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Microplastic Analysis with FTIR and Raman Spectroscopy

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GOALS AND OBJECTIVES

Develop a methodology for the isolation and analysis of microplastic particles using FTIR and Raman spectroscopy.

Isolate microplastic particles from solutions using filters that allow direct spectroscopic analysis.

Determine the size and shape of the microplastic particles using image analysis.

Identify the type of microplastic using FTIR or Raman spectroscopy by matching to spectral databases

EXPERIMENTAL

The drinking water sample was just a commercially available 500 ml bottle of drinking water.

The silicon filters and filtering apparatus are part of the Thermo Scientific™ Microparticle Sample Preparation Kit.

The analysis of the particles was carried out using either a Thermo Scientific™ Nicolet™ iN10 MX FTIR Microscope or a Thermo Scientific™ DXR2 Raman Microscope (see Figure 1).

Automated particle analysis was accomplished using the Particle Wizard option in the Thermo Scientific™ OMNIC™ Picta™ Software for the iN10 MX FTIR Microscope and the Thermo Scientific™ Altμs Particle Analysis option in the Thermo Scientific™ OMNIC™ for Dispersive Raman Software for the DXR2 Raman Microscope.

Figure 1: (a) Thermo Scientific Nicolet iN10 MX FTIR Microscope, (b) Thermo Scientific DXR2 Raman Microscope

(a)

(b)

SAMPLE PREPARATION

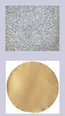
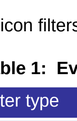
Microplastic particles are typically isolated by filtration.

The choice of filter material should allow for direct analysis.

Considerations for evaluation of the different type of filter materials (Table1):
- Compatible with both FTIR & Raman (one type of filter for both)
- Provide a good visual image (essential for image analysis)

Silicon filters were selected as the best overall choice.

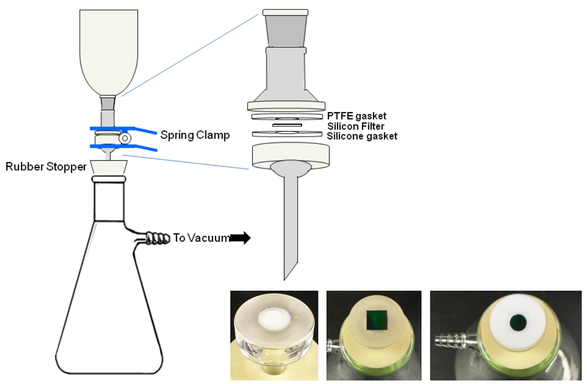
Table 1: Evaluation of common types of filters

Filter type	Advantages	Disadvantages	FTIR	Raman
Gold coated Polycarbonate 	Readily available	Does not lay flat Expensive	Good choice for reflection	Possible to see polycarbonate peaks through gold Some broad baseline offset with some lasers
Silver 	All metal Less expensive than gold coated	More rigid than gold coated PC More of a textured surface at high magnification More reactive surface - reported problems with pH of carbonated water	Reasonable for reflection – less reflective than gold	Some spectral artifacts from filters themselves (highest with 20X but less at higher magnification)
Al_2O_3 	Readily available More rigid Transmitted light possible if intense enough Less expensive option	Delicate – easily broken Visual images – contrast an issue – surface not clearly defined. Some features on surface that might be detected as particles	Can be used in transmission but limited to > 1250 cm^{-1} Some spectral peaks and some variation in peaks over the filter. Reflection weak	Some Raman spectral contributions from the filters – broad features Baseline offsets Laser light transmits through
Silicon 	Rigid Good visible images	Square Needs gasket development Fragile Expensive	Transmission Some variation across filter (filter background: (Si-O)) – broad baseline offset Reflection not as good as gold	Silicon peaks

MODIFICATIONS TO FILTERING APPARATUS

Needed to adapt the filtering apparatus to accommodate for square silicon filters. Added gaskets to cushion the brittle silicon filters and to adapt the shape to the glassware (see Figure 2).

Figure 2: Modifications to the filtering apparatus to support square silicon filters



Filter Sample: Example: 500 ml bottle of drinking water

Ready for Spectroscopic Analysis

WORKFLOW FOR SPECTROSCOPIC ANALYSIS

Workflow is similar for FTIR and Raman Analysis (See Figure 3)

Step 1: Collect Visual Mosaic Image

Step 2: Selection of particles
Adjust sensitivity – contrast differences
Adjust size sieve

Step 3: Automatically collects spectra from targeted particles

Step 4: Report Results
Size and shape from image analysis
Identification from FTIR or Raman Spectra
Most particles are not microplastics (protein, minerals, biological, etc.) – identify which ones are microplastics

Figure 3: Particle analysis workflow

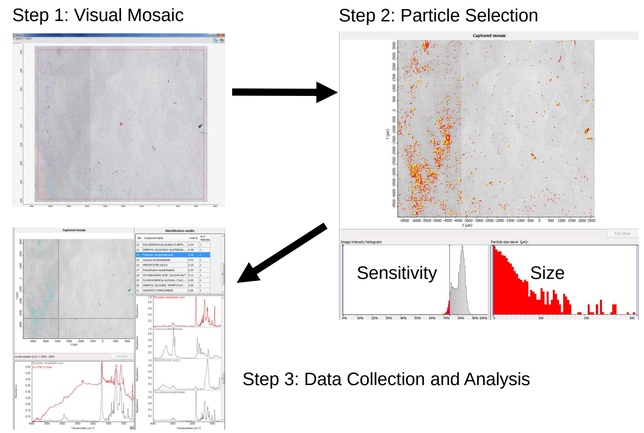


Table 2: Summary of Microplastic Results

Type of microplastic	FTIR (total particles – 801)	Raman (total particles 1065)
PTFE	5	9
Polyester (PET)	3	3
Polystyrene	Not observed	3
Polypropylene	3	3
Unspecific long chain aliphatic hydrocarbon containing materials	2	1
Polyethylene	2	1

CONCLUSIONS

FTIR and Raman spectroscopy are techniques that are well suited for the identification of different types of polymers and micro-spectroscopy allows for the analysis of very small particles. The selection of an appropriate filter material is an important consideration for facilitating the direct FTIR and Raman analysis of particles on the filter. Silicon filters combine good visual properties with suitable spectroscopic properties. Image analysis of visual images affords a basis for targeting particles and determination of size and shape while FTIR and Raman spectral analysis identifies the particles. Overall this approach provides a good approach for the characterization of microplastic particles.

TRADEMARKS/LICENSING

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