METHOD OPTIMIZATION FOR REDUCTION ANALYSIS OF MICROPLASTICS IN WASTEWATER

BY: SARAH E. ABNEY
Global average weekly plastic consumption

On average people swallow this number of plastic particles each week from the following foods/drinks that have the highest plastic levels:

- Plastic particles (0-1mm)

Total plastic ingested = 5g

The equivalent of one credit card

- Tap/bottled water: 1,769
- Beer: 10
- Salt: 11
- Shellfish: 182

Source: WWF/University of Newcastle, Australia
Graphic: Woojin Lee, CNN
Figure 3: Map of average percentage of tap water sample containing plastic fibers and average number of fibers (>100μm) per 500 ml

- Percentage of tap water samples containing plastic fibers
- Average number of fibers per 500 ml

Countries with high percentages include:
- US: 94.4%
- US: 72.2%
- US: 98%
- Ecuador: 79.2%
- Uganda: 80.8%
- Indonesia: 82.4%
- Europe: 1.9
- Lebanon: 4.5
- India: 4
- Indonesia: 1.9
Impacts of baseflow and flooding on microplastic pollution in an effluent-dependent arid land river in the USA.
Typical Removal Rates of Wastewater Treatment Plants
# Microplastics to Scale

Micro- and nanoplastics are of similar size to many biological organisms, and become harder and more expensive to analyse as they get smaller.

<table>
<thead>
<tr>
<th>Biological objects</th>
<th>Non-biological particles</th>
<th>Tools for analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Nanoplastics</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10 nm</td>
<td>100 nm</td>
<td>1 μm</td>
</tr>
<tr>
<td>Particles may cross blood-brain barrier</td>
<td>Fish eggs and larvae</td>
<td></td>
</tr>
<tr>
<td>May cross into cells</td>
<td>Unicellular marine algae</td>
<td></td>
</tr>
<tr>
<td>Asbestos flakes</td>
<td>Sand and sediment</td>
<td></td>
</tr>
<tr>
<td>PM$_{2.5}$*</td>
<td>&gt;100 μm</td>
<td>Naked eye</td>
</tr>
<tr>
<td>&lt;$1 μm</td>
<td>≈ 100 μm</td>
<td>Optical microscope ($700–3,000)</td>
</tr>
<tr>
<td>Py-GC/MS* ($200,000–300,000)</td>
<td>&gt;10–20 μm</td>
<td>FTIR* ($&gt;25,000)</td>
</tr>
<tr>
<td>&gt;1 μm</td>
<td>Micro-Raman spectroscopy ($&gt;50,000)</td>
<td>(Black or dark-coloured particles can't be identified)</td>
</tr>
</tbody>
</table>

*Particulate matter less than 2.5 micrometres (PM$_{2.5}$) or less than 10 μm (PM$_{10}$) in diameter, often from spoil, vehicle exhaust or dust. FTIR: Fourier-transform infrared spectroscopy. Py-GC/MS: pyrolysis-gas chromatography-mass spectrometry.
Fourier transform infrared (FTIR) spectroscopy is the traditional choice for plastics analysis.

Con: The large incoherent light source can be difficult to focus onto a small microparticle.

Laser Direct Infrared (LDIR) chemical imaging system features a bright infrared laser source with proprietary Quantum Cascade Laser (QCL) technology

Analysis time for a 33 GB data file:

FTIR = 11 hours
LDIR = 2 hours
The key benefits claimed by Agilent

- **Automated** sample analysis.
- Ability to survey large sample areas and then explore smaller areas of interest in **more detail** without changing any optics.
- Full software control allows changing the field of view from **microns to centimeters** or the pixel size from 1 to 40 μm.
- Acquire ATR imaging data with pixel size as small as 0.1 μm for **unmatched image detail and spectral quality**.
- **Rapidly identify** unknowns using either commercial or custom libraries via ATR capabilities.
- Obtain relative quantitative information of sample constituents **without complex method development**.
- No requirement for liquid nitrogen **reduces operating costs** and simplifies maintenance.
<table>
<thead>
<tr>
<th>Source</th>
<th>Method</th>
<th>Volume Processed</th>
<th>Analysis</th>
<th>Cost</th>
</tr>
</thead>
<tbody>
<tr>
<td>Löder (2017)</td>
<td>Basic Enzymatic Purification Protocol (BEPP) w/ SSS</td>
<td>&gt;10L</td>
<td>FPA-based FTIR analysis</td>
<td>3214.65</td>
</tr>
<tr>
<td>Long et al. (2019)</td>
<td>Filtration</td>
<td>&lt;350L</td>
<td>micro-Raman spectroscopy</td>
<td>759.3</td>
</tr>
<tr>
<td>Ziajahromi et al. (2017)</td>
<td>Seiving</td>
<td>&lt;200L</td>
<td>FT-IR analysis</td>
<td>345.7</td>
</tr>
<tr>
<td>Mason et al. (2016)</td>
<td>Seiving</td>
<td>500-21000L</td>
<td>visual-only identification</td>
<td>1657</td>
</tr>
<tr>
<td>Schymanski (2017)</td>
<td>Filtered</td>
<td>700 mL - 1500 mL</td>
<td>u-Raman spectroscopy</td>
<td>2200</td>
</tr>
<tr>
<td>Murphy (2016)</td>
<td>Filtering</td>
<td>1000</td>
<td>FT-IR analysis</td>
<td>1455</td>
</tr>
<tr>
<td>Carr (2016)</td>
<td>Filtering</td>
<td>2.8x10^6</td>
<td>Visual identification//FT-IR</td>
<td>4194.5</td>
</tr>
<tr>
<td>Magnuusson (2014)</td>
<td>Plankton Sieves</td>
<td>cubic meters</td>
<td>FT-IR analysis</td>
<td>4350</td>
</tr>
<tr>
<td>Simon (2018)</td>
<td>Filtering</td>
<td>&lt;81.5L</td>
<td>FT-IR analysis</td>
<td>3548</td>
</tr>
<tr>
<td>Uurasjärvi (2020)</td>
<td>Plankton Sieves</td>
<td>&lt;468L</td>
<td>FT-IR analysis</td>
<td>1590.1</td>
</tr>
</tbody>
</table>
Designation: D8332 – 20

Standard Practice for
**Collection** of Water Samples with High, Medium, or Low Suspended Solids for Identification and Quantification of Microplastic Particles and Fibers¹

This standard is issued under the fixed designation D8332; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

Designation: D8333 – 20

Standard Practice for
**Preparation** of Water Samples with High, Medium, or Low Suspended Solids for Identification and Quantification of Microplastic Particles and Fibers Using Raman Spectroscopy, IR Spectroscopy, or Pyrolysis-GC/MS¹

This standard is issued under the fixed designation D8333; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.
NEW METHOD FOR LARGE VOLUME COLLECTION:
(SUBMITTING FOR ASTM STANDARD)
SUMMARY:

• Polymer plastics (PolyVinyl Chloride, Polystyrene, Polyethylene terephthalate, Polytetrafluoroethylene, Cellulose Acetate) along with some polyamides (protein), cellulosic, silica (sand) particles were observed in the samples.
• Polyamides was the most prevalent particle identified which could be wool, silk and other textile product or the protein rich debris introduced from sample preparation.
• Polyurethane, Synthetic wax, Alkyd, fatty acids, and acrylic polymers were grouped into one as these spectra has high similarity.
• Manual analysis has now been replaced by automated particle analysis workflow in the software.
• Example analysis based on automated particle analysis feature in the software was shown.
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