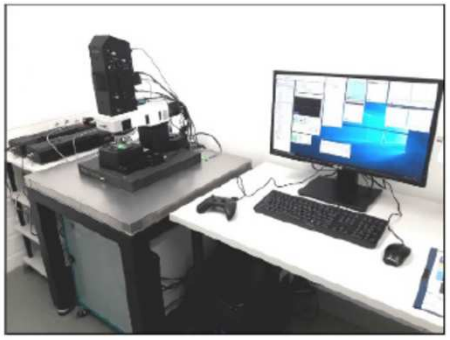

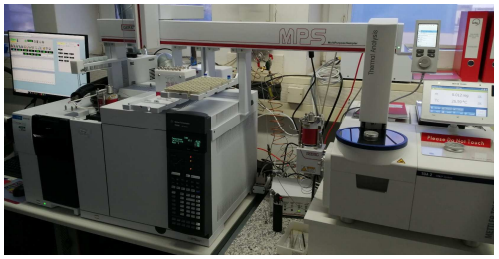

Sample preparation

Extraction Methods						
	Manual Extraction	**Electrostatic Separation**	**Consecutive Matrix Removal, mineral Fraction**			
			Oil extraction	Density separation	(Froth) Flotation	Magnetic Extraction
Size	>500 µm	>63 µm	no restriction	no restriction, tested for particles >40	no restriction	no restriction
further information	time intensive	tested for sand and sediment samples, adhesice forces to a metal drum, 3-4 h/150 g sample	lipophilic surface properties of most plastic particles	different devices available, different density of the solution dependend on the used salt	no chemicals, no effect on microplastics, small sample amount, hydrophobic adhesion on air bubbles	iron nanoparticle with hydrophobic hydrocarbon tails binding on MP surface; fragmentation of the MP
Recovery rates	high amount of false positives	90-100%	90-100%; ~ 74% real samples	95-100%; >13-40% aged plastic	~ 10-50% MP type dependent	~ 55%

Möller et al. 2020; Wenzel et al. in Prep.

➔ For thermo analytical methods like Pyrolysis or TED-GCMS the reduction of matrix is not necessary, but an enrichment step, to reach the limit of quantification

➔ For Spectroscopic Methods a reduction of the matrix is mandatory, but lower amounts of polymer particles can be detected

- Sample preparation must be as time saving and gentle as possible to avoid any effect on the Microplasticparticles

Sample preparation

Extraction Methods

Consecutive Matrix Removal, organic Fraction			
Acidic an… Diges…			…igestion
use of stro… alkaline soluti… the MP shap… concentration	Leading to different results	Leading to different results	…oped for …ples --> …uitable to …c materials of terrestrial plants, long reaction times

→ Most methods tested and validated for aquatic samples or sediments
→ Combination of different methods suitable (e.g. Density separation, Fenton, enzymatic digestion)
→ Ideally, a method that requires little or no chemicals

Al-Azzawi et al. 2019; Möller et al. 2020, Wenzel et al. in prep.

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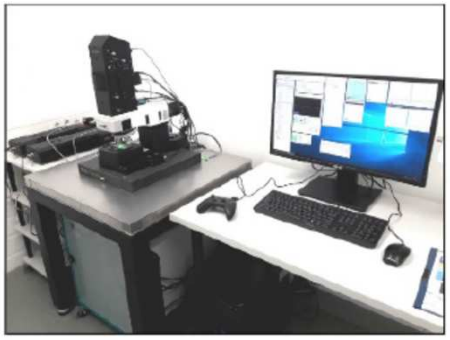
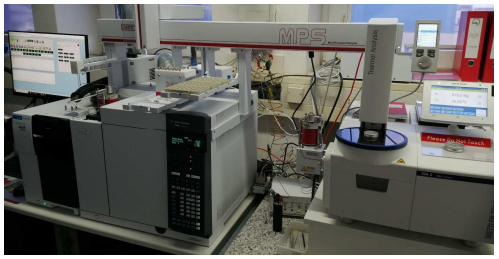
Sampling → Sample Preparation → Detection





Detection of microplastic

- **Optical methods**
  - Identification by shape and colour → false positive & negative results
- **RAMAN or FTIR spectroscopy**
  - Single
  - Data e
  - Requi

ut the shape

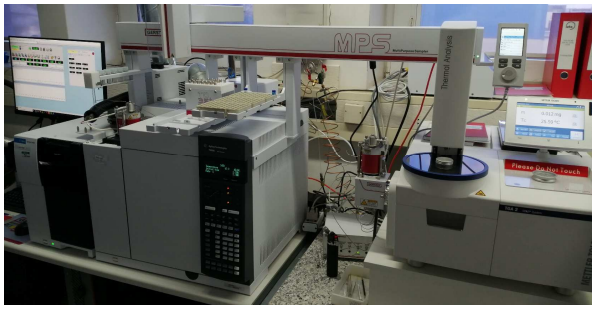
→Ideal is a combination of spectroscopy and mass spectrometry to gain information about the size, shape, particle number and mass quantity of microplastics

- **Pyrolysis or Thermal Extraction Desorption (TED)-GC-MS**
  - Fast chemical identification and quantification
  - Samples were pyrolysed and pyrolysis products were identified and quantified
  - No size or shape information
  - less laborious sample preparation as no matrix reduction is necessary but enrichment step to reach the LOQ

Example – iMulch – Method development

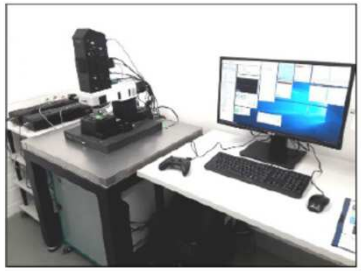
Development of a method wich allows the quantification, chemical identification, particle size distribution and shape identification of the particles

TED-GC-MS



- Samples were pyrolysed and pyrolysis products were identified and quantified
- Up to 100 mg can be analysed
- Less laborious sample preparation

RAMAN Spectroscopy

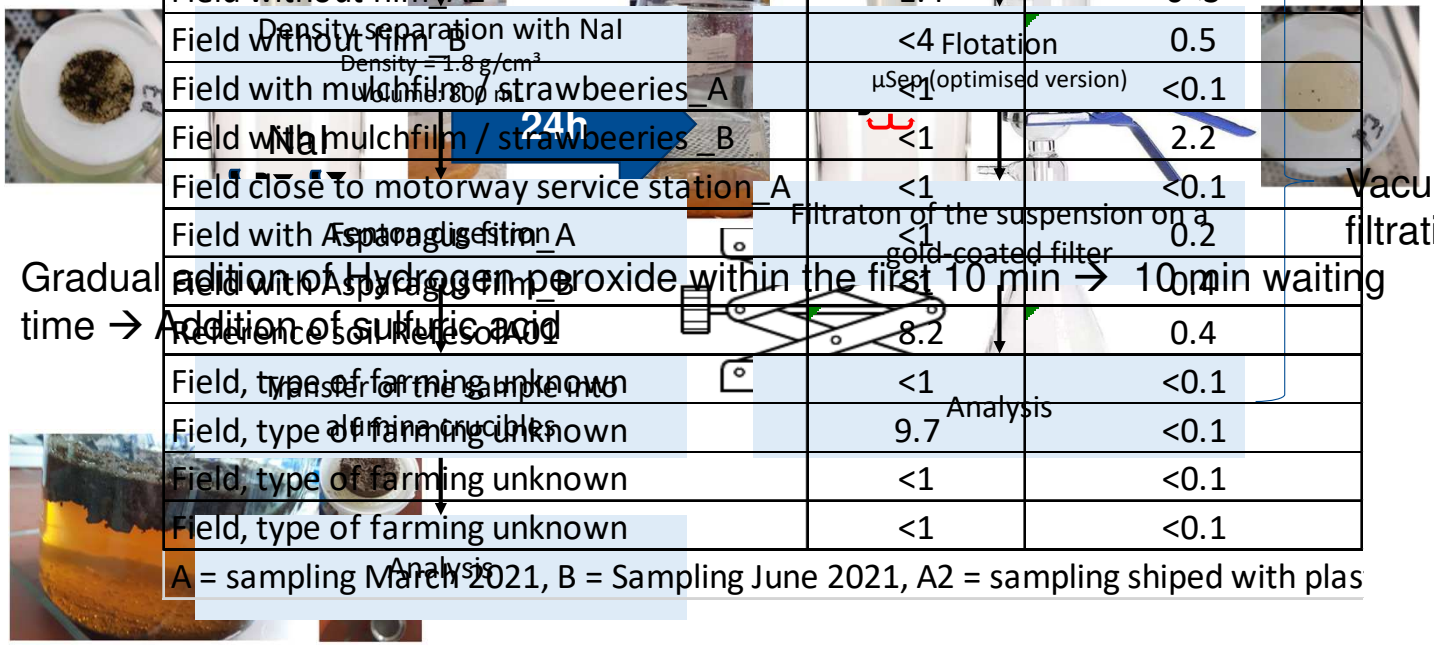


- Automated single particle analysis
- Size and shape information
- > 50 µm Particle detection limit
- Sample analysis within one working day
- Intensive sample preparation to reduce matrix particles

Example – iMulch – Sample preparation

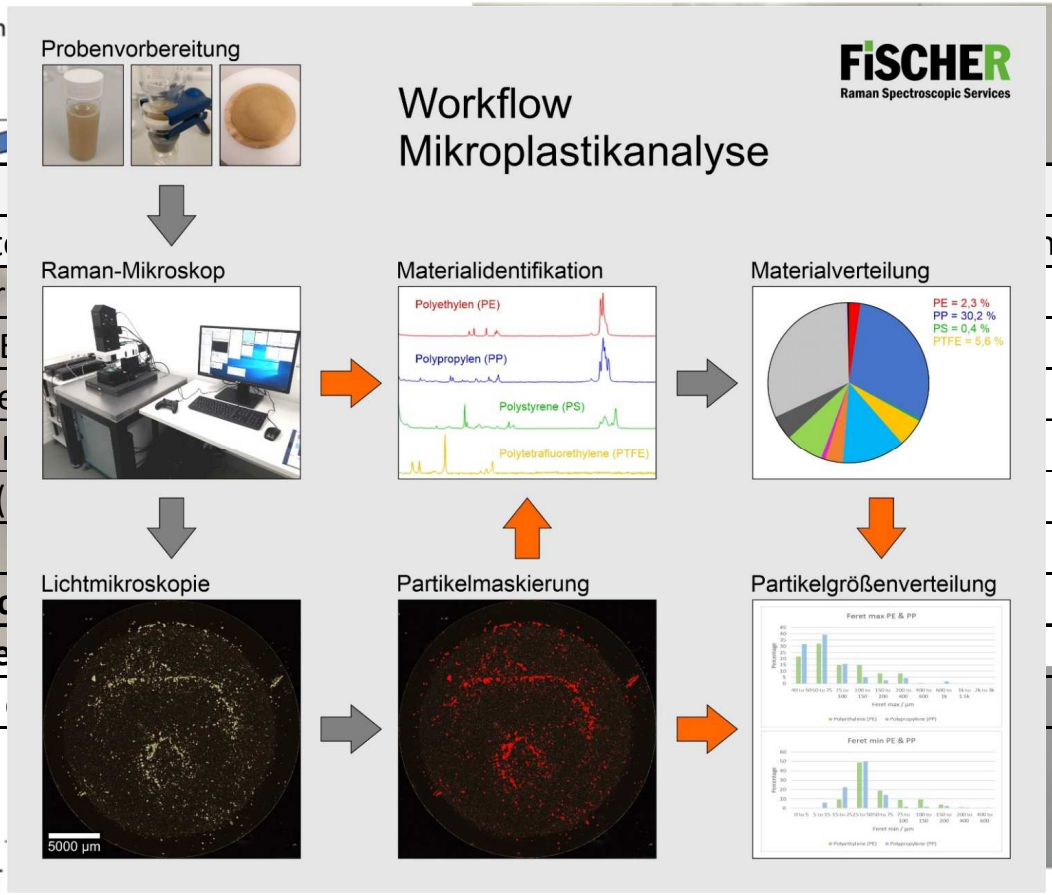
Field	µg PE / g Soil	µg PLA/PBAT / g Soil
Field with Biodegradable film_A	0.2	0.3
Field with Biodegradable film _A2	4.4	<0.1
Field with Biodegradable film_B	5.2	0.5
Field without film _A	0.8	<0.2
Field without film_A2	1.4	0<3
Field without film_B	<4	0.5
Field with mulch film / strawbeeries_A	<1	<0.1
Field with mulchfilm / strawbeeries_B	<1	2.2
Field close to motorway service station_A	<1	<0.1
Field with Asparagus film_A	<1	0.2
Field with Asparagus film_B	<1	0.4
Reference soil Reiseto A01	8.2	0.4
Field, type of farming unknown	<1	<0.1
Field, type of farming unknown	9.7	<0.1
Field, type of farming unknown	<1	<0.1
Field, type of farming unknown	<1	<0.1

A = sampling March 2021, B = Sampling June 2021, A2 = sampling shiped with plas



Gradual addition of Hydrogen peroxide within the first 10 min → 10 min waiting time → Addition of sulfuric acid

Example – iMulch – RAMAN



	2
Mat	nare in %
Polyethylenter	0.0323
Polyethylen (PE	-
Polytetrafluore	0.323
Polypropylen (	-
Polycarbonat (	0.0323
Polystyrol (PS)	0.0969
Polymer partic	**0.4845**
Matrix particle	**99.5155**
Total number	**100**

Conclusion

1. Sampling:

- At this stage it is not yet clear which form is most suitable
- More research needed

2. Sample preparation:

- Methods tested and validated for aquatic samples or sediments
- Combination of different methods suitable (e.g. Density separation, Fenton, enzymatic digestion)
- Ideally, a method that requires little or no chemicals
- Standardization needed

3. Detection methods:

- Spectroscopic methods – primary particle analysis – intensive sample preparation – small sample amount
- Thermoanalytical methods – LOD – less laborious sample preparation
- Ideal is a combination of spectroscopy and mass spectrometry to gain information about the size, shape, particle number and mass quantity of microplastics

Thank you for your attention

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