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Identification of Small Microplastic Particles in Water Samples by FT-IR Micro-Spectroscopy and Imaging – Possibilities & Challenges

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Introduction

For particles smaller than 1 mm it is hardly feasible to extract potential microplastics from the sample matrix by hand or manually sort and count them. Alternatively, we identify small microplastics by infrared (IR) spectroscopy coupled with microscopy and imaging features (*FT-IR imaging*).

As in typical chemical analysis, only a small fraction of the original sample is subjected to analysis. Sample pretreatment aims at removing as much interfering non-plastic matter as possible. A semi-automated procedure for data analysis of FT-IR images is presented. Having the number of microplastic particles in one sample aliquot, what is the expected weight of identified plastic items in the original sample?

Methods

Step 1. Take a representative water sample incl. solids that are potentially microplastics. Make sure the particle size you are interested in is not already excluded by the sampling method, i.e. prefilters of pumps, small hose diameter, inappropriate mesh size.

Fig. 1: Solid residues of clean WWTP-effluent (waste water treatment plant), separated on-site from 500 m³ of WWTP-effluent by 250 µm mesh net.

Step 2. Increase the sample's concentration of plastic by removal of plants (algae, leaves, seeds, wood etc.), invertebrates or inorganic material (sand).

- **Possibilities:**
  - Chemical treatment: H_2O_2, acids
  - Enzymatic digestion
  - Density separation in water, NaCl, $ZnCl_2$ …
  - Fractioning by size (sieves, filter)
- **Challenges:**
  - Don't dissolve the plastic material.
  - Know your sample well as to choose the right chemical/enzyme.
  - Don't take the density of the raw polymer for granted.
    - Density of the polymers is often altered by additives,
    - can change due to ageing effects in the environment or
    - can increase with biofilm forming on the particle surface.
  - When sieving consider agglomeration with gelatinous biomaterial as well as non-spherical (microplastic) particles.

Step 3. Analyse a representative sample aliquot, and determine **number** and **mass** of small microplastic particles in the original water sample.

Microplastic Analysis

1.FT-IR transmission measurement and imaging of sample aliquot on IR transparent filter material (Fig.2).

2.Calculate correlation with various plastic materials (PE, PP, PA…) on FT-IR image, display spots with correlation above threshold (e.g. > 75%, Fig.3)
→ **number of plastic particles on filter**

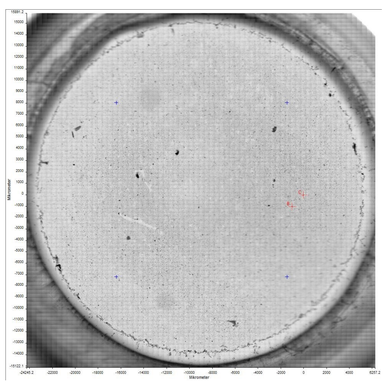

Fig. 2: Visual image of IR transparent filter covered with 1-2 mg micro particles
(image section: 2.5 cm x 2.5 cm)

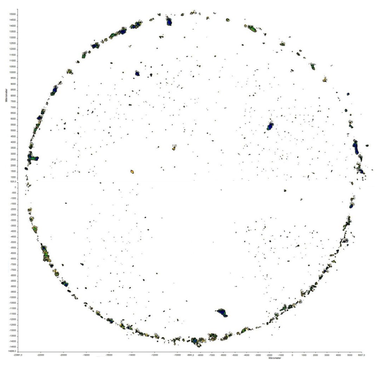

Fig. 3: Graphical result of FT-IR imaging, particles shown have high similarity to polypropylene (PP) plastics, and are counted via software.

3.Example for mass estimation of plastic particles

- Original sample: 500 m³ water with 10 mg/l total suspended solids.
- PP items counted in 1 mg aliquot: 1500 (±20% error).
- Assume: PP spheres, density 0.91 g/cm³, diameter?

Example Calculation	gram plastics in 500 m³ water sample		
Counts error:	*-20 %*	***0 %***	*+20 %*
25 µm diameter	45	**56**	67
50 µm diameter	357	**447**	536
100 µm diameter	2 859	**3 574**	4 288

- **Challenges:**
  - What is a representative aliquot mass or volume?
  - How to avoid overlapping particles and agglomerations?
  - Knowing the particle size is crucial for mass estimation.

Conclusions & Outlook

- **Small microplastic particles can be successfully identified by FT-IR Imaging.**
- **The number of particles can be counted in small sample aliquots.**
- **For reliable mass estimation of microplastics more knowledge on the particle size distribution and the plastic density is required.**

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