

Overview of methods and challenges for microplastic analysis

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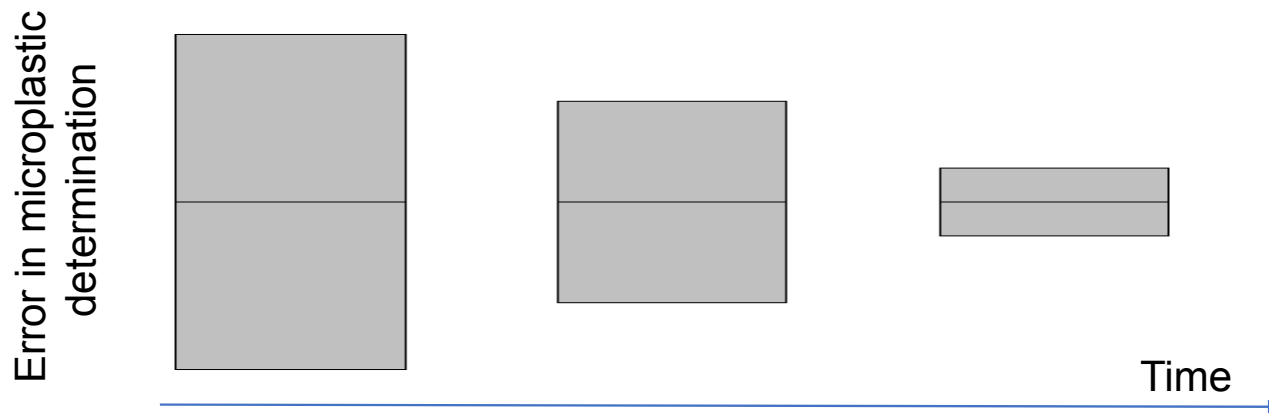
A major challenge

BASEMAN

Defining the baselines and standards for microplastics analyses in European waters

Citation from BASEMAN:

Although microplastics (MP) are recognized as an emerging contaminant in the environment, currently neither sampling, extraction, purification nor identification approaches are standardized, making the increasing numbers of MP studies hardly -if at all- comparable.



The scientific community works hard to reach valid methods

– but we are not there yet

Standardized and trustworthy analytical methods are needed

- Without proper analytical methods we cannot:
 - Assess the amount of microplastic in the environment
 - Distinguish which are the most important sources
 - Quantify impacts of microplastic
- We (the scientific community) do not (yet) have the final answer to how microplastic should be analyzed
- Over the later years, certain methods have shown promising results, while others have been deemed unsatisfactory

Lack of **Standardized Operation Protocols**

Analyzing for microplastic in the environment, there are many ways of doing:

- **Experimental design**
 - where and how to look for MP?
- **Sample collection**
 - Mesh sizes? Sample sizes?
- **Sample purification**
 - How to get rid of irrelevant substances without biasing the analysis?

Lack of Standardized Operation Protocols

Analyzing for microplastic in the environment, there are many ways of doing:

- **Microplastic identification**
 - How to safely distinguish artificial polymers from naturally occurring substances?
- **How to report results**
 - Particle sizes: What is the “size” of a particle? Particle mass: How best to quantify the mass of a particle?
- **Document the validity of the analysis – this is often forgotten ...**
- **Document uncertainties – this is often forgotten ...**

Size – why is it important?

- The traditional wisdom is that microplastic toxicity increases with decreasing size

ENVIRONMENTAL
Science & Technology

Article

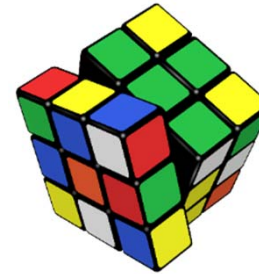
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Microplastic Size-Dependent Toxicity, Oxidative Stress Induction, and p-JNK and p-p38 Activation in the Monogonont Rotifer (*Brachionus koreanus*)

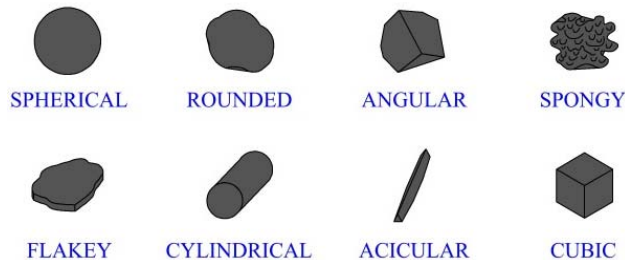
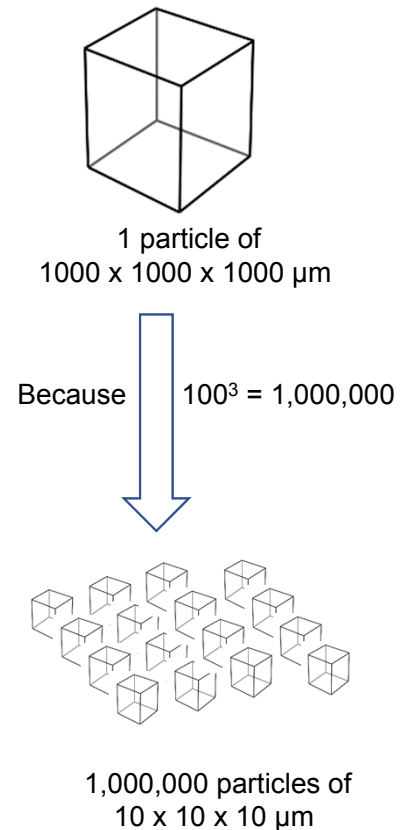
Chang-Bum Jeong,^{†,‡} Eun-Ji Won,^{†,§} Hye-Min Kang,[†] Min-Chul Lee,[†] Dae-Sik Hwang,[†] Un-Ki Hwang,^{||} Bingsheng Zhou,[±] Sami Souissi,[#] Su-Jae Lee,[∇] and Jae-Seong Lee^{*,†}

- So size matters

Size – why is it problematic?

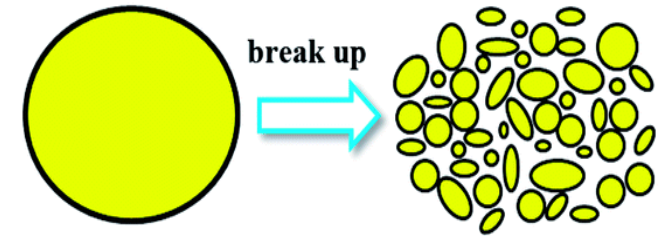


- Microplastic degrades in the environment, continuously creating smaller particles
 - One **Big** Particle becomes **Many Small** Particles
 - So what does a particle number really tell you?
- What dimension is it we report when we say “size”?



There is no clear consensus on this

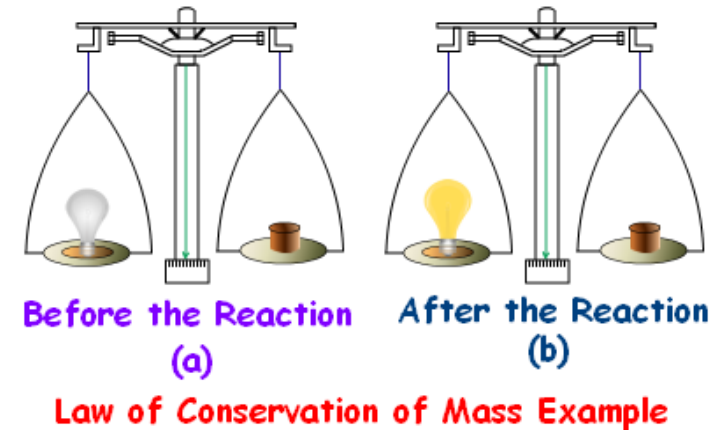
Size – why is it problematic?



- Any sample preparation applies forces on the particles
 - Sample preparation causes large particles to break up into smaller ones – the extent hereof is unknown
- Particle number is not a conserved unit – there is no law of particle number conservation
- Hence particle numbers and sizes cannot be used to establish balances like “which source is the more important”

Mass – why is it important?

- Mass is a consistent measure
 - There does exist a law of mass conservation !!!
 - Only mineralization will affect this measure
- Estimates on plastic loads to the environment must be made in units of mass (particle numbers make no sense here)
 - Mass must be measured to allow this



Mass – why is it problematic?



- Plastic is not one thing
 - Measuring plastic mass requires measuring the mass of many different polymer particles
 - No analytical method can actually detect all polymer types ...
- μ FT-IR imaging
 - Yields a mass estimates with unknown accuracy
 - Cannot measure car tire rubber
- TDU-Pyr-GC/MS; TED-GC-MS
 - Can measure many but not all polymers (PVC?)
 - (But it cannot measure particle sizes)

Size ranges and analytical methods

Microplastic range: 1 – 5000 μm

1 μm

10 μm

100 μm

1000 μm

10000 μm

Increasing uncertainty

Optical microscopy

Mikro-ATR-FTIR (single point analysis of particles on a filter)

Increasing uncertainty

ATR-FTIR (particles hand-picked, analyzed on bench)

Possibly down to a few μm (not proven)

Imaging $\mu\text{FT-IR}$ using filters, windows, or slides

Imaging μRaman – possible methods, not well proven

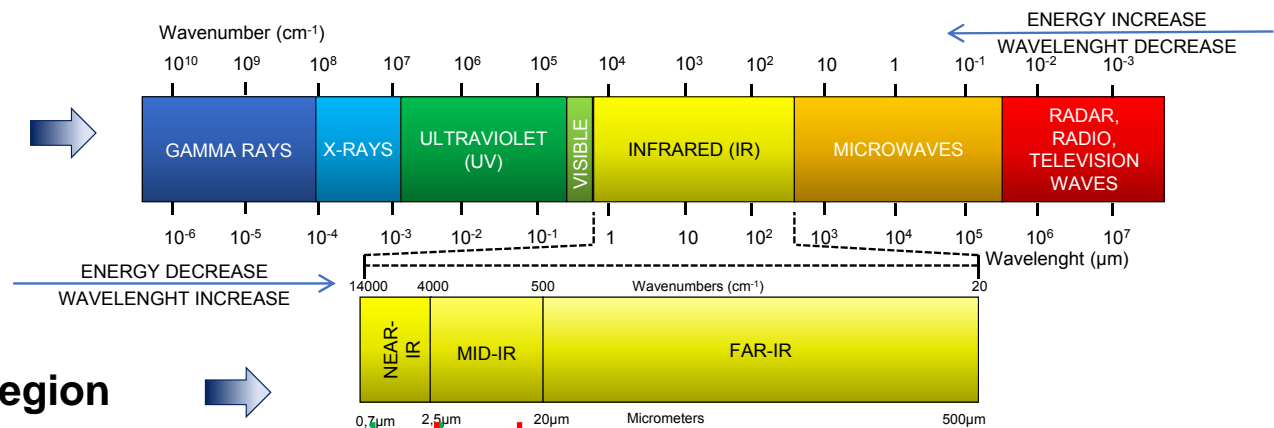
Macro Raman (particles hand-picked, analyzed on bench)

NIR (pre sorting) + Hy-Spec.
Imaging NIR (not well proven)

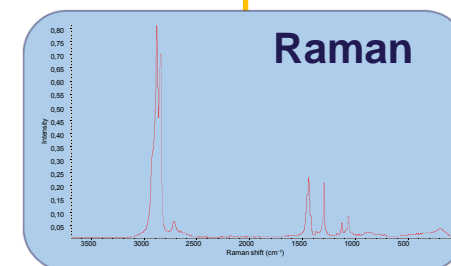
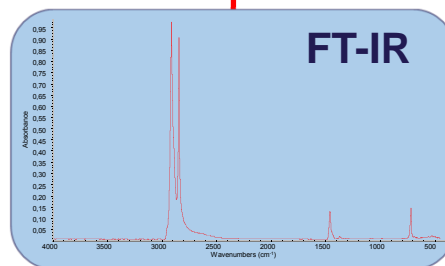
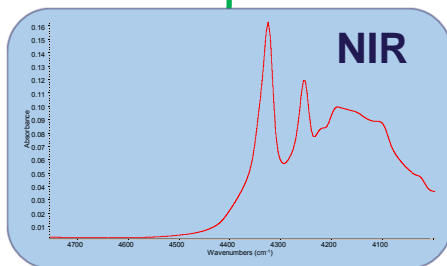
TDU-Pyr-GC/MS; TED-GC-MS

Infrared is the most suitable wavelengths for spectroscopic methods

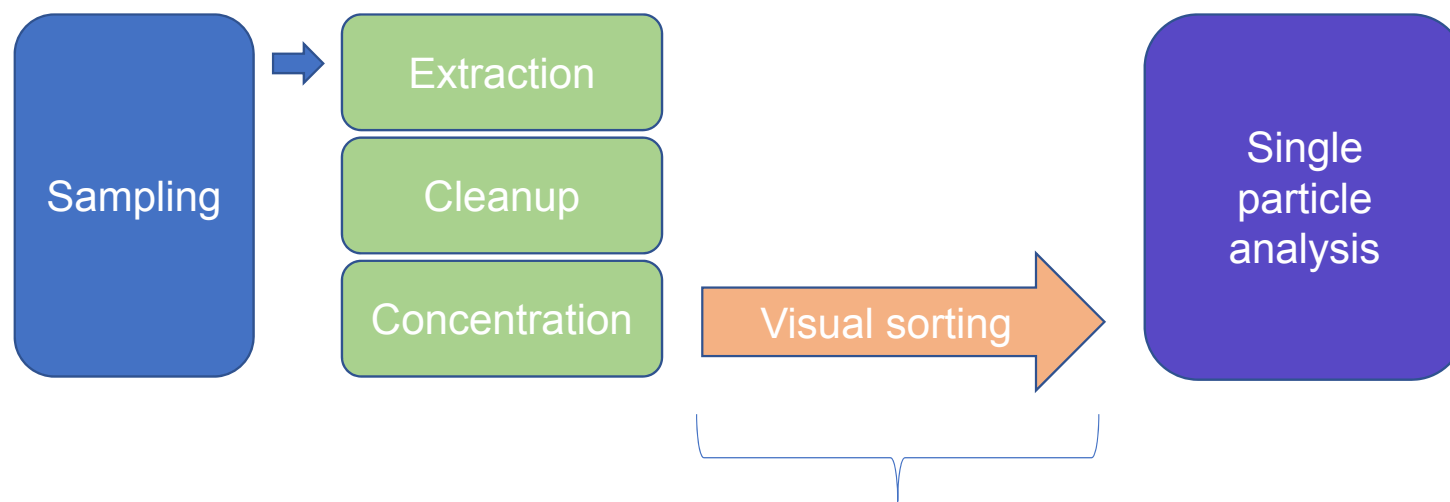
Electromagnetic spectrum



IR region



Typical work flow for single-particle analysis



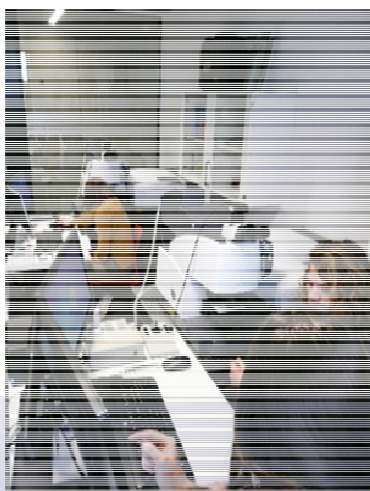
Main issues:

- Sorting is operator dependent
- Very difficult and time consuming for small particles

Methods ranked according to certainty (in my opinion....)

- Compound or stereo microscopy
- Fluorescence microscopy
- Single point Raman
- TDU-Pyr / TGA-GC-MS
- Single point FT-IR
- ATR-FT-IR

The work flow for imaging analysis



Alvise will talk about this method in the next lecture

μ FT-IR

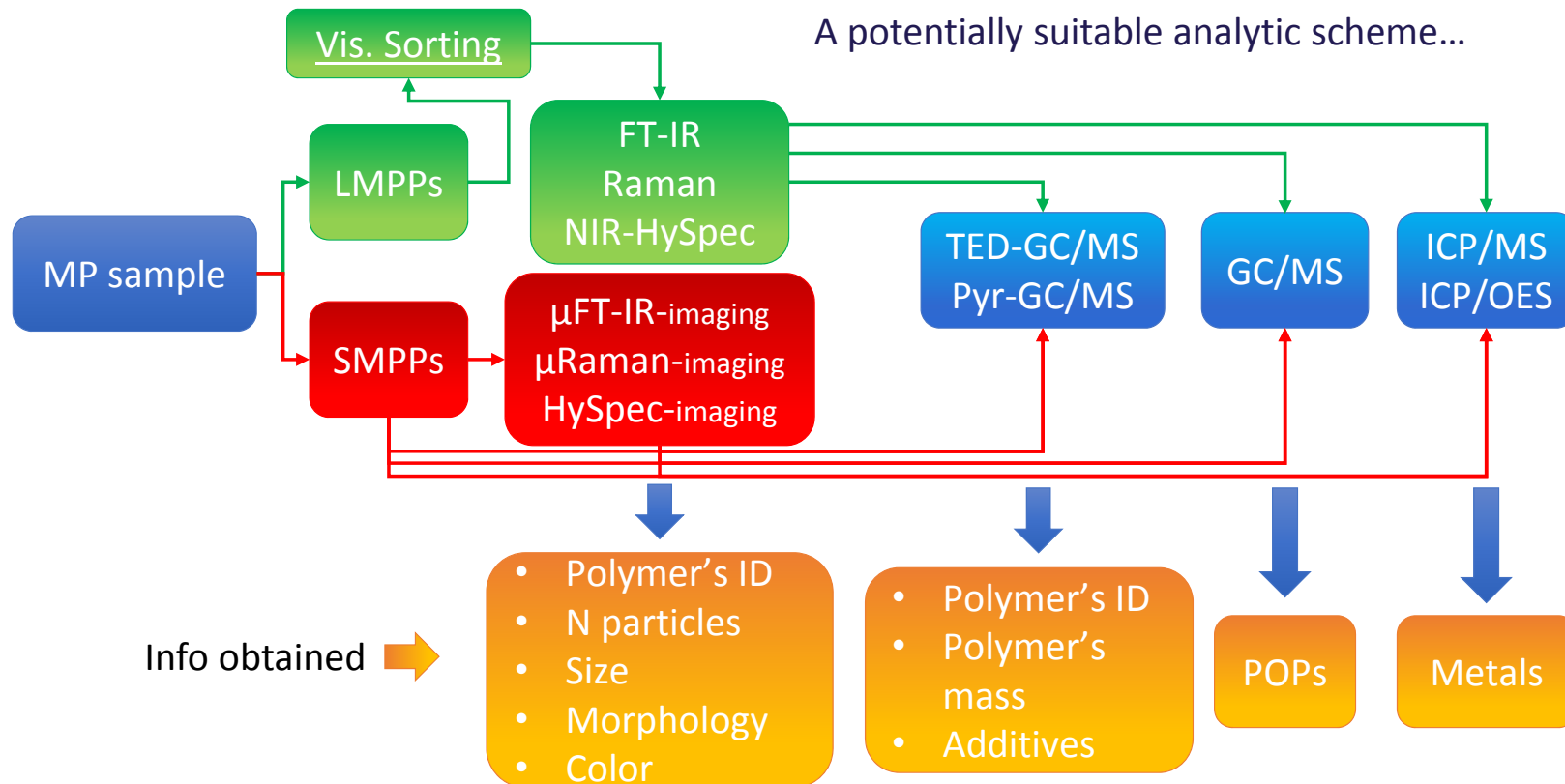
- Well proven and tested. Most used approach

μ Raman

- Possible method but not well proven and tested

I believe the solution is not one method, but a suit of methods

Analytical flow



The Aalborg University microplastic research group

- Foundation in urban polluted waters
 - Wastewater, stormwater, sludge,
- Receiving environment impacted here by
 - Soil, water, air
- Focus on developing better, faster, and more valid methods for microplastic quantification
- Focus on quantifying microplastic in the environment

