Example of microplastic analysis

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Before you begin

Know your target

It determines ALL your next steps

Where from this sample was taken

LIQUID *seawater, freshwater, wastewater*

SOLID *sediment, soil, sludge*

BIOTA *algae, insects, fish, etcetera*

- *surface? subsurface? Above ocean floor? Before settling tank? before primary treatment?*
- *homogenised?* **LOCATION** *depth? top layer? low thermodynamic flow? Overturn depth?*
- *Pristine population? juvenile? contaminated area? lab-grown? DGI tract only?*

Sample treatment

- To use as less steps as possible

MethodsX 9 (2022) 101603

Contents lists available at ScienceDirect

MethodsX

journal homepage: www.elsevier.com/locate/mex

Method Article

Hide-and-seek: Threshold values and contribution towards better understanding of recovery rate in microplastic research

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ABSTRACT

Microplastic pollution has become one of the most pressing environmental issues. A fundamental criterion for risk assessment is the concentration of found microplastic that can be altered during microplastic isolating from the sample. Recovery rate (i.e. positive control) is an important feedback component that identifies accuracy, quality and efficiency of sample processing, same as physical and chemical impact. Here, using 100 µm red polystyrene (PS) beads we have tested some methodological steps that can be responsible for the possible microplastic losses during sample treatment and based on that, we provided a recovery rate threshold values. Our results support that the choice of the extraction method (vacuum filtration versus wet sieving) results in lower recoverability when vacuum filtration is used and that used separatory funnels size versus material amount impacts the efficiency or recoverability in density separation. We have also analysed microplastic recovery rate

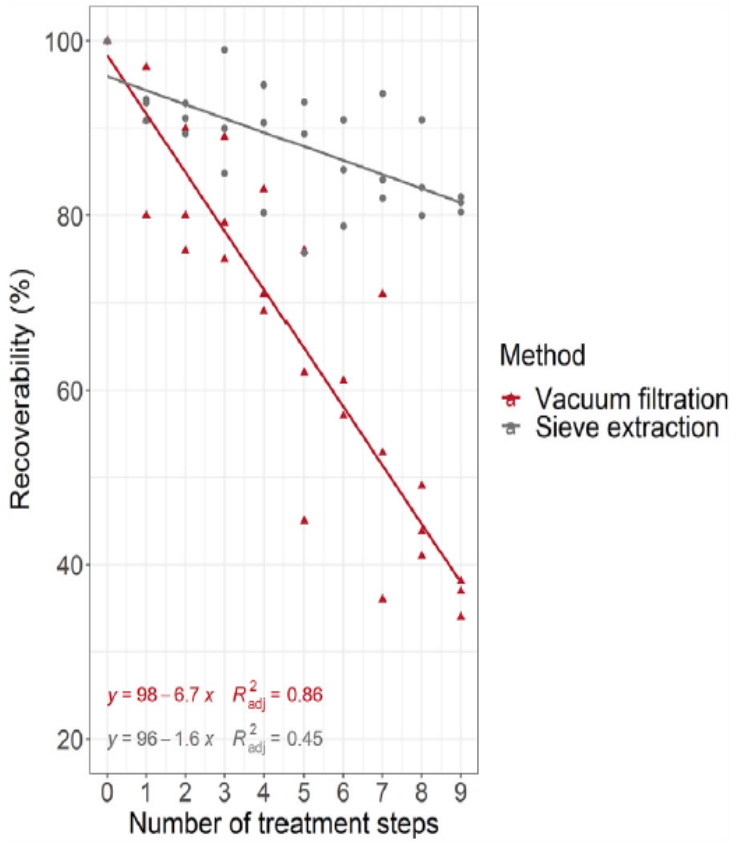


Fig. 4. Recoverability (%) of the 100 polystyrene (PS) beads depending on extraction method (vacuum filtration and sieve extraction) during nine consecutive treatment steps.

Chemical treatment
Physical separation

For water samples oxidation with H2O2 (30%) for 2 days at room temperature

For sediment samples no oxidation was made, density separation with NaI (1.8 g/cm3) repeated for three times

Sieving through 300um and 10 um metal filters

Substrate choice

Sample properties

(Un)known compound

Reflectiveness

Thickness

Roughness

Compatibility

Substrate choice

Depth of analysis

Entire sample

Subsample

Substrate choice

Deposition method

Filter

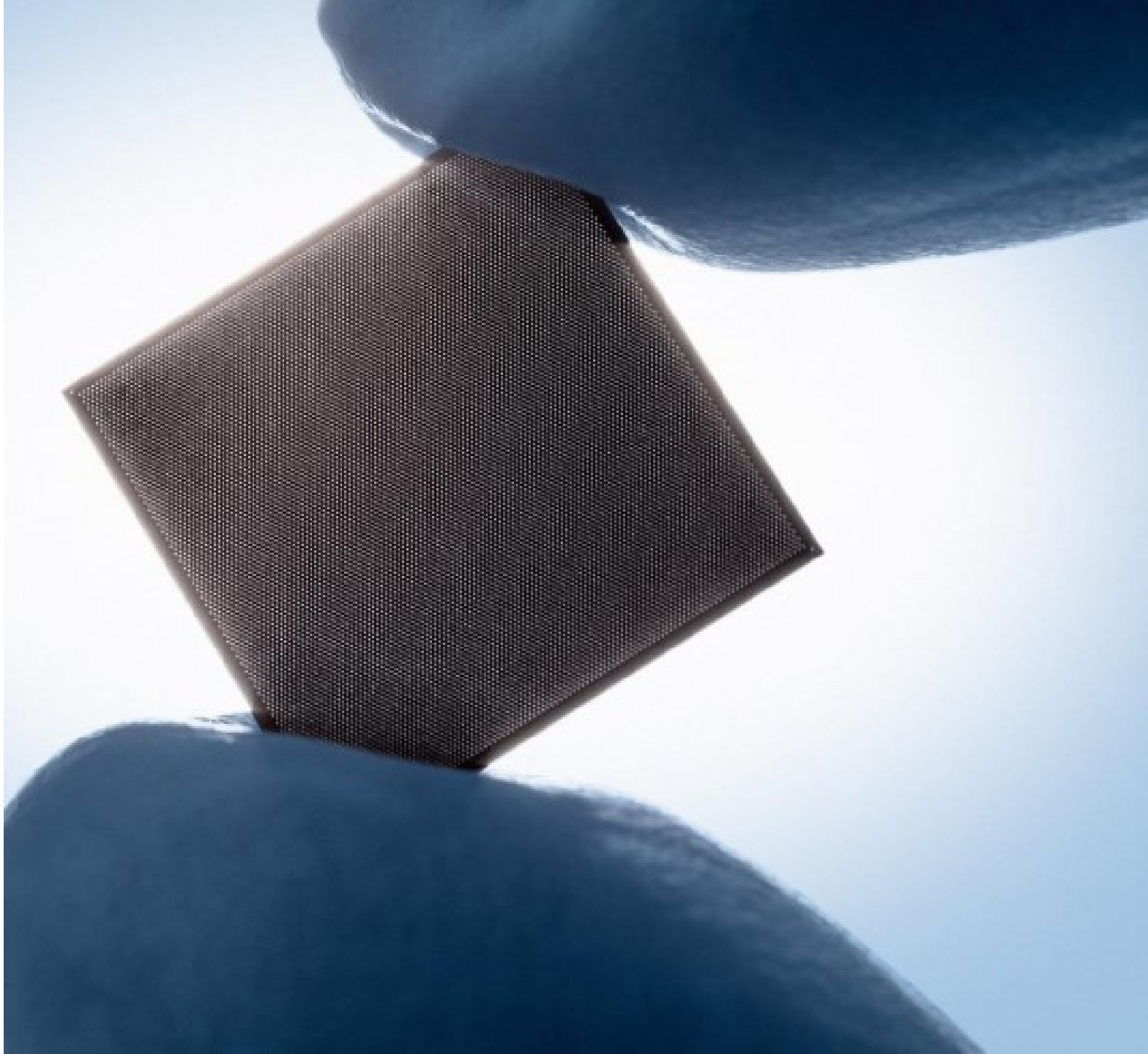
Smear

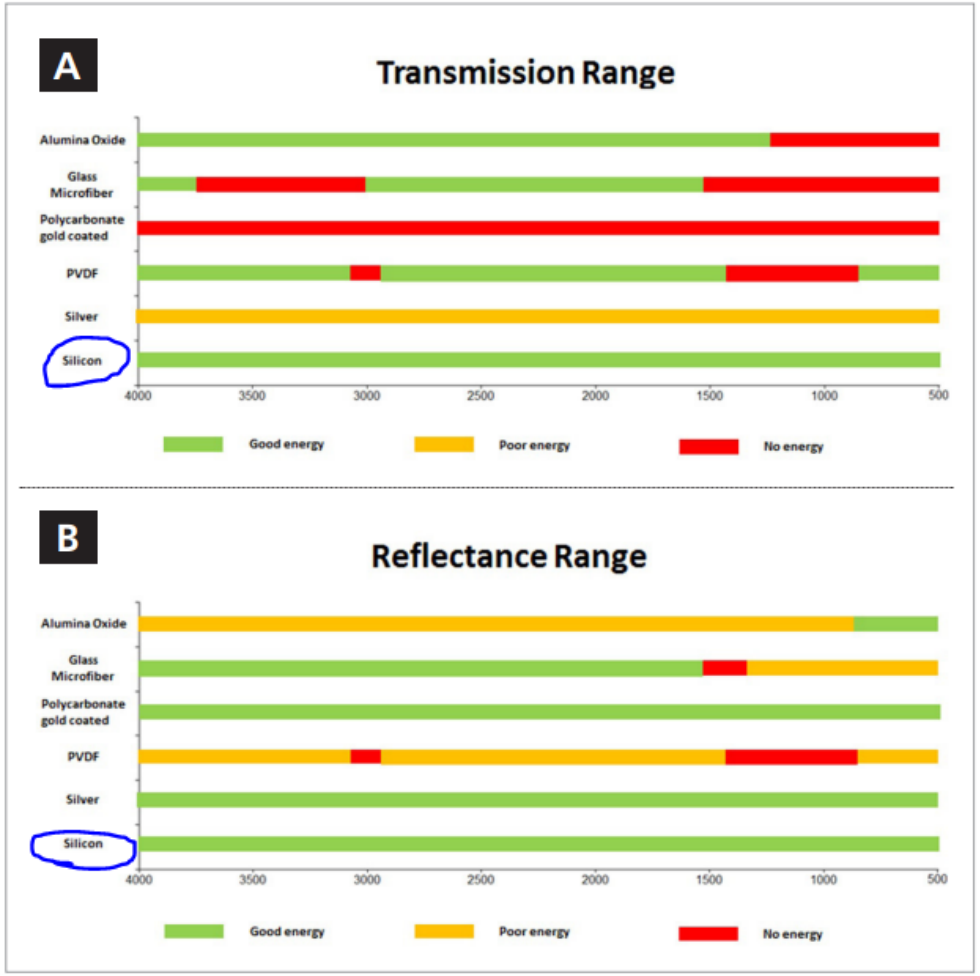
Compacting disc

Liquid evaporation

Substrate for FTIR imaging

- Silicon filter (transmission) for size < 300 um- u FTIR Imaging (Size 11 x 11 mm2, Si-thickness: ~170 – 230µm, pore size 10 um in diameter)
- Optical ZnSe window (transmission) for size > 300 um- measuring a spectra from particle (point mode)





Why silicon filter?

- Silicon filter can be used to perform both transmission and reflectance analysis in range 500- 4000
- The only negative points about the silicon are the relative cost and the “non-standard” sizes (rectangular dimensions) that are not directly compatible with standard filtration systems

u-FTIR Spotlight 400 (Perkin Elmer)

✓Particle information

✓Material type per particle

±Estimated mass

–Need for substrate in lower size range

–Rubber identification difficult

Scans interpretation

For particles > 300 um- comparing spactras with in-house built library of polymers

For particles < 300 um- scanning the whole filter and interpreting using siMPle programme

Microplastic Identification Using siMPle Software: Summary of Methodology and Optimization

- **Software Used:** Polymer identification was performed using the **siMPle** program (classic 2020 version), with the **AAU pipeline** and derivative-based analysis (Pearson weights: 0/1/1).
- **Method Strengths:**
  - Transparent and **robust approach**, requiring no reference spectra for natural materials
  - **Good reproducibility**
- **Limitations and Considerations:**
  - Polymer **Threshold Values (TVs)** need to be carefully optimized to balance **false positives (~5%)** and false negatives
  - Even with optimization, **some MPs may be underreported** due to spectral/matrix limitations.

Microplastic Identification Using siMPle Software: Summary of Methodology and Optimization

- **Quality Control:**
  - Essential step – **manual visual QC** of MPs is more reliable than automated checks
  - Doubtful identifications were cross-checked using an additional spectral database (Open Specy)
- **Conclusion:**
  - This method is **not perfect**, but with manual QC and optimized settings, it yields **plausible and conservative** estimates of MP presence
  - **Standardization** of spectral analysis methods is essential for cross-study comparability


TAL TECH
0,33 mm
0,33 mm
0,33 mm
MSI
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1 mm
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