# Microplastics in the Environment

## Selecting Membrane Filters to Detect Microplastics in High-Particulate Environmental Matrices

Lindsay D. Lozeau, Kevin Sydlowski, Ranjani Muralidharan

04 AUGUST 2025

MilliporeSigma is the U.S. and Canada Life Science business of Merck KGaA, Darmstadt, Germany.

# Science & Lab Solutions - Biology

# Filtration and Sample Preparation across Environmental Workflows

**Sample Collection**

**Accessories**

**Sample Preparation**

**Sample Prep**

**Solvents & Reagents**

**Sample Analysis**

**Columns**

**Reference Standards**

Analytical Sample Preparation

## Nonsterile Millex® syringe filters

## Cut Disc Filter Membranes

## Analytical Equipment and Hardware

**General Filter Holders & Supporting Equipment**

**Specialized Hardware**

# EMERGING CONTAMINANTS

NATIONAL OCEANIC AND ATMOSPHERIC ADMINISTRATION
NOAA
U.S. DEPARTMENT OF COMMERCE

**2015** **2018** **2020** **2023**

# MICROPLASTICS

# Analytical Methods
# **Published and In Process**

*Updated as of March 2025*

| **Name** | **Date** | **Portion of Workflow** | **Matrix** | **Sample Prep** | **Analytical Method(s)** |
|---|---|---|---|---|---|
| NOAA NOS-OR&R-48 | JUL 2015 | Entire workflow | Seawater, sediment, bed samples | Sieve, density settle & digest | **Microscopy** |
| ASTM D8332-20 | AUG 2020 | Sampling | Drinking water, surface water, wastewater influent, effluent, marine waters | Sieve | **Py-GC/MS**, **IR** or **Raman** Spectroscopy, **Microscopy** |
| ASTM D8333-20 | AUG 2020 | Sampling | Drinking water, surface water, wastewater influent, effluent, marine waters | Sieve, wet peroxide oxidation | **Py-GC/MS**, **IR** or **Raman** Spectroscopy, **Microscopy** |
| SWRCB | **NOV 2021**<br>AUG 2022 | Entire workflow | Drinking water | Sieve, filtration, microscopy | **Microscopy**, **IR** or **Raman** Spectroscopy |
| ISO 5667-27 | **MAR 2025** | Sampling | Drinking water, surface water, freshwater, seawater, wastewater & effluents | | |
| ISO 4484-2 | **NOV 2023** | Entire workflow (pt2=analysis) | Textiles in water | | **Micro-FTIR**, **Micro-Raman**, **Microscopy** |
| ASTM D8401 | **APR 2024** | Entire workflow | Drinking water, wastewater, surface water, ground water, marine waters | Sieve, filtration, microscopy | **Microscopy**, **Py-GC-MS** |
| ASTM D8489 | **MAY 2023** | Entire workflow | Water, wastewater (high to low suspended solids) | Sieve, wet peroxide oxidation | Dynamic particle **imaging** |
| ISO 24187 | **SEP 2023** | Entire workflow | Various environmental matrices | Various | Various |
| ISO/WD 24899 | **Draft** | Sampling | Compost (industrial or home) | TBD | TBD |
| ISO/FDIS 16094-2 | **Draft** | 3 parts, sampling through analysis | Clean waters | Sieve, filtration, microscopy | **MicroFTIR, microRaman** |
| ASTM DXXXX | **Working group** | Entire workflow | Influent, Effluent, wastewater, ambient water, drinking water, bottled water | Sieve, filtration, microscopy | **Microscopy**, **IR** Spectroscopy |
| ASTM 67563 | **Draft** | Sampling | Sewage, wastewater effluent | Sieve | N/A |

**Published Method**

**In development**

# Analytical Methods

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|---|---|---|---|---|---|
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| ASTM D8333-20 | AUG 2020 | Sampling | Drinking water, surface water, wastewater influent, effluent, marine waters | Sieve, wet peroxide oxidation | **Py-GC/MS**, **IR** or **Raman** Spectroscopy, **Microscopy** |
| **SWRCB** | **NOV 2021**<br>AUG 2022 | Entire workflow | Drinking water | Sieve, filtration, microscopy | **Microscopy**, **IR** or **Raman** Spectroscopy |
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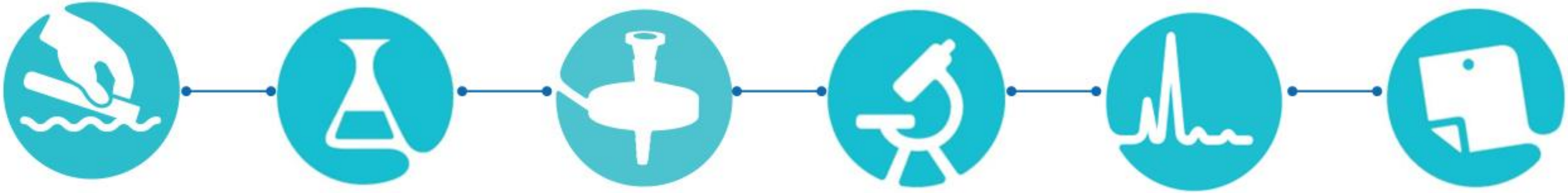
| ASTM 67563 | **Draft** | Sampling | Sewage, wastewater effluent | Sieve | N/A |

**Published Method**

**In development**

# The Microplastics Workflow is **Diverse**



**Sample Collection**

**Sample Preparation**

**Filtration**

- Digestion
- Sieving/filtration
- Density/oil separation

**Visual Identification**

- Microscopy
- Fluorescence
- Electron Microscopy

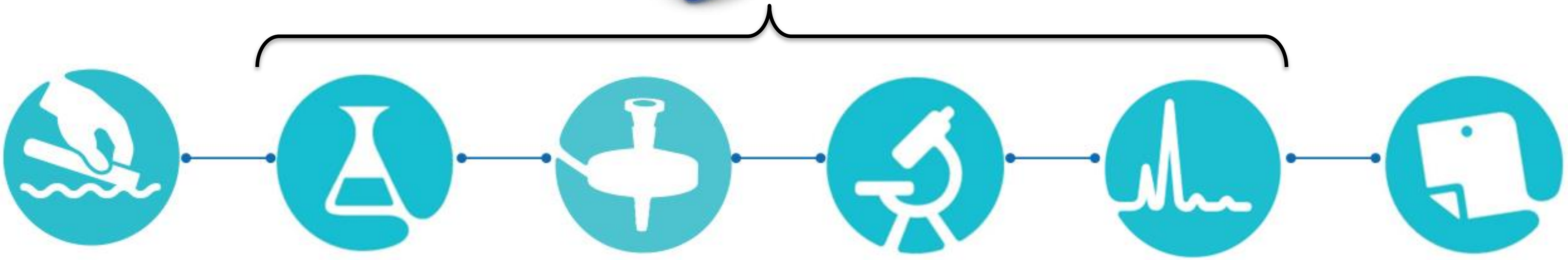
**Spectral or Chromatographic Analysis**

- FTIR spectroscopy
- Raman spectroscopy
- Pyrolysis-GC-MS

**Data Analysis & Reporting**

MilliporeSigma, **2024**. *Membrane filters for microplastics analysis*.

# The Microplastics Workflow is Diverse
## **Filtration Involved in Majority of Workflows**



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MilliporeSigma, **2024**. *Membrane filters for microplastics analysis*.

# One Membrane to Rule them All?

# Microplastics characterization: Where we left off

## Optimizing Membrane Filter Selection

🟢 Recommended
🟤 Recommended, with caveats
🔴 Not recommended

| Technique/Application | Recommended Millipore® Membrane Filter(s) | | | | | |
|---|---|---|---|---|---|---|
| | Glass fiber (GFF) | Quartz fiber (QFF) | Polycarbonate (PC) | Mixed cellulose ester (MCE) | Polypropylene (PP) | Aluminum oxide ($Al_2O_3$) |
| Production of MAG water | 🟢 | 🟢 | 🟢 | 🟢 | N.T. | N.T. |
| Visual analysis | 🟢 | 🟢 | 🟢 | 🟢 | 🟢 | N.T. |
| Nile Red Fluorescence | 🟢 | 🟢 | | | | |
| Drying & Handling | 🟢 | 🟢 | 🟤 | 🟢 | 🟢 | 🔴 |
| Improvement on handling | | | | | | |
| Oil flotation | 🟢 | 🟢 | | | 🟢 | N.T. |
| Salt Separation | | | | | | |
| Chem. digestion/30% $H_2O_2$ | 🟢 | 🟢 | 🟤 | 🟢 | N.T. | 🟤 |
| Chem. digestion/Fenton Rxn | 🟢 | 🟢 | 🟤 | 🔴 | N.T. | 🟤 |
| Chem. digestion/KOH | 🟢 | 🟢 | 🟤 | | N.T. | 🟤 |
| Chem. Digestion + Salt (NaI) | | | | | | |
| Spectroscopy | | | | | | |
| Pyrolysis-GC/MS | | | | | | |

MilliporeSigma, **2024**. *Membrane filters for microplastics analysis*.

# The Microplastics Workflow is Diverse
## Filtration Involved in Majority of Workflows

**Sample Collection**

**Sample Preparation**

**Filtration**

**Visual Identification**

**Spectral or Chromatographic Analysis**

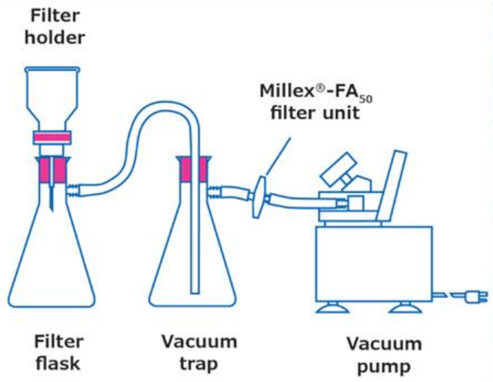
**Data Analysis & Reporting**

**Vacuum Workflow**

*Vacuum filtration hardware, Membrane handling*

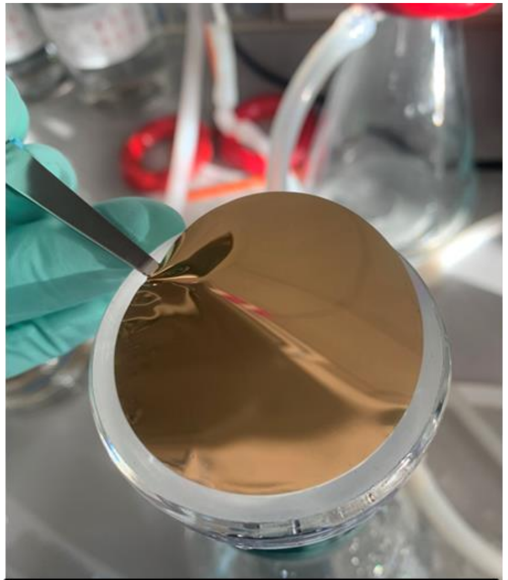
MilliporeSigma, **2024**. *Membrane filters for microplastics analysis*.

# Establishing the Vacuum Workflow to Isolate Microplastics



**Vacuum Setup**

**1**: Wet membrane

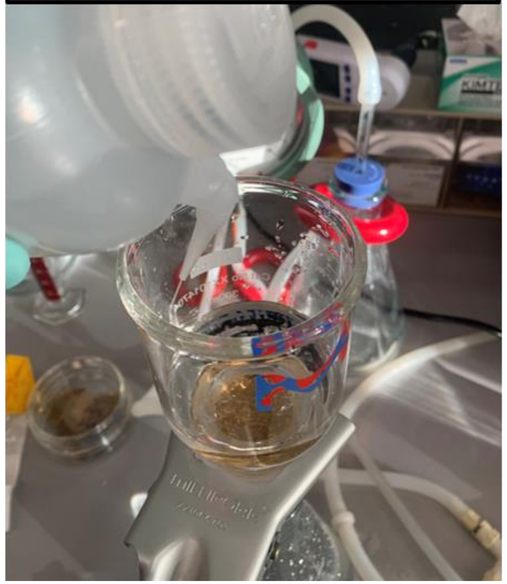


**2**: Place membrane

**3**: Align and clamp

Ways to reduce particle loss →

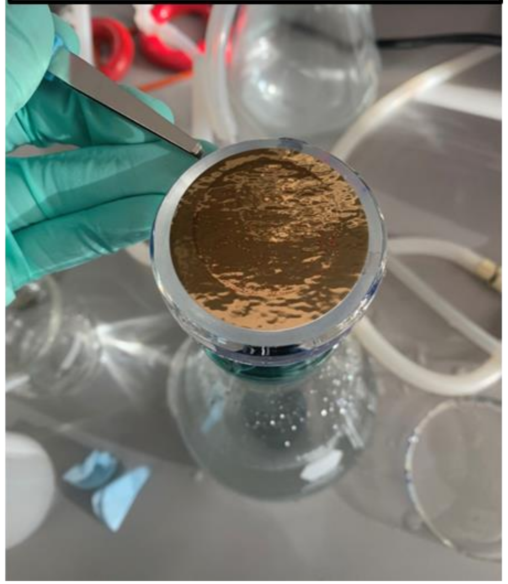
**4**: Filter and rinse



**5**: Remove funnel

**6**: Rinse funnel

**7**: Remove membrane



K. Sydlowski (**2023**)

# Why is the vacuum workflow so important?
## **There are many ways to lose particles**

Without careful consideration of rinsing vacuum equipment and drying, particles can be lost (up to 10% per step!)

**Lost particles** = underestimation of particle contamination

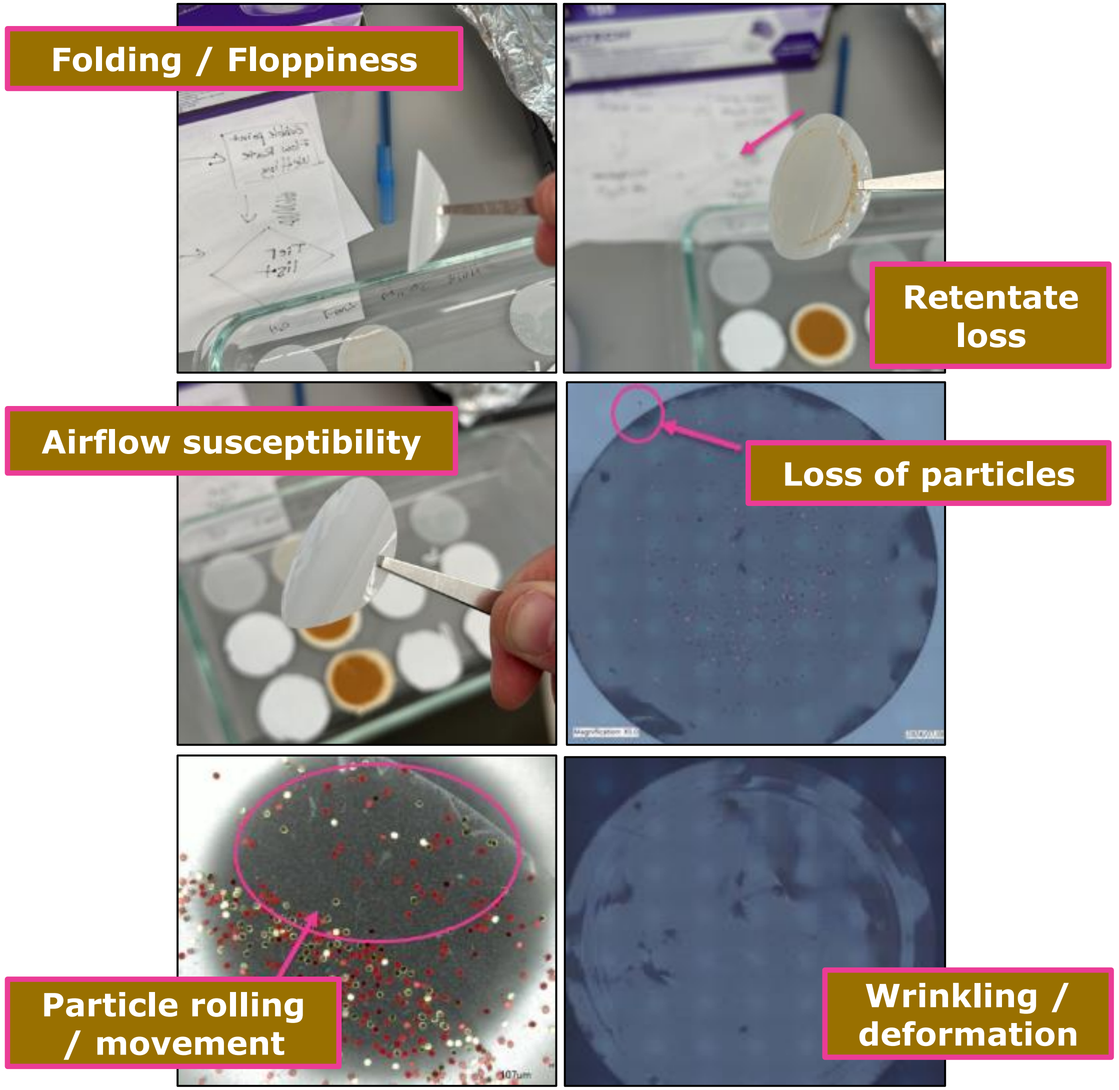
# Why is the vacuum workflow so important?
# **There are many ways to lose particles**

**Polycarbonate** (PC) is popular, but…

Without careful consideration of rinsing vacuum equipment and drying, particles can be lost (up to 10% per step!)

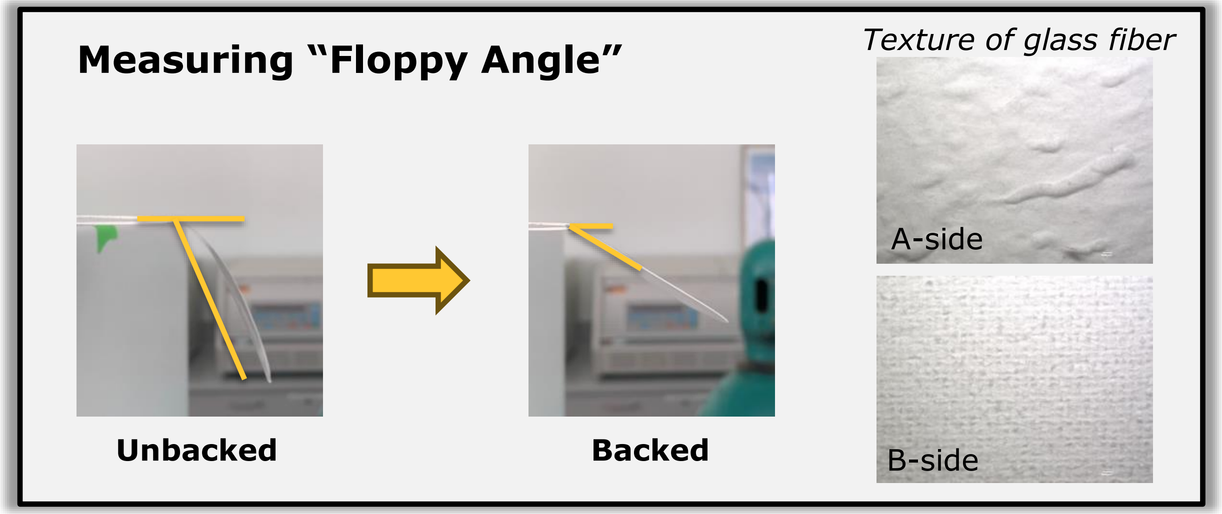
**Lost particles** = underestimation of particle contamination

## Handling PC & other thin filters

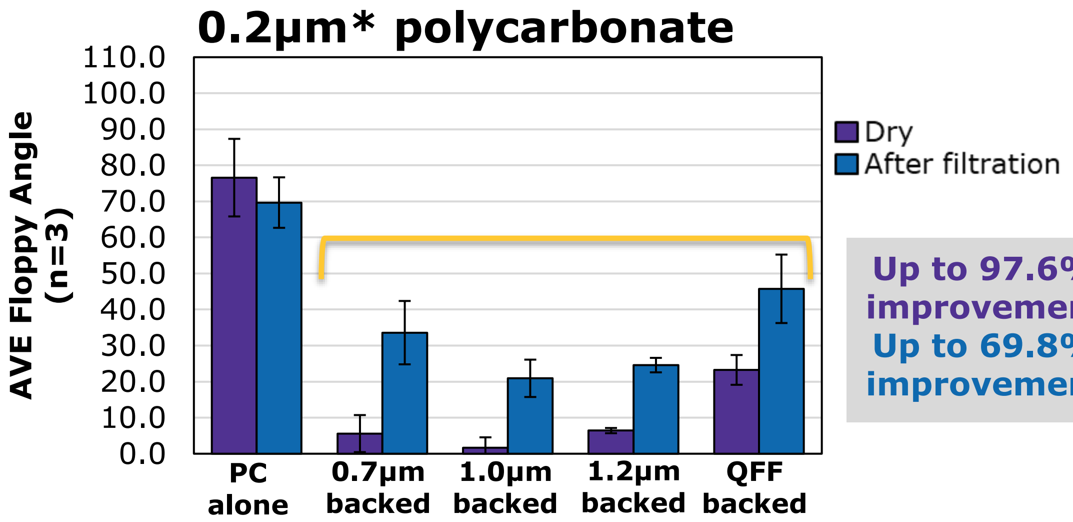


# Are there ways to improve handleability of thin membranes?

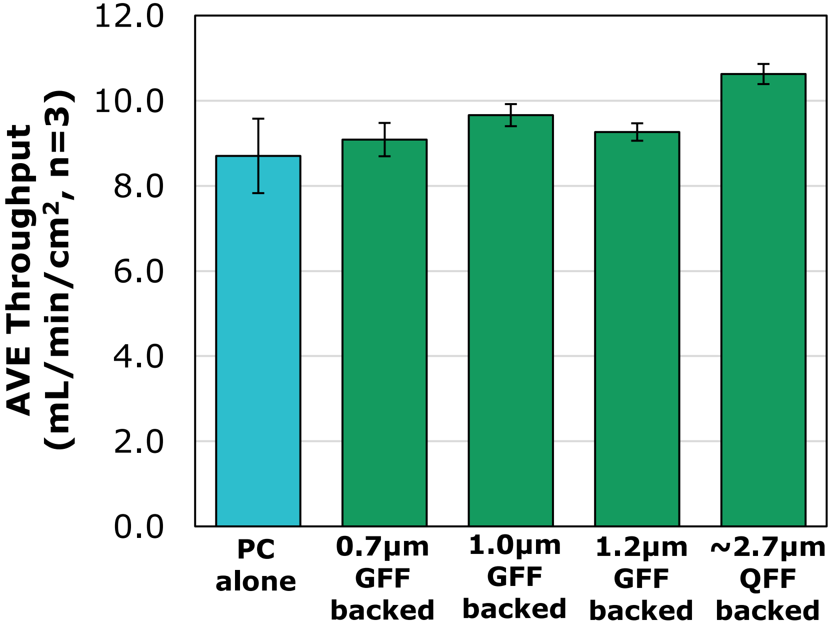
## Backing improves handleability


**Measuring “Floppy Angle”**

*Texture of glass fiber*



**Up to 97.6% improvement**
**Up to 69.8% improvement**



- All glass fiber and quartz fiber “backers” **significantly improved the handleability** of plain polycarbonate
  - ➢ GFF better “backer” than QFF
  - ➢ Recommend backing with the B-side
- Backing **did not significantly impact flow rate** for polycarbonate membranes (influenced by thickness)
- Handling closer to the funnel mark also improved handleability (*data not shown*)

*similar trends seen for 1.2µm and 10µm PC

# Does vacuum equipment matter?
## **Do certain filtration setups facilitate better microplastics collection?**

### **Types of filtration hardware**

**47mm**


Glass base


PTFE base


Hydrosol


Plain funnel


Millicup - FLEX

**25mm**


Glass base


Plain funnel

**13mm**


Epi-fluorescence

# Does vacuum equipment matter?
# **Do certain filtration setups facilitate better microplastics collection?**

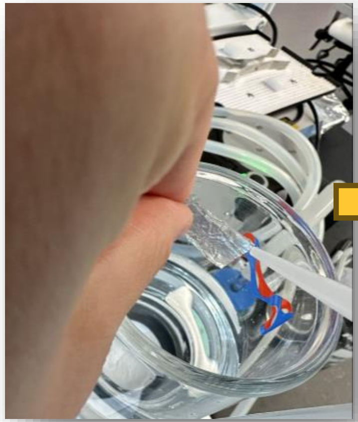
## Types of filtration hardware

**47mm**

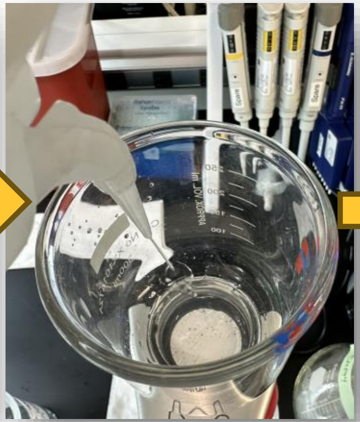
**25mm**

**13mm**

## Determine the number of collected AND lost particles

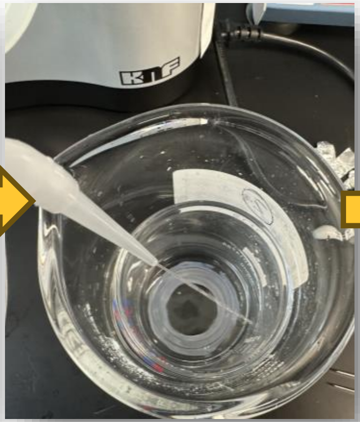


**1:** Spike with glitter particles



**2:** Rinse inside only; collect membrane

**3:** Remove funnel & place in water



**4:** Thoroughly rinse funnel in the water

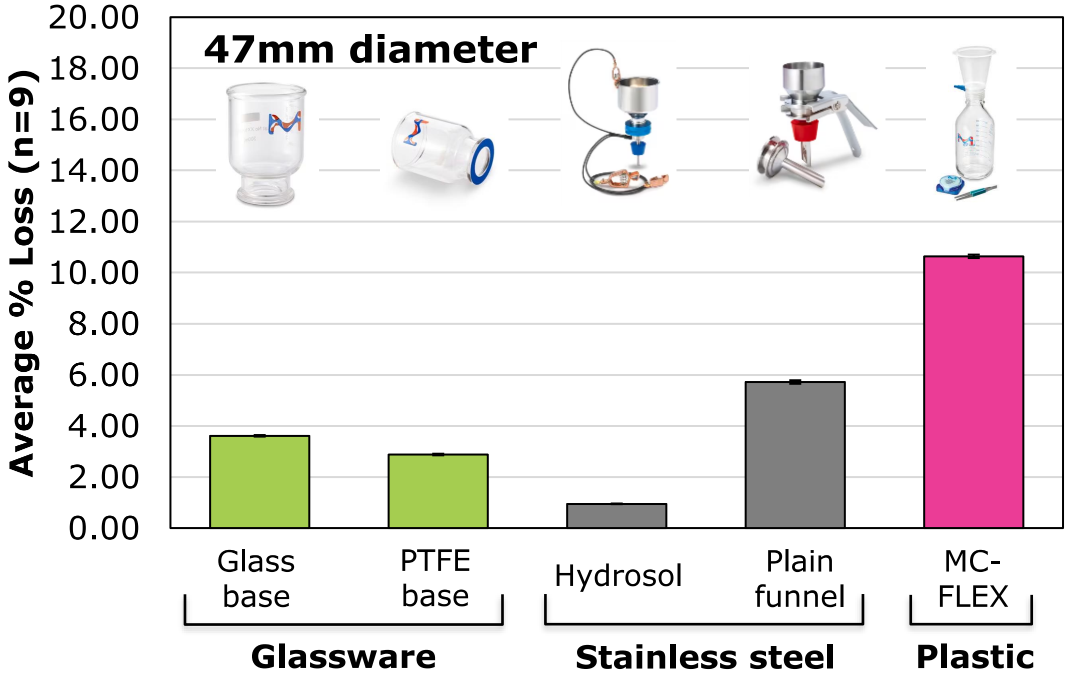
**5:** Filter all the water onto a GFF

**6:** Count particles on both membranes

# Microplastic recovery of different filtration systems

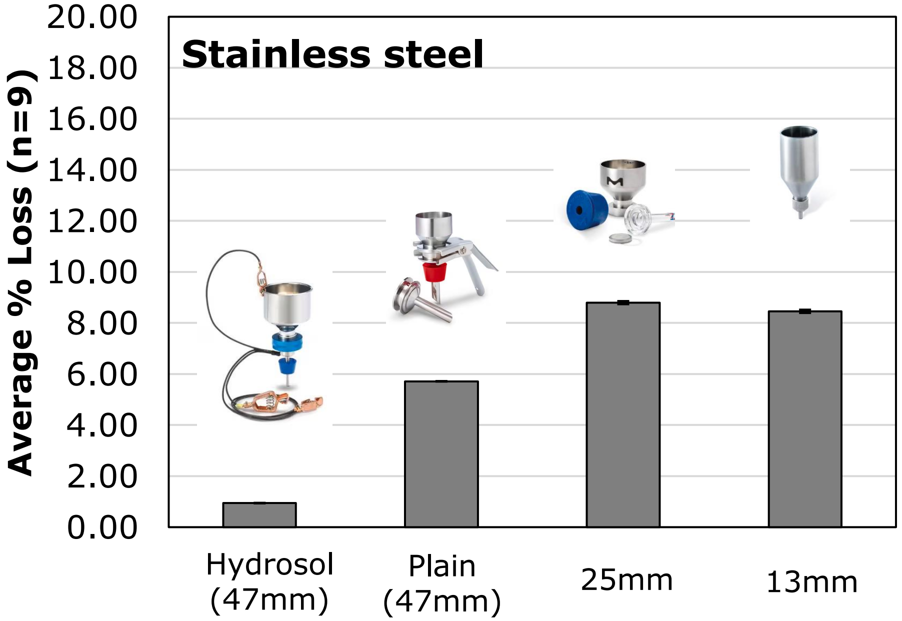
# **Do certain filtration setups facilitate better microplastics collection?**

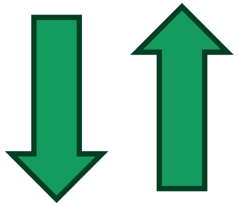
## **Impact of Material**



- **Glassware**: Strong clamp
- **PTFE liner**: Reduction of glass-glass interactions
- **Hydrosol**: Locking seal prevents capillary action
- **Millicup-FLEX**: “Shelf” design improves seal but lodges particles underneath

## **Impact of Equipment Diameter**





Diameter

% Loss of particles

# Microplastic recovery of different filtration systems
# Membrane type impacts recovery & distribution

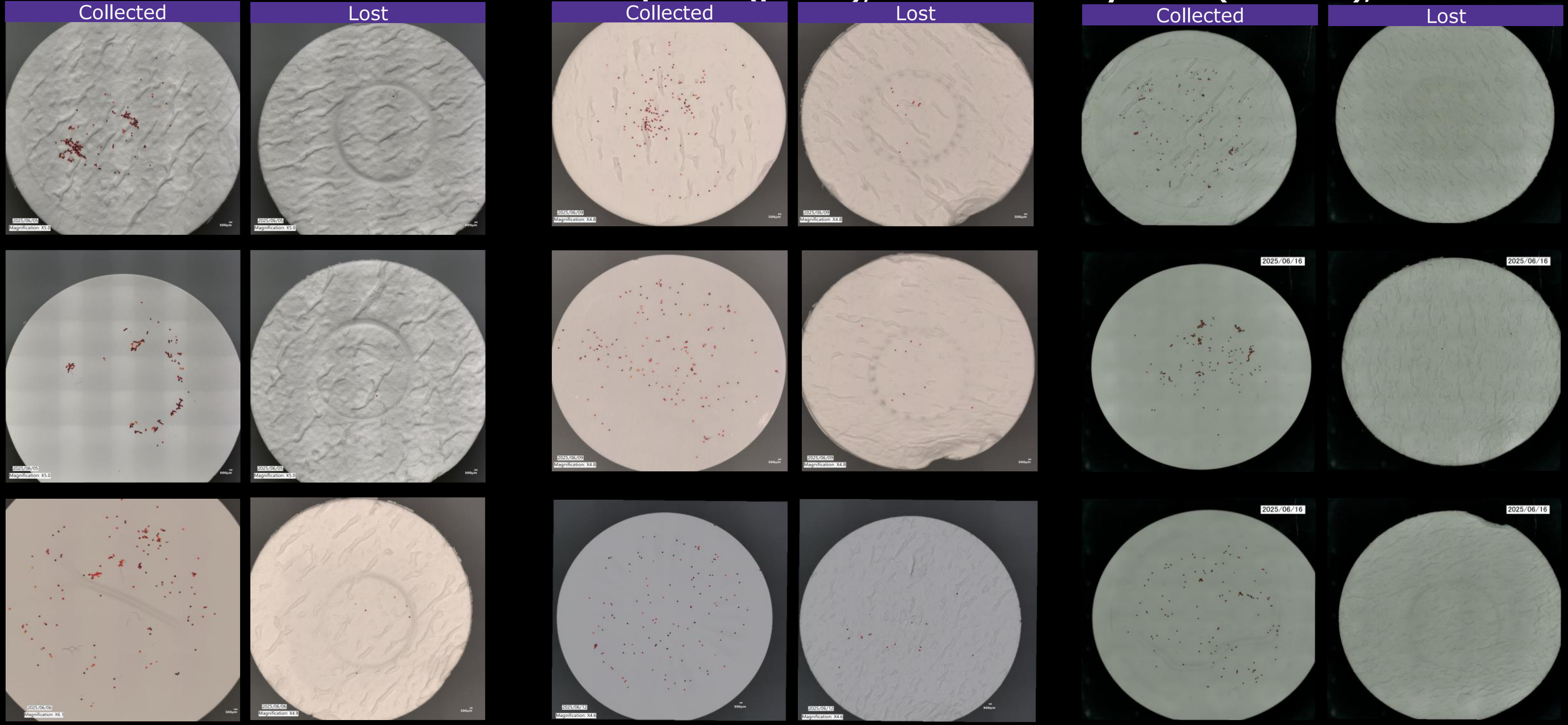
**Glassware (glass), 47mm** | **Millicup-FLEX (plastic), 47mm** | **Hydrosol (stainless), 47mm**

Collected | Lost | Collected | Lost | Collected | Lost

**Glass fiber**

**Polypropylene**

**Nylon**



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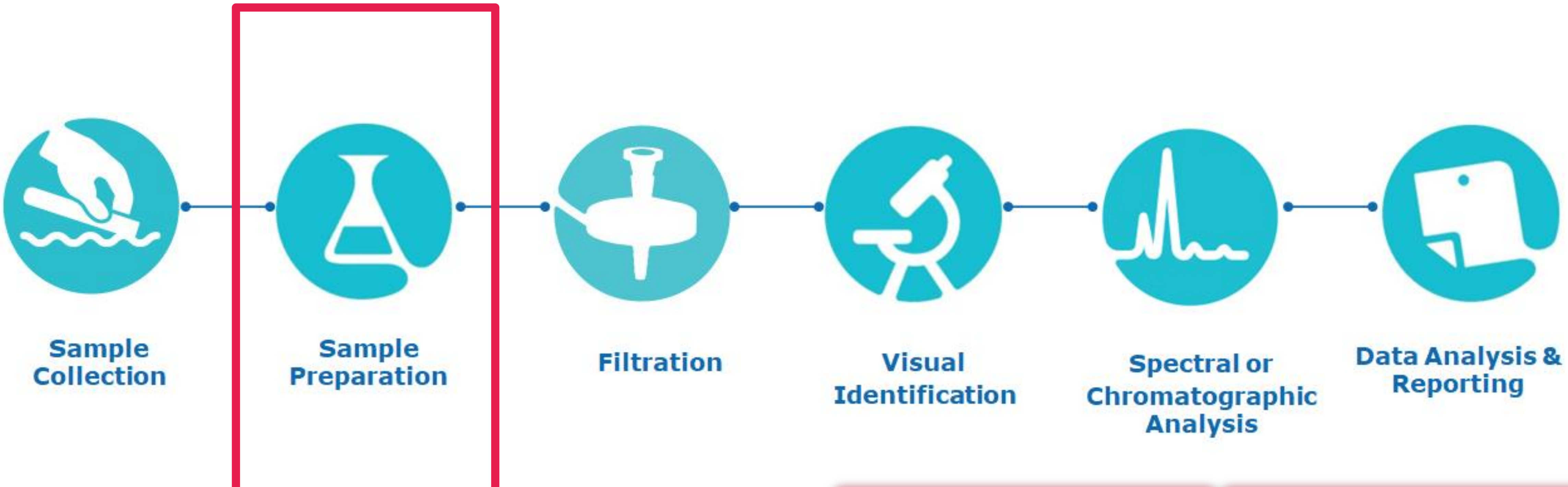
Bar = 500 µm

# The Microplastics Workflow is Diverse
## Filtration Involved in Majority of Workflows



Sample Collection

Sample Preparation

Filtration

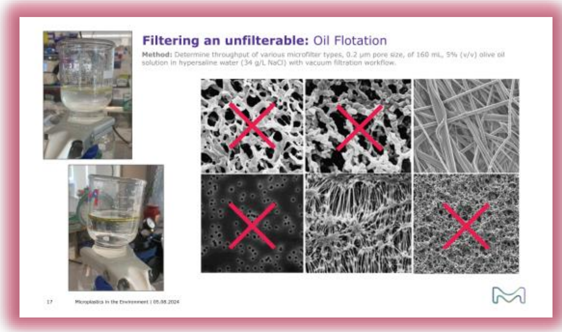
Visual Identification

Spectral or Chromatographic Analysis

Data Analysis & Reporting

**High Particulate Samples**

Separation methods



**Filtering an unfilterable:** Oil Flotation

**Method:** Determine throughput of various microfilter types, 0.2 µm pore size, of 160 mL, 5% (v/v) olive oil solution in hypersaline water (34 g/L NaCl) with vacuum filtration workflow.

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Chemical Digestion & Image Analysis

**Membrane Compatibility – Image Quality, Function & Handleability**

| **Filter Type** | **Water** (control)<br>+ salt separation | **Hydrogen peroxide** (30%v/v)<br>+ NaI separation | **Fenton's Reaction**<br>+ NaI Separation | **Potassium Hydroxide, alkaline** (10%v/v)<br>+ NaI Separation |
|---|---|---|---|---|
| **Glass Fiber** | Recommended | Recommended<br>• *Possible polymer aggregation* | Recommended | Recommended |
| **Quartz Fiber** | Recommended | Recommended<br>• *Possible polymer aggregation* | Recommended | Recommended |
| **Polycarbonate** | Okay<br>• *Scratches* | Not recommended<br>• *Flaking, low retention* | Okay<br>• *Scratches* | Okay<br>• *Scratches* |
| **Mixed Cellulose Ester (MCE) white** | Recommended | Recommended | Not recommended<br>• *Curling/deformation* | Not recommended<br>• *Complete hydrolysis* |
| **Mixed Cellulose Ester (MCE) White/grids** | Recommended | Recommended | Not recommended<br>• *Curling/deformation* | Not recommended<br>• *Complete hydrolysis* |
| **Mixed Cellulose Ester (MCE) black** | Recommended | Okay<br>• *Possible NaI interaction* | Not recommended<br>• *Curling/deformation* | Not recommended<br>• *Complete hydrolysis* |
| **Aluminum Oxide** | Okay *(brittle)* | Okay *(brittle)* | Okay *(brittle)* | Okay *(brittle)* |

K. Sydlowski (**2024**)

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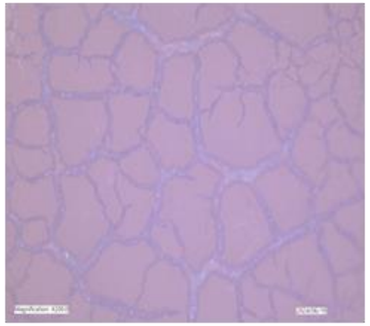
MilliporeSigma, **2024**. *Membrane filters for microplastics analysis*.

# PART 1: Chemical Digestion
## Compatibility after filtering digests

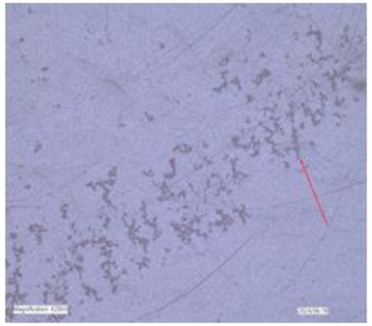
**Method:** Various membranes were used to filter three common the digestion fluids using vacuum filtration [(1) 30% v/v $H_2O_2$, (2), Fenton's reagent – 1:1 30% (v/v) $H_2O_2$ + 0.05 mM $FeSO_4$ in Milli-Q® water, and (3) alkaline – 0.05M KOH]. Filtration was observed. Then, filters were dried in an oven for 1 h at 50°C and imaged, and tested by walking through the lab with forceps (~60 ft).

| **Filter Type** | **Overall compatibility** | | |
|---|---|---|---|
| | **30% $H_2O_2$** | **Fenton Reaction** | **10% KOH** |
| **GFF**, 1.0 µm | Good | Caking | Good |
| **Quartz fiber** | Good | Caking | Good |
| **PC**, 0.8 µm | OK | Flaking | OK |
| **MCE**, 0.8 µm *White/grids* | Good | Flaking | Bad |
| **MCE**, 0.8 µm *black* | Good | Flaking/ sorption | Bad |
| **$Al_2O_3$**, 0.2µm | OK | OK | OK |

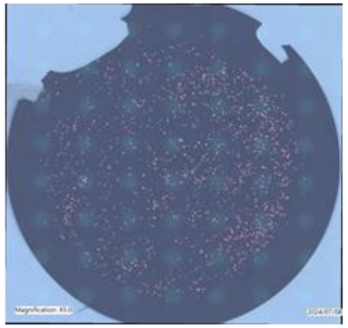
Glass & quartz
(rafts/cakes)



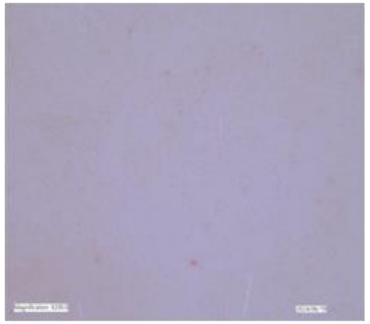
PC 0.8µm
(remnants of flakes)



$Al_2O_3$ 0.2µm
(brittle)

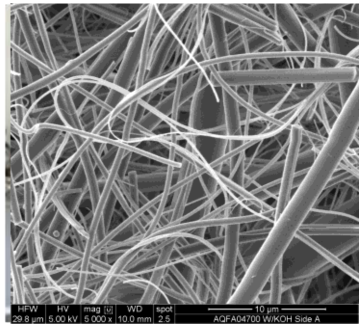


MCE 0.8µm
(orange tint)



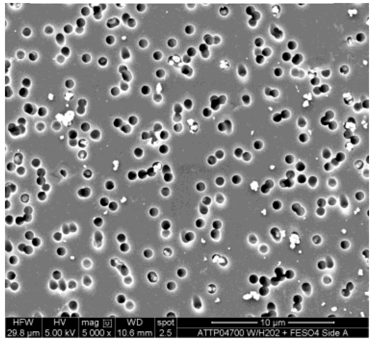
MCE 0.8µm
(hydrolyzed)

Quartz
(fiber shrinking)

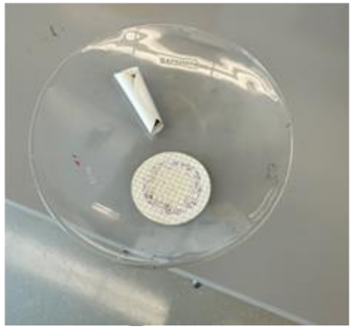


Glass fiber
(adsorbs iron)

PC 0.8µm
(clogging)



MCE 0.8µm
(curling)



K. Sydlowski & David Bell (**2024**)

# PART 2: Salt Density Separation
# **Membrane Compatibility**

**Salts are typically omitted** from chemical compatibility charts[1], but are common for microplastic isolation[2].

**Are common membranes compatible with density separation salts?**

| Reagent | GFF | QFF | MCE | PC |
|---|---|---|---|---|
| $H_2O$[3] | **R** | **R** | **R** | **R** |
| NaCl[3] | ND | ND | **R** | **R** |
| $MgCl_2$ | ND | ND | ND | ND |
| NaI | ND | ND | ND | ND |

*R = recommended; ND = not determined*

**Method**: Flood membranes with NaCl, $MgCl_2$, NaI salts (75% saturation, *aq*) and Milli-Q® water control and let sit for 1 hour at 20°C[4]. Take images on watch glasses after 1 hr and characterize the flow rates of 10 mL Milli-Q® water at 23 inHg.

[1] Prata, J.C., *et al*. *TrAC Trends in Analytical Chemistry*, **2019**, *110*, 150-9.
[2] Soursou, V., Campo, J., Pico, Y. *TrAC Trends in Analytical Chemistry*, **2023,** *166*, 117190.
[3] MilliporeSigma. Membrane Learning Center.
[4] Cutreoneo, L. *et al. Marine Pollution Bulletin*, **2021**, *166*, 112216.

# PART 2: Salt Density Separation

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|---|---|---|---|---|
| $H_2O$[3] | **R** | **R** | **R** | **R** |
| NaCl[3] | ND | ND | **R** | **R** |
| $MgCl_2$ | ND | ND | ND | ND |
| NaI | ND | ND | ND | ND |

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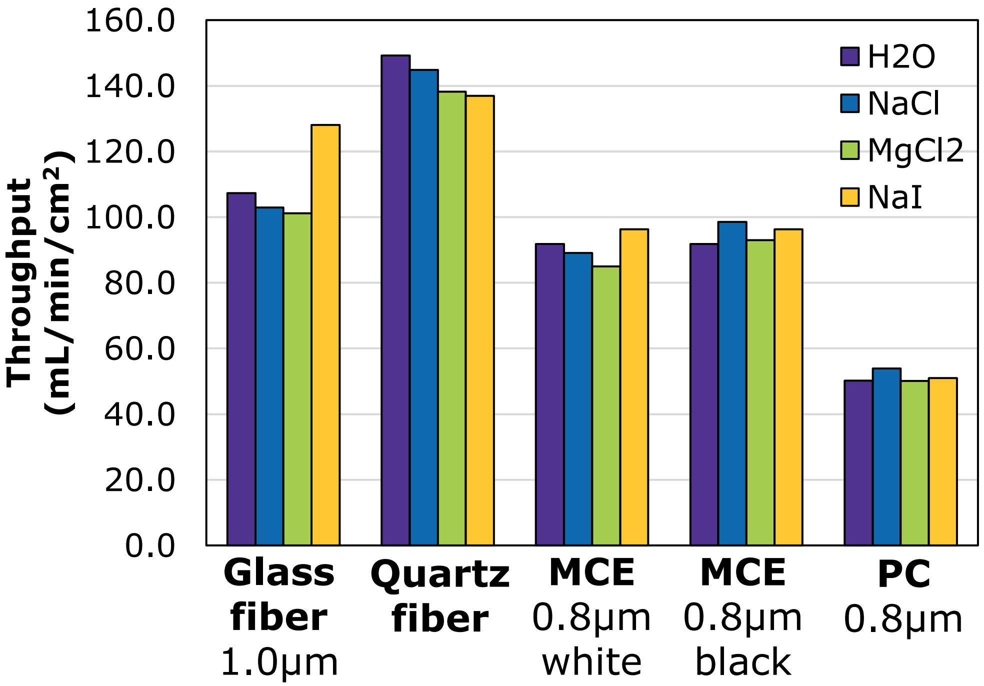
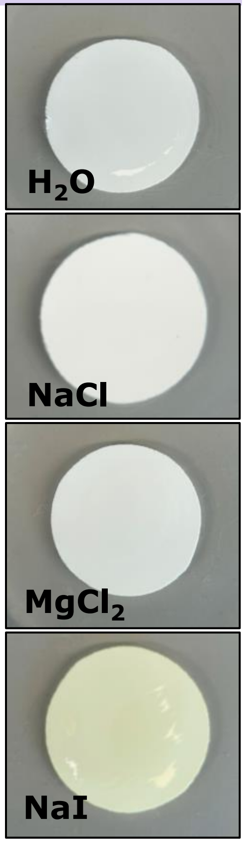
## **Function (flow rate) and morphology**





[1] Prata, J.C., *et al*. *TrAC Trends in Analytical Chemistry*, **2019**, *110*, 150-9.
[2] Soursou, V., Campo, J., Pico, Y. *TrAC Trends in Analytical Chemistry*, **2023,** *166*, 117190.
[3] MilliporeSigma. Membrane Learning Center.
[4] Cutreoneo, L. *et al. Marine Pollution Bulletin*, **2021**, *166*, 112216.

# PART 2: Salt Density Separation
## Membrane Compatibility

**Salts are typically omitted** from chemical compatibility charts[1], but are common for microplastic isolation[2].

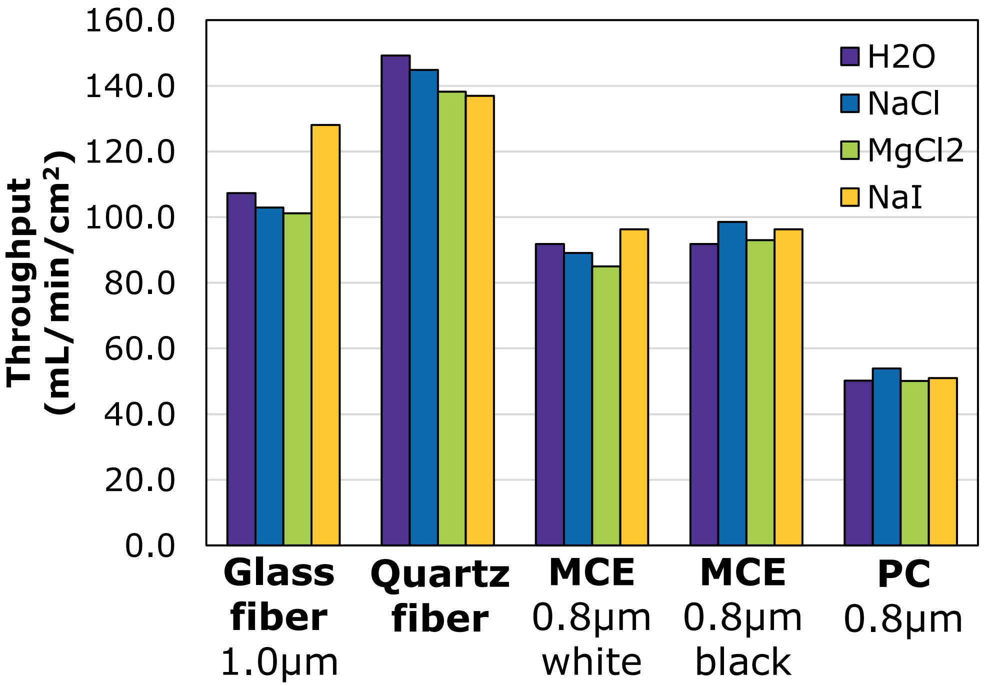
**Are common membranes compatible with density separation salts?**

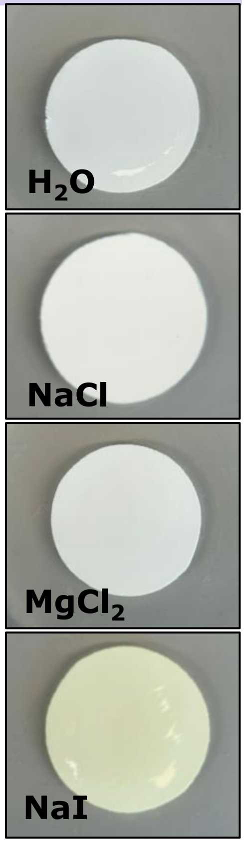
| **Reagent** | **GFF** | **QFF** | **MCE** | **PC** |
|---|---|---|---|---|
| $H_2O$[3] | R | R | R | R |
| NaCl[3] | R | R | R | R |
| $MgCl_2$ | R | R | R | R |
| NaI | R | R | R | R |

*R = recommended; ND = not determined*

**Method**: Flood membranes with NaCl, $MgCl_2$, NaI salts (75% saturation, *aq*) and Milli-Q® water control and let sit for 1 hour at 20°C[4]. Take images on watch glasses after 1 hr and characterize the flow rates of 10 mL Milli-Q® water at 23 inHg.

### Function (flow rate) and morphology





[1] Prata, J.C., *et al*. *TrAC Trends in Analytical Chemistry*, **2019**, *110*, 150-9.
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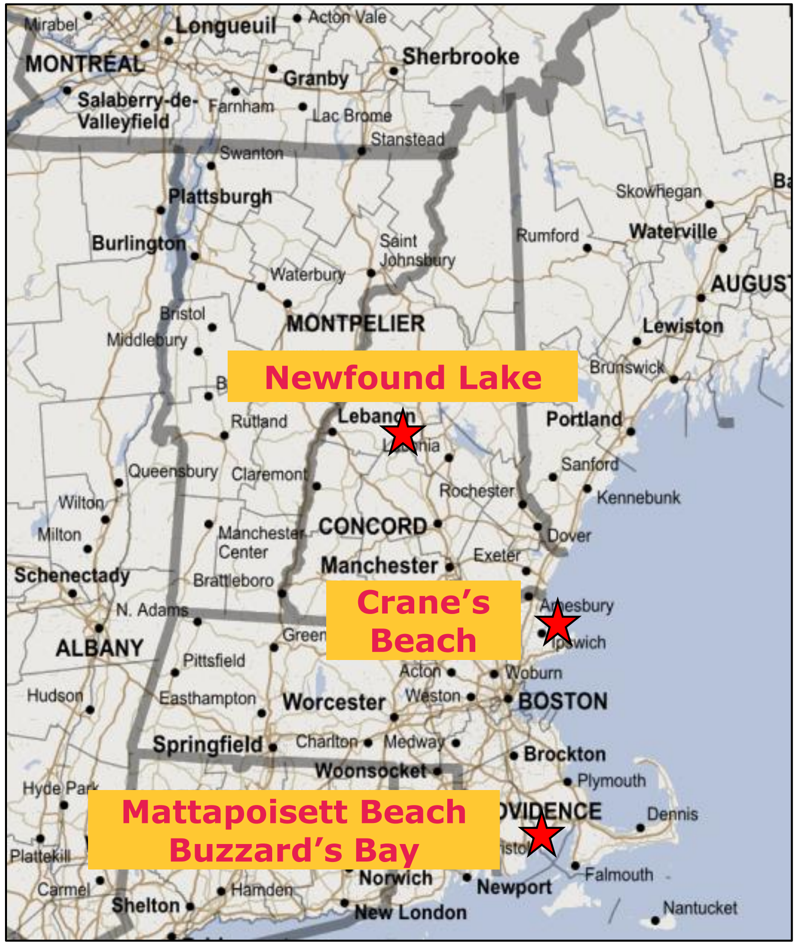
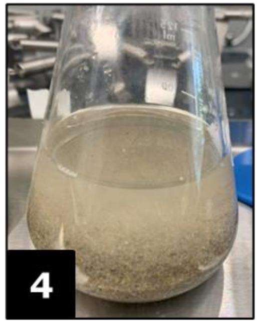
[3] MilliporeSigma. Membrane Learning Center.
[4] Cutreoneo, L. *et al. Marine Pollution Bulletin*, **2021**, *166*, 112216.

# PART 2: Salt Density Separation

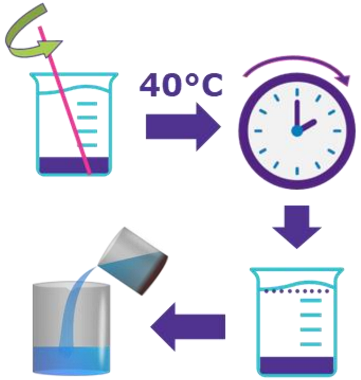
## How successful is density separation for beach sand?





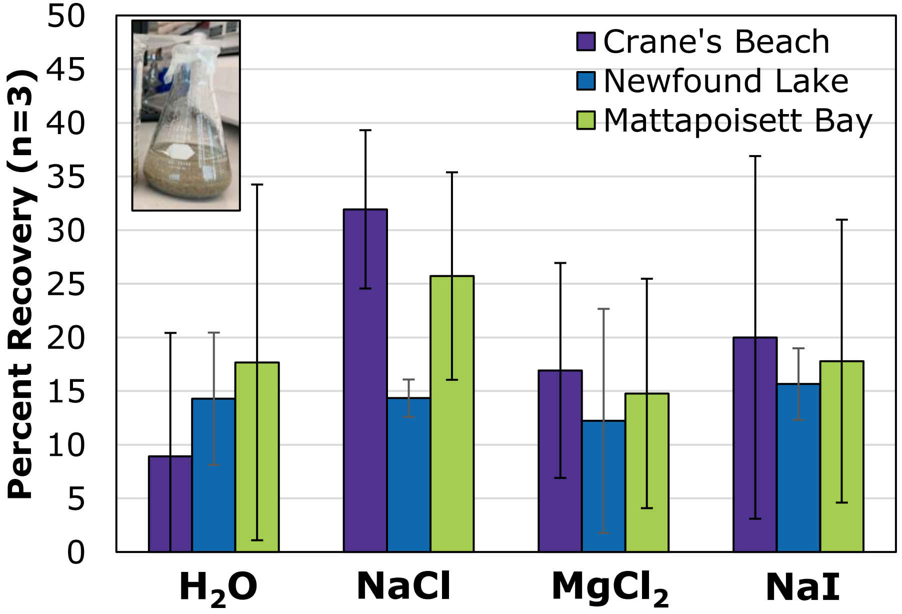
**Density Separation of Beach Sand:**

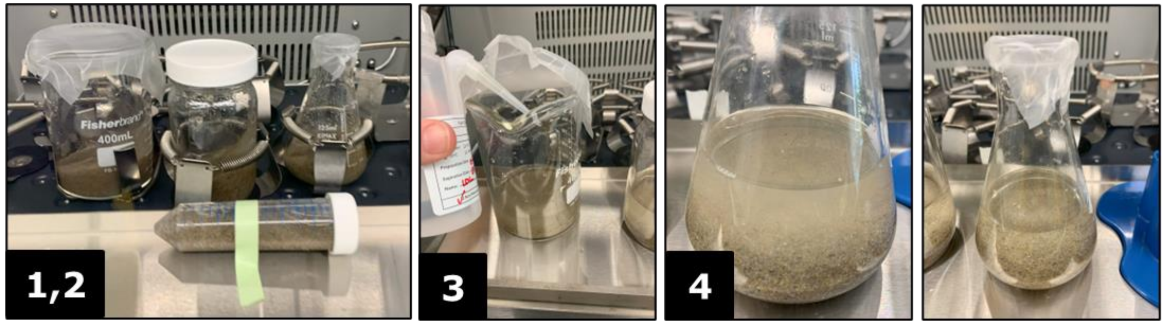
1. Added sand and density separation salt (75% saturated), spike with 0.5-0.7 mg glitter
2. Mixed at 250 RPM, 40°C for >1 hour
3. Rinsed the sides of the container with NaCl
4. Allowed to settle overnight (~16 hours)
5. Pour into vacuum filtration setup, image wet



# PART 2: Salt Density Separation
# How successful is density separation for beach sand?

**Workflow**: Ease of decanting & shaking
**Cross-Contamination**: Least "messy"
**Best Recovery**: Highest surface area





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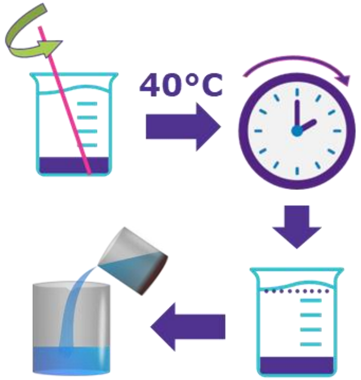

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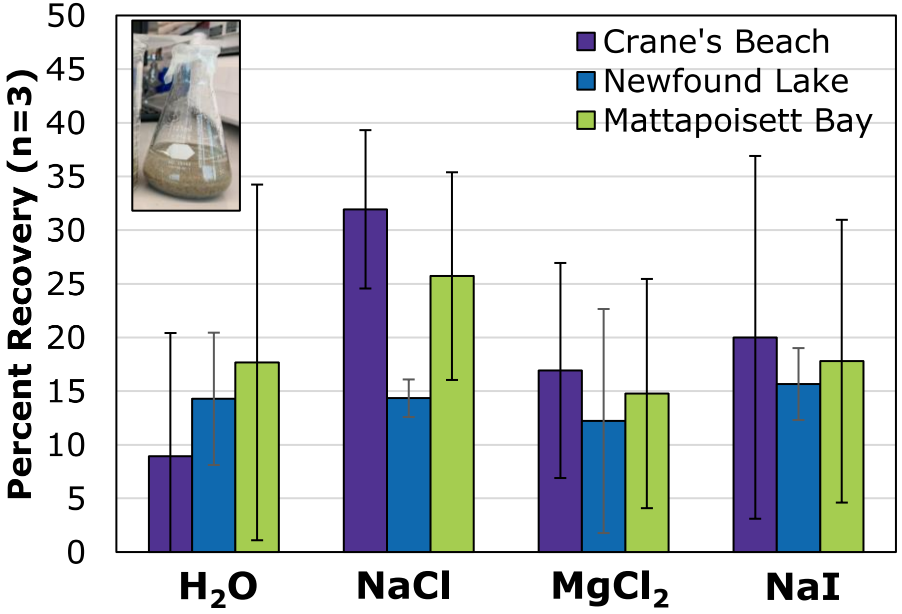

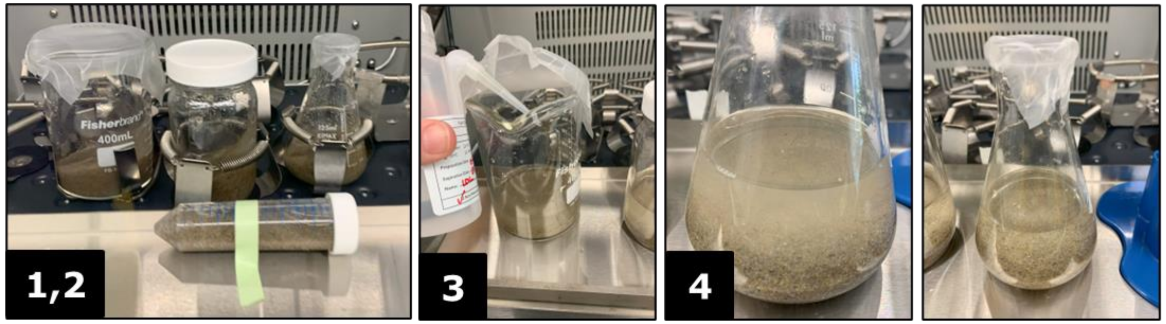

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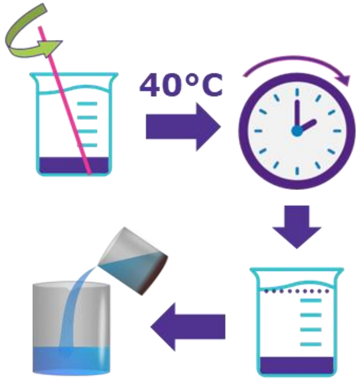
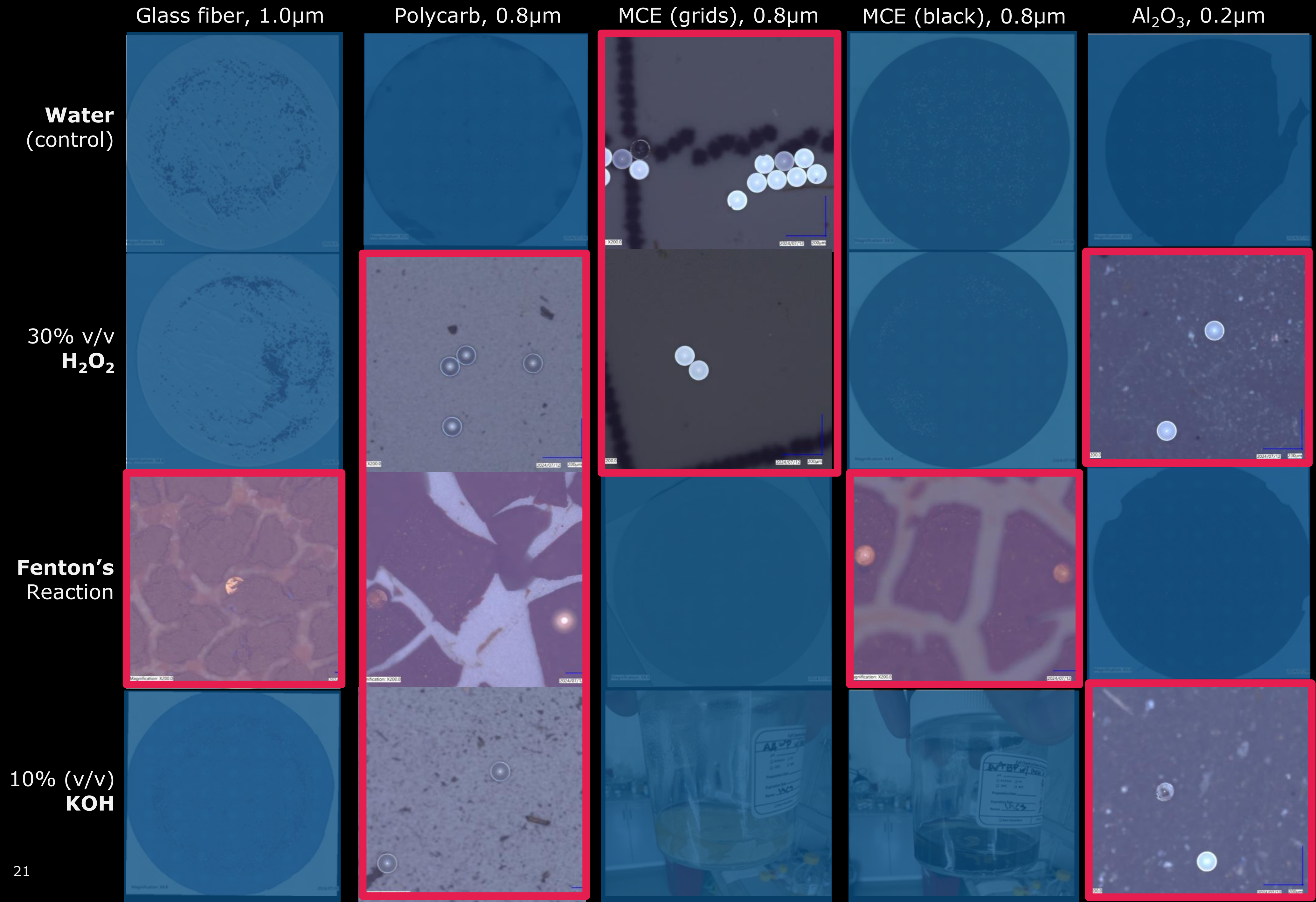


## Combining density separation and chemical digestion methods…

# Chemical digestion & NaI separation with PS Beads

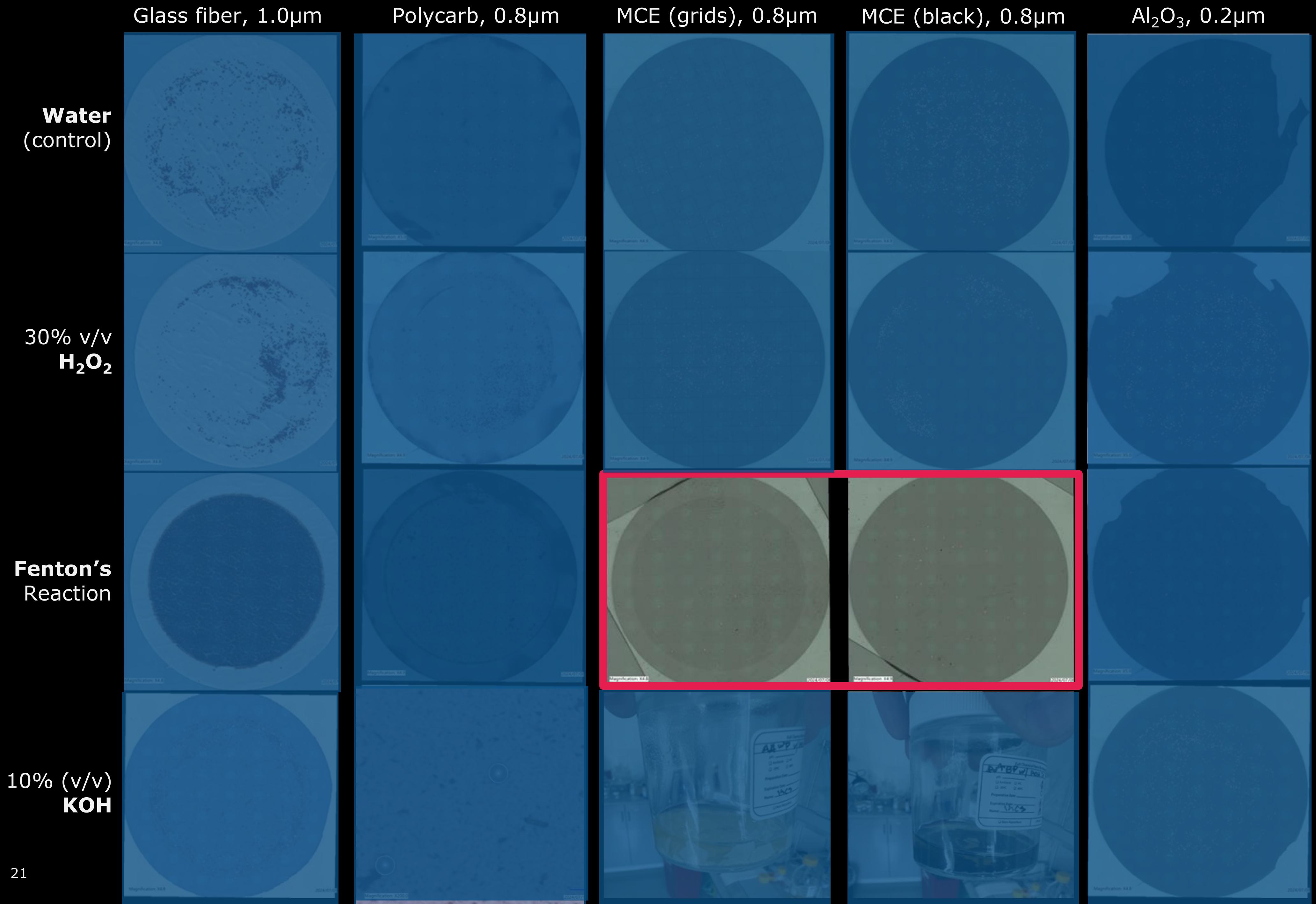


- No damage
- Clear contrast for MCE and $Al_2O_3$
- Beads "stuck" in filter cakes
- Low PC retention

**Method:** Spike NaI solution with 100 µm PS beads, then digest with one out of the three methods. Filter, dry at 50°C, "handle" and image. Mag = 200x; Bar = 200µm.

K. Sydlowski (**2024**)

# Chemical digestion & NaI separation with PS Beads



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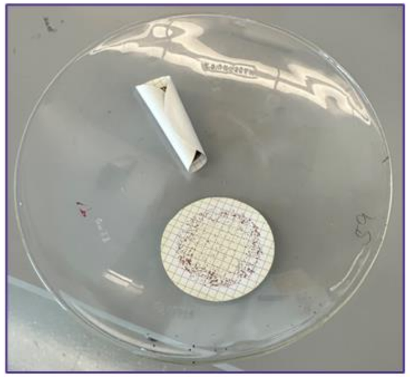
K. Sydlowski (**2024**)

# Chemical digestion & NaI separation with PS Beads

- No damage
- Clear contrast for MCE and $Al_2O_3$
- Beads “stuck” in filter cakes
- Low PC retention

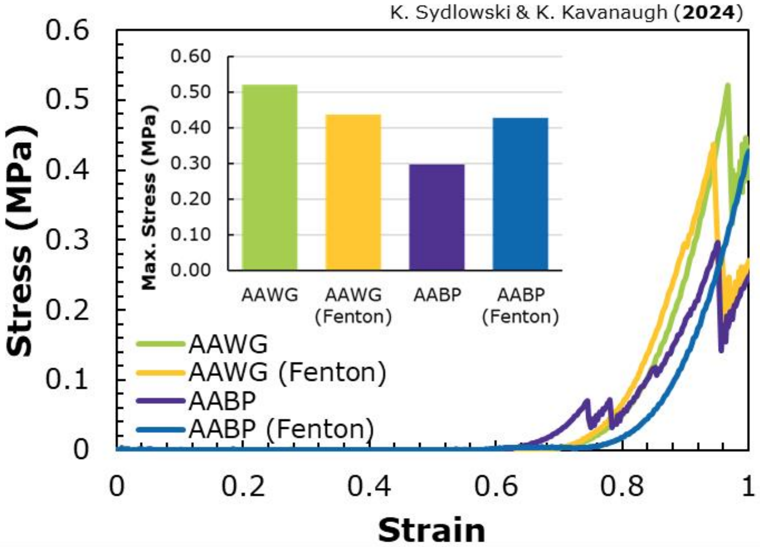
**Unexpected results:**

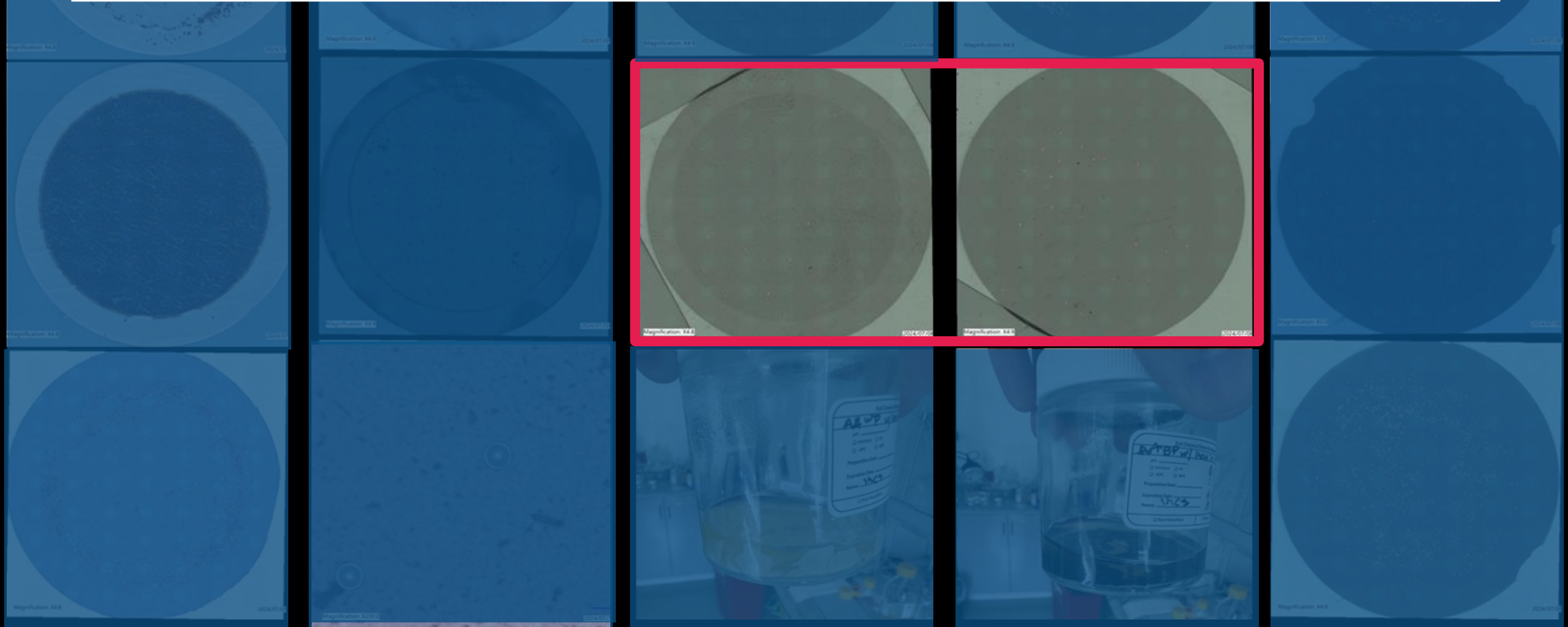
Significant curling with Fenton’s reaction with NaI versus 30% $H_2O_2$ with NaI and those without NaI



**Hypothesis:**

Sodium and iron REDOX reaction with partial negative groups on cellulose acetate


K. Sydlowski & K. Kavanaugh (**2024**)



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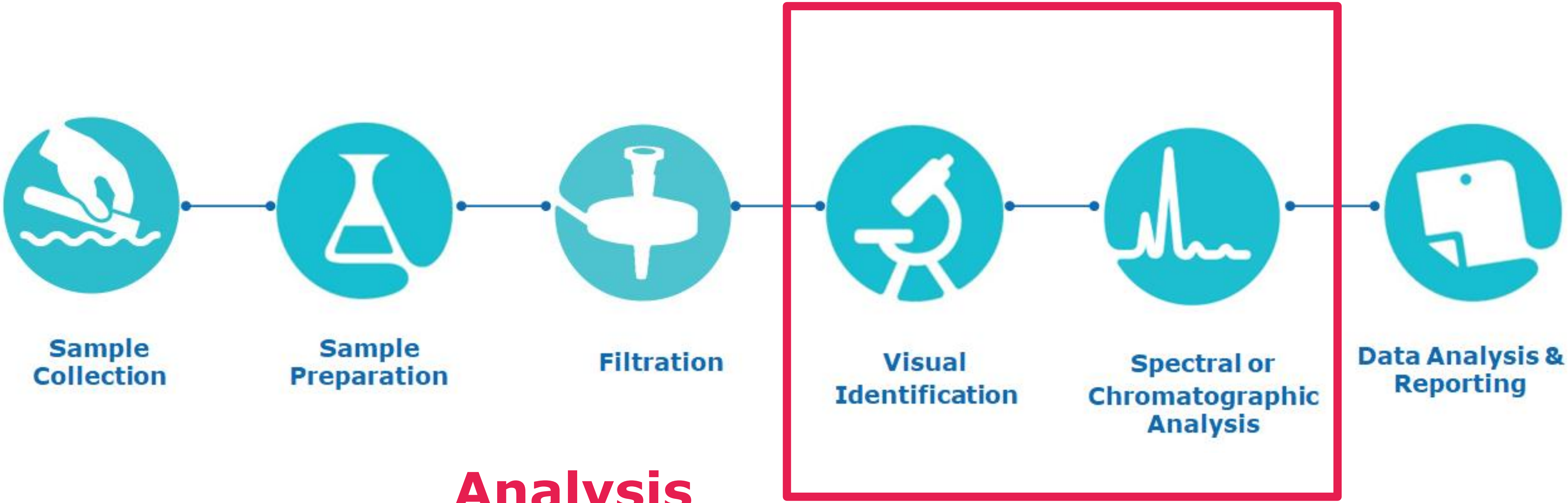
K. Sydlowski (**2024**)

# The Microplastics Workflow is Diverse
## **Filtration Involved in Majority of Workflows**



**Sample Collection**

**Sample Preparation**

**Filtration**

**Visual Identification**

**Spectral or Chromatographic Analysis**

**Data Analysis & Reporting**

**Analysis**

Fluorescence microscopy, Pyr-GC, Spectroscopy

- FTIR spectroscopy
- Raman spectroscopy
- Pyrolysis-GC-MS

MilliporeSigma, **2024**. *Membrane filters for microplastics analysis*.

# Image Analysis

# Fluorescent Detection using Nile Red

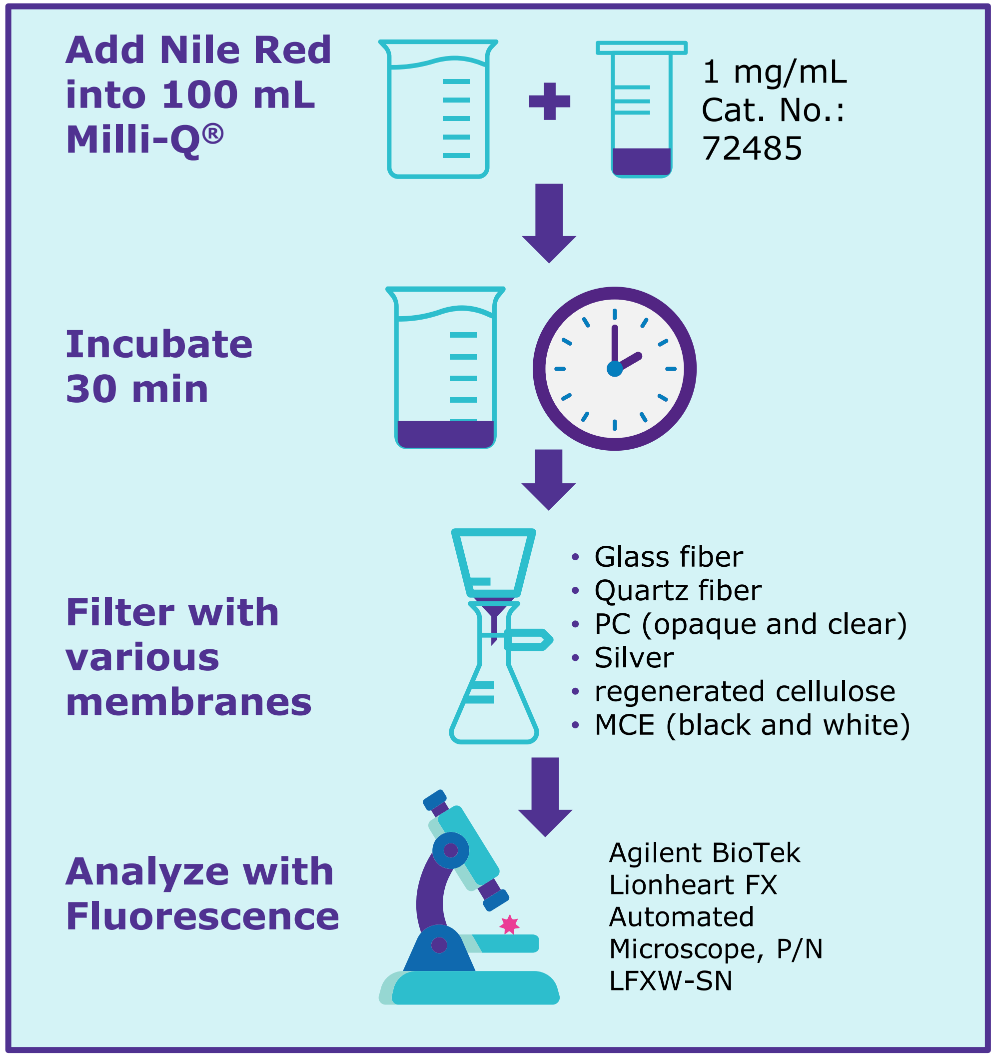
**Nile Red is a common method of detecting microplastics[1]**

Previously, we found that Millipore® glass fiber membranes are suitable for this workflow, and collect more particles in smaller size ranges

## Are there other membrane options that could be used for this method?

| Filter Material | Millipore® Cat. No. | Acetone Compatibility |
|---|---|---|
| Glass fiber (GFF) | APFB04700 | **NR**[2] |
| Quartz fiber (QFF) | AQFA04700 | ND |
| Mixed cellulose esters (MCE) | | |
| *Black* | AAWP04700 | **NR**[3] |
| *White* | AABP04700 | **NR**[3] |
| Regenerated cellulose (RC) | WHA68096022 | **R**[3] |
| Polycarbonate (PC) | | |
| *Opaque* | ATTP04700 | **NR**[3] |
| *Clear* | TCTP04700 | |
| Silver | Z623032 | **R**[2] |

K. Sydlowski (**2025**)



**Add Nile Red into 100 mL Milli-Q®**

1 mg/mL
Cat. No.:
72485

**Incubate 30 min**

**Filter with various membranes**

- Glass fiber
- Quartz fiber
- PC (opaque and clear)
- Silver
- regenerated cellulose
- MCE (black and white)

**Analyze with Fluorescence**

Agilent BioTek Lionheart FX Automated Microscope, P/N LFXW-SN

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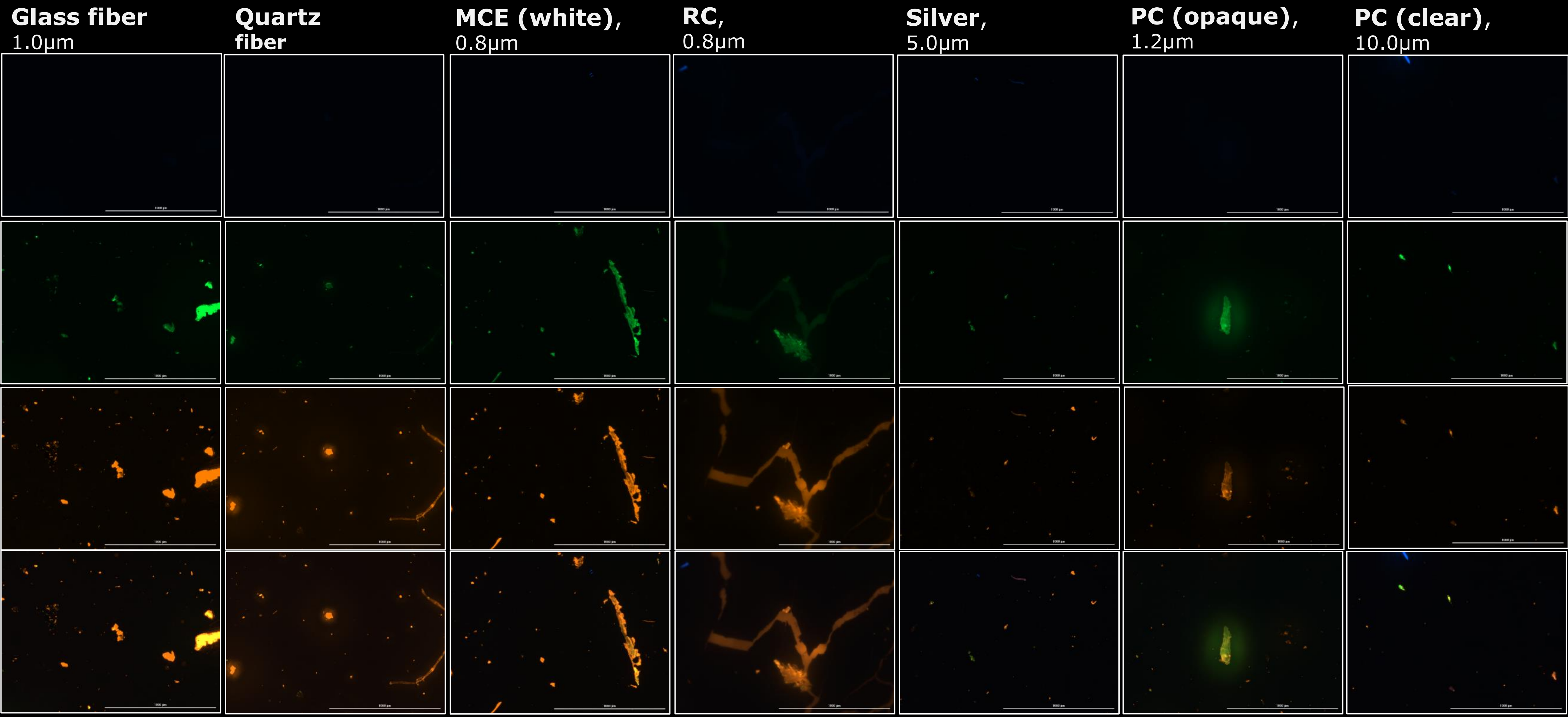
[1] Mason, S.A, Welch, V.G, Neratko, J. *Front. Chem.*, **2018**, *6*, 1-10.
[2] Sterlitech. https://www.sterlitech.com/chemical-compatibility-chart.
[3] MilliporeSigma. https://www.emdmillipore.com/Web-CA-Site/en_CA/-/CAD/ShowDocument-Pronet?id=201510.399&usg=AOvVaw3h0KMcgRcLW-ZMsoV9AlbV.



**Glass fiber** 1.0µm

**Quartz fiber**

**MCE (white),** 0.8µm

**RC,** 0.8µm

**Silver,** 5.0µm

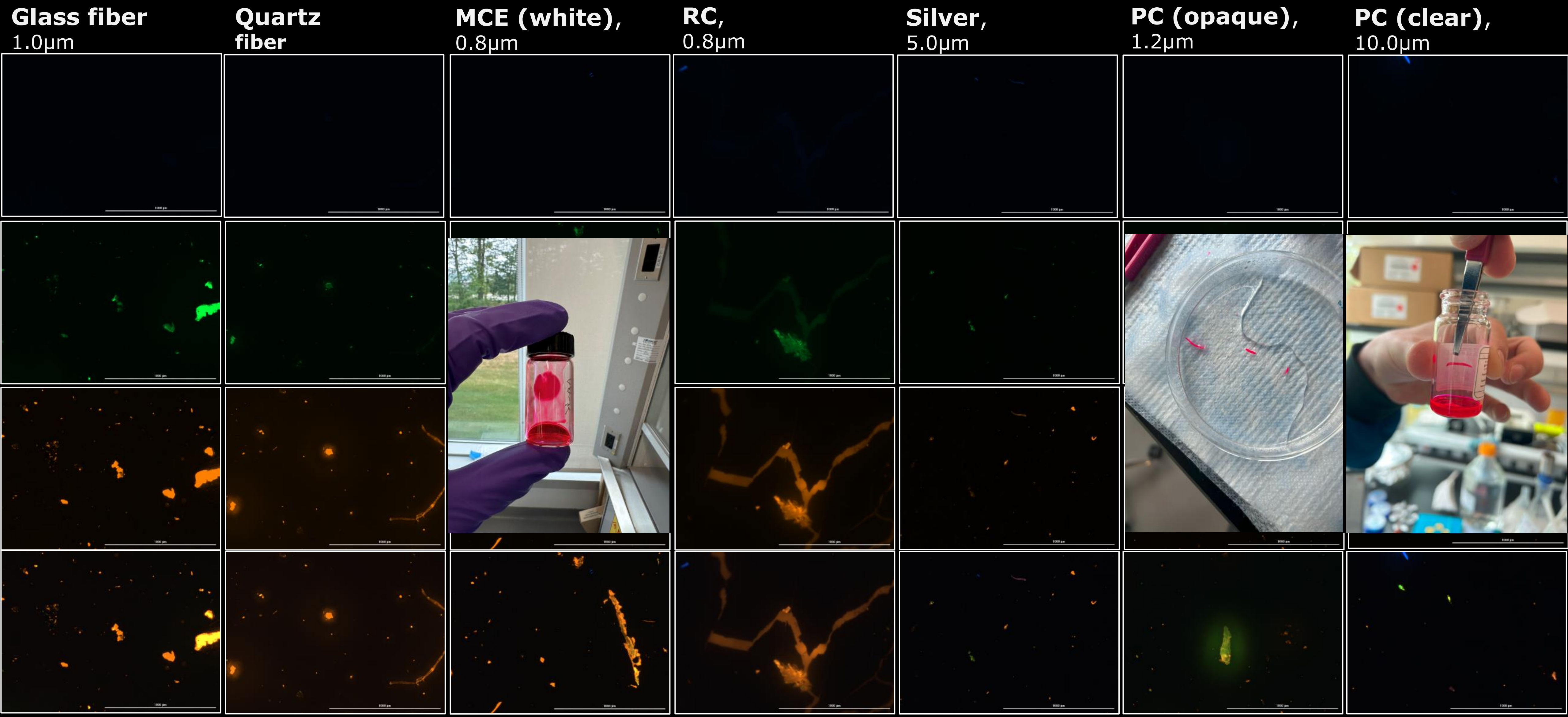
**PC (opaque),** 1.2µm

**PC (clear),** 10.0µm

- Row 1: DAPI (377/447 nm)
- Row 2: GFP (469/525 nm)
- Row 3: RFP (531/593 nm)
- Row 4: Overlay

Bar = 200µm

K. Sydlowski (**2025**)



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- Row 4: Overlay

Bar = 200µm

K. Sydlowski (**2025**)

# Spectral and Chromatographic Characterization
# **Membrane considerations for GC-pyrolysis**

### Filter Considerations for Spectroscopy (IR and Raman)

Spectral interference

  - Signal masking
  - Thickness
- Reflectivity
- Signal enhancement
- Laser compatibility
  - Handleability & fragility
- Disc size & instrument compatibility
- Filter dryness
- Particle size as it relates to particle retention
- Filter availability & cost
- If coated, coating reactivity

### Filter Considerations for GC/MS

- Filter diameter
  - Ability to fit in pyrolysis cup
- Subsampling and/or punching out filter sections
- Sturdiness vs. pyrolysis method
- Low or highly distinguished background from polymers

Filtration is often used as a tool for collection and keeping samples from falling out of pyrolysis cups[1,2]. Common filters are **glass fiber, close to 1 µm**[3] or **quartz fiber**[4,5].

[1] Pico, Y., Barcelo, D. *TrAC Trends in Analytical chemistry*. **2020**, *130*, 115964.
[2] Kappler, A., *et al*. *Analytical and bioanalytical chemistry*, **2018**, *410*, 5313-27.
[3] Fischer, M., Scholz-bottcher, B.M. *Analytical methods*, **2019**, *11*, 2489-97.
[4] ASTM D8401-24. (April 2024).
[5] Stainmetz, Z, *et al*. *Journal of analytical and applied pyrolysis*, **2020**, *147*, 104803.

- Small enough diameters rare = need for subsampling or folding/crumpling membrane
- Membrane should be easy to deform without losing particles

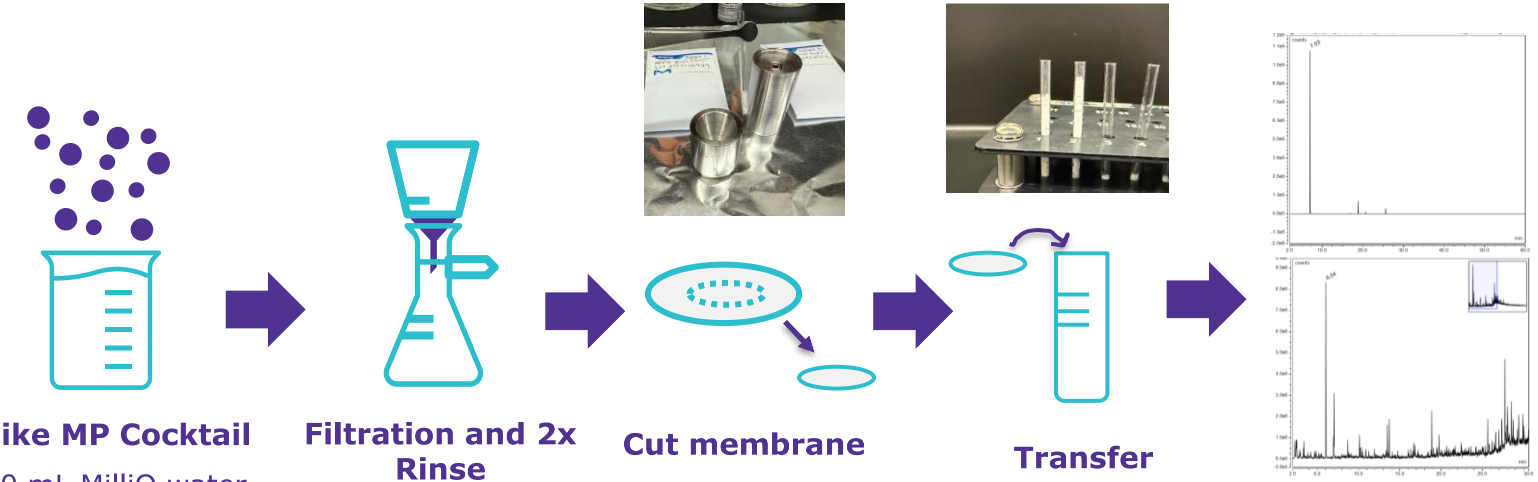
- Inorganic membranes

MilliporeSigma, **2024**. *Membrane filters for microplastics analysis*.

# Microplastics analysis via GC-pyrolysis (ASTM 8401)
## Are glass and quartz fiber suitable?

PennState Institute of Energy and the Environment
Environmental Contaminants Analytical Laboratory

Measurlabs



**Spike MP Cocktail**

500 mL MilliQ water + 0.01% w/v of 1 and 100 µm PS and 1 and 100 µm PP MPs (0.1 mg/mL)

**Filtration and 2x Rinse**

2-3 inHg, 13mm inner diameter on a 25mm disc. Rinse twice with 5mL water

**Cut membrane**

Use circular die to cut 16mm membrane disc

**Transfer**

Move 16mm membrane disc into pyrolysis tube

**Pyr GC-MS**

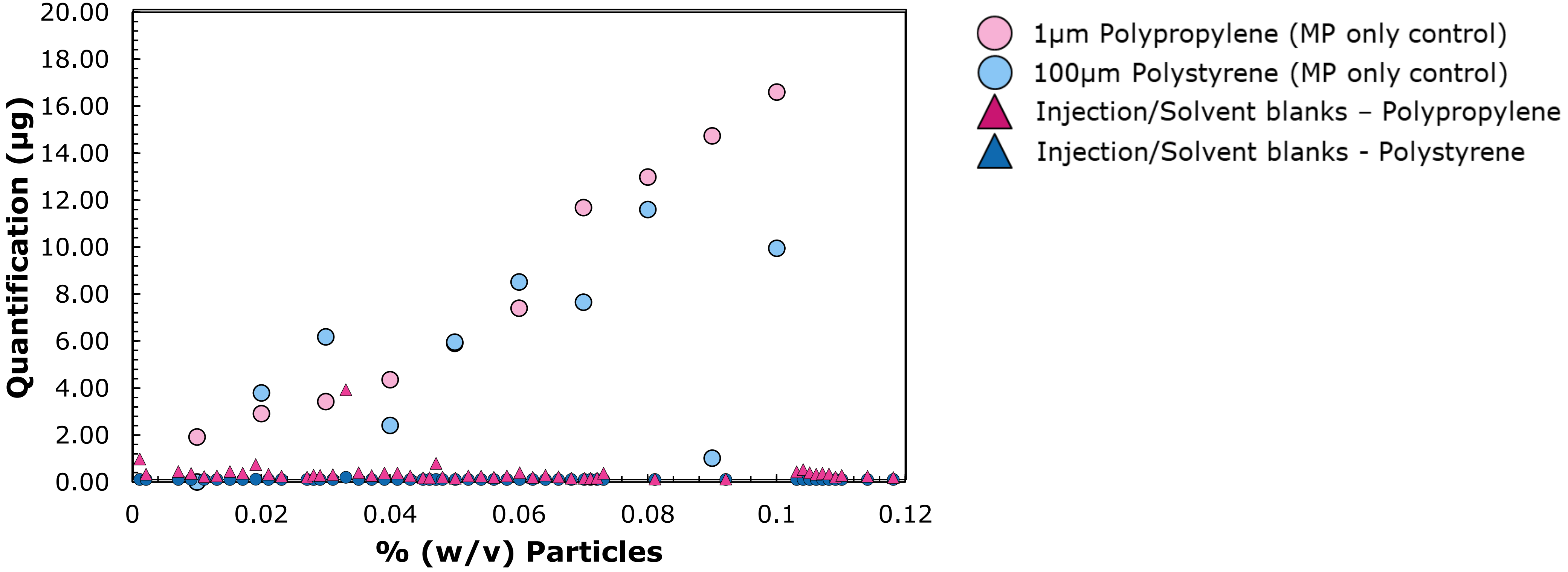
Analyze

| | |
|---|---|
| **Pyrolysis Conditions** | |
| **Instrument** | CDS Pyroprobe 6200 |
| **Clean** | 1000 °C for 20s |
| **Pyrolysis** | 600 °C for 30s |
| **Interface** | 300 °C |
| **Transfer line** | 325 °C |
| **Valve oven** | 300 °C |
| **Flow rate** | 100 mL/min |
| **GC Conditions** | |
| **Instrument** | ThermoFisher Trace 1310 GC |
| **Column** | Supelco® SLB-5ms (30m x 0.25mm x 0.2µm) |
| **Oven** | 40 °C for 2 minutes<br>10 °C/min to 320 °C (30 min) |
| **Injection temp.** | 325 °C |
| **Carrier gas** | Helium 1.2 mL/min |
| **Split ratio** | 50:1 |
| **Detector** | ISQ MS |
| **Injection** | Filter + sample |
| **Total run time** | 60 min |
| **MS Conditions** | |
| **Acquisition** | 35-600 amu |
| **MS transfer line** | 300 °C |
| **Solvent delay** | 2 min |
| **Ion Source temp.** | 300 °C |
| **Electron energy** | -70 eV |

| **Filters Tested (n=3 reps, 2 lots)** | **Millipore® Cat. No.** |
|---|---|
| Glass fiber, 1.0 µm | APFB02500 |
| Quartz fiber | AQFA02500 |

# Microplastics analysis via GC-pyrolysis (ASTM 8401)

## Glass fiber and Quartz fiber demonstrate low background

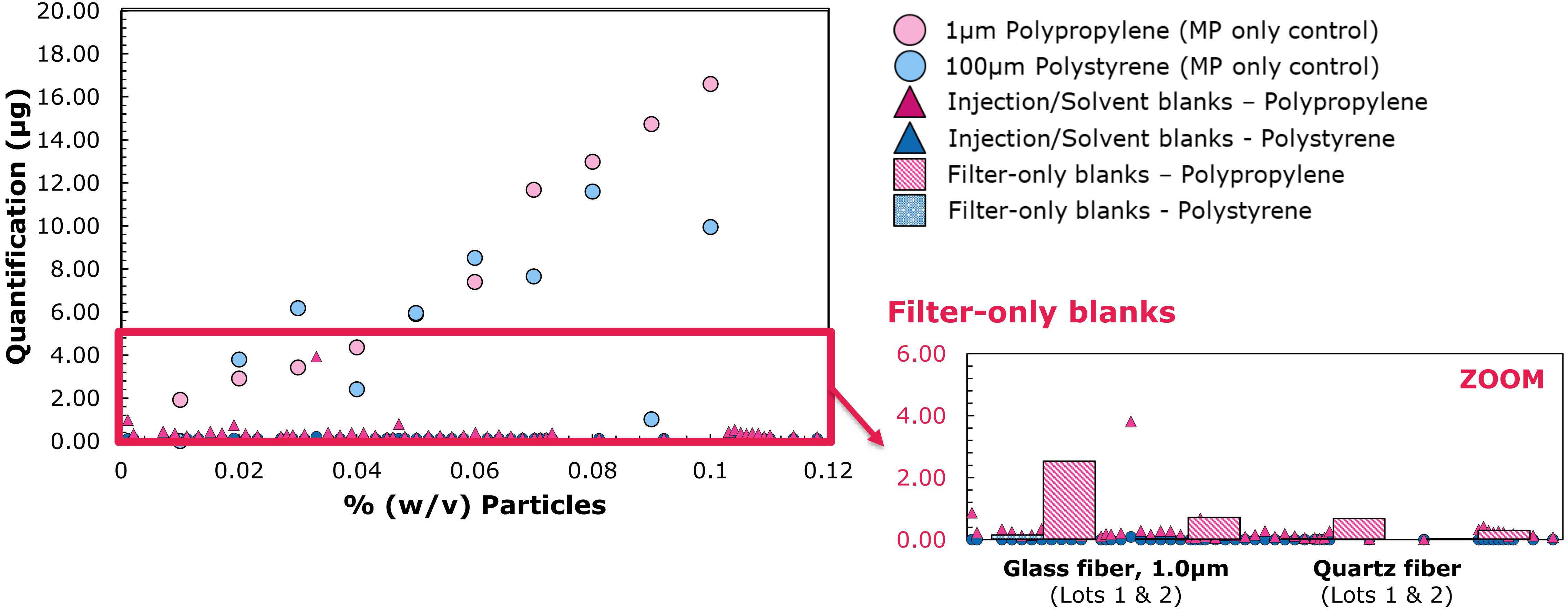


- **Injection and solvent blanks near ND** levels when probed for PP and PS

PennState Institute of Energy and the Environment
Environmental Contaminants Analytical Laboratory
Measurlabs

# Microplastics analysis via GC-pyrolysis (ASTM 8401)

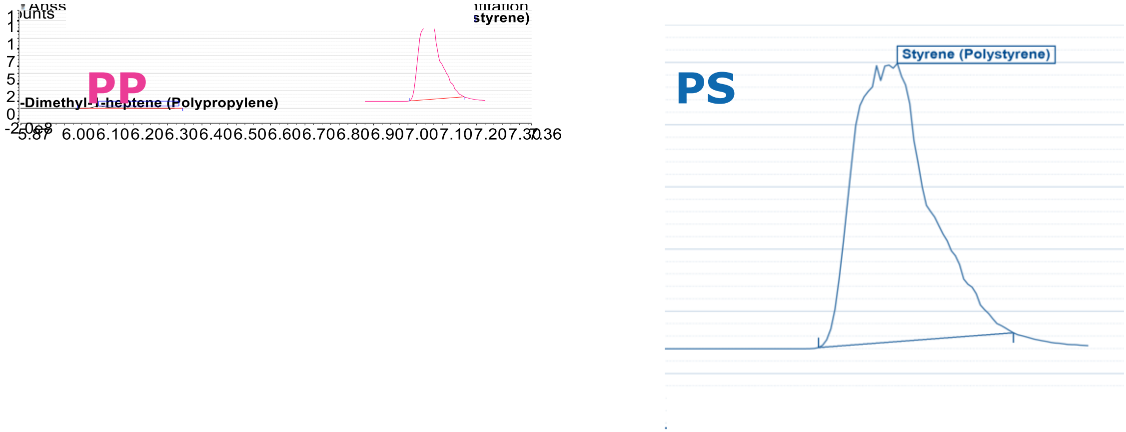
## Glass fiber and Quartz fiber demonstrate low background



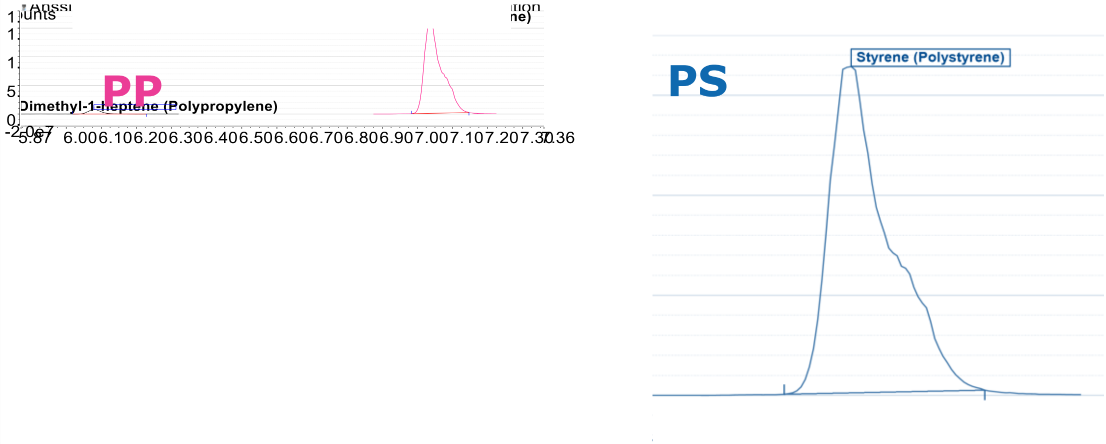
- **Injection and solvent blanks near ND** levels when probed for PP and PS
- Glass fiber & Quartz fiber filters both show **low background levels**

# Microplastics analysis via GC-pyrolysis (ASTM 8401)
## Glass fiber and Quartz fiber allow quantification, but…

### Glass fiber, 1.0 µm



### Quartz fiber



- When collected onto both filter types, polystyrene and polypropylene were **successfully quantified**
- **Data quality issues** observed with peak tailing and low resolution compared to controls
- Indicates suitability of glass fiber and quartz fiber in this method, but requires investigation into **possible matrix effects**
- Supelco® SLB®-5ms Capillary GC column (Cat. No. 28471-U) is suitable

# Spectral and Chromatographic Characterization

# Membrane Substrate Considerations: FTIR/Raman Spectroscopy

### Filter Considerations for Spectroscopy (IR and Raman)

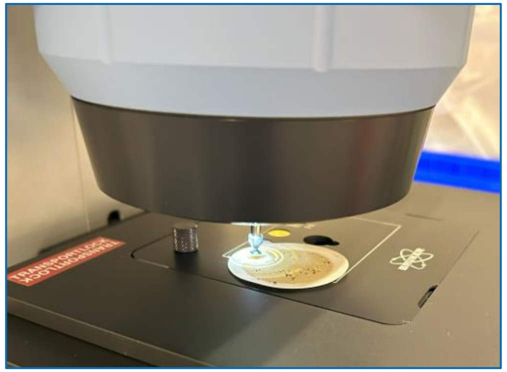
Spectral interference
  - Signal masking
  - Thickness
- Reflectivity
- Signal enhancement
- Laser compatibility
  - Handleability & fragility
- Disc size & instrument compatibility
- Filter dryness
- Particle size as it relates to particle retention
- Filter availability & cost
- If coated, coating reactivity

- IR transparency vs. subtractable background?
- Possible loss of particles
- Curling & deformation can lead to artefacts and difficulties fitting in sample holders/clamps
- IR-transparent filters generally cost more

### Filter Considerations for GC/MS

- Filter diameter
  - Ability to fit in pyrolysis cup
- Subsampling and/or punching out filter sections
- Sturdiness vs. pyrolysis method
- Low or highly distinguished background from polymers

### LUMOS II (Bruker)



**Similar to upright microscopes**

Membranes should be **handleable enough** to place onto the stage

- For ATR, the crystal contacts the membrane = **need a stiff surface**
- For Reflection, there is no contact, but it can be less accurate

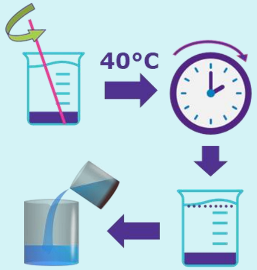
MilliporeSigma, **2024**. *Membrane filters for microplastics analysis*.

Gregory Weiss, Kathryn Garbuzinski (**2025**); Julia McCarthy and Maria Portie (**2024**)

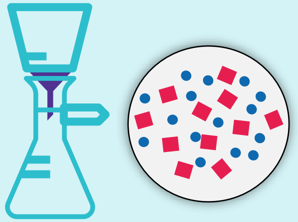
# Microplastics analysis via FTIR Microscopy

## Testing different membrane substrates in ATR and Reflection

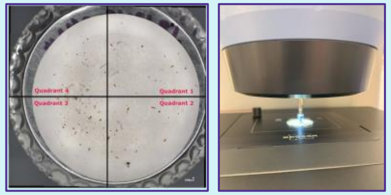
**Collect and sieve** beach sand



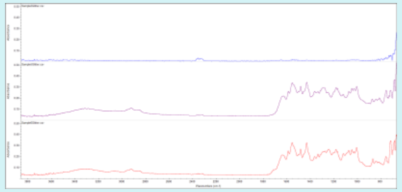
**Density separation**



**Spike** with glitter & **vacuum filter** onto various membranes



Segment into quadrants & **analyze** (ATR or Reflection)



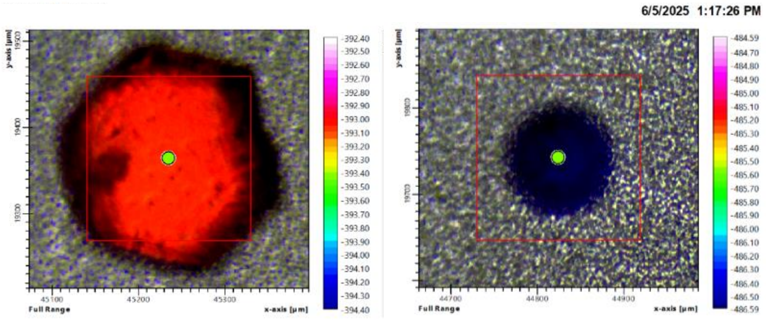
**Export** from OPUS (Bruker) → OMNIC (ThermoFisher)

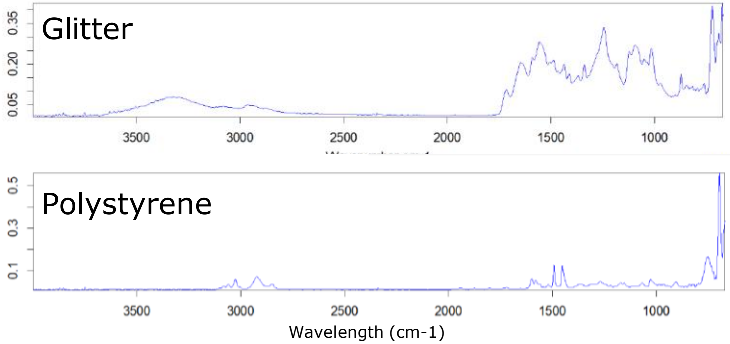
| Membrane Type | Millipore® Cat. No. | Pore (µm) | Backer? | Notes |
|---|---|---|---|---|
| **Silver** | Z623032 | 0.8 | No | • Extremely fragile<br>• Expensive |
| **Aluminum Oxide**<br>Supporting Ring | WHA68096022 | 0.2 | Both | • Extremely fragile<br>• Expensive |
| **Plain PC**, opaque | ATTP04700 | 0.8 | Yes | • Some interferences (not invisible)<br>• Extremely flimsy |
| **Plain PC**, transparent | TMTP04700 | 5.0 | Yes | |
| **Hydrophilic PTFE** | JAWP04700 | 1.0 | Yes | • Handleability at higher temperatures |

Gregory Weiss, Kathryn Garbuzinski (**2025**); Julia McCarthy and Maria Portie (**2024**)

# Microplastics analysis via FTIR Microscopy
## Testing different membrane substrates in ATR and Reflection

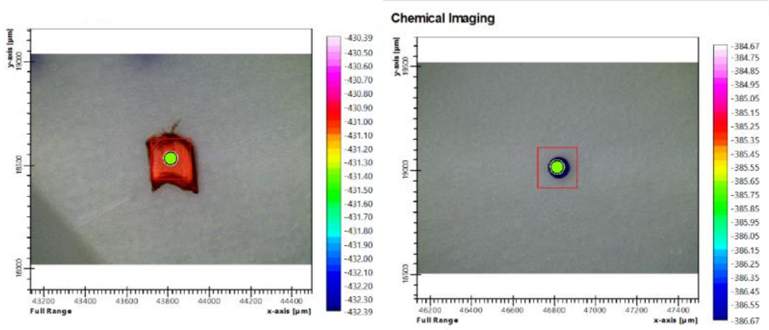
### Silver

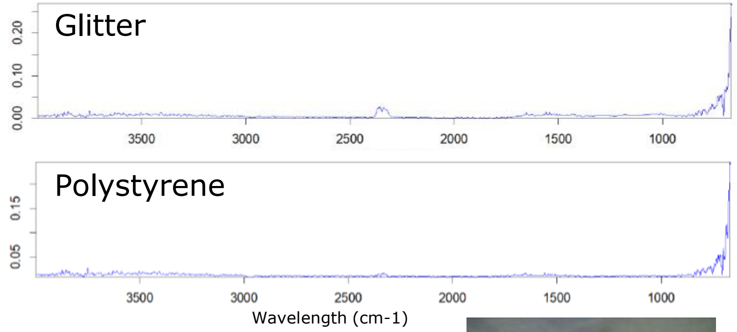




- ✓ Good matches in ATR & Reflection
- ✓ Library match **58%** PET and **93%** PS matches
- ✗ Membrane is wrinkly after wetting and drying; hard to focus

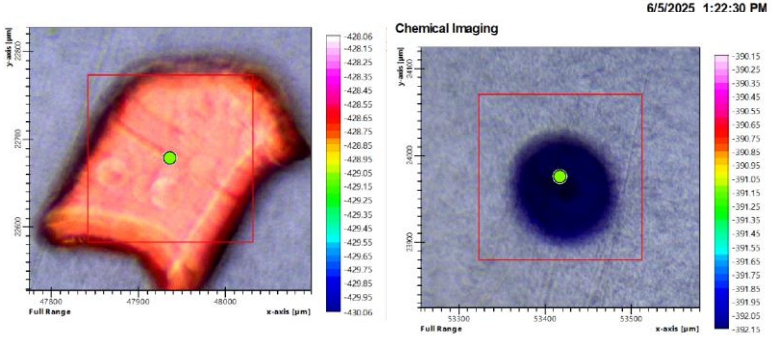
### Aluminum oxide

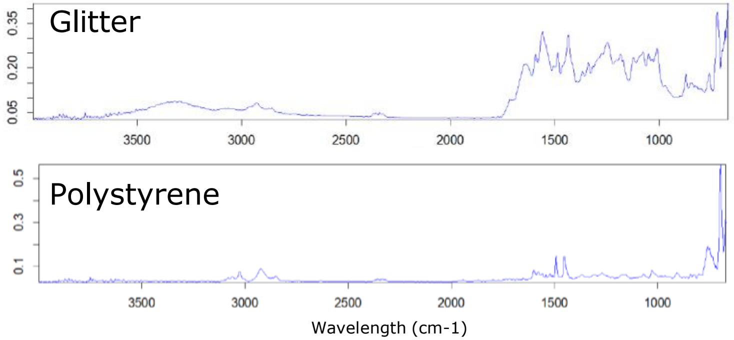




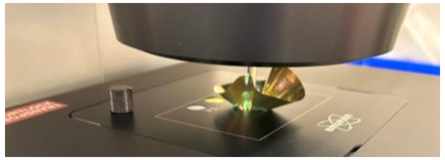
- ✗ No matches observed for glitter or PS beads
- ✗ Smooth surface & extremely breakable membrane causes movement of the particles (even when backed)

### Polycarbonate (opaque & clear)





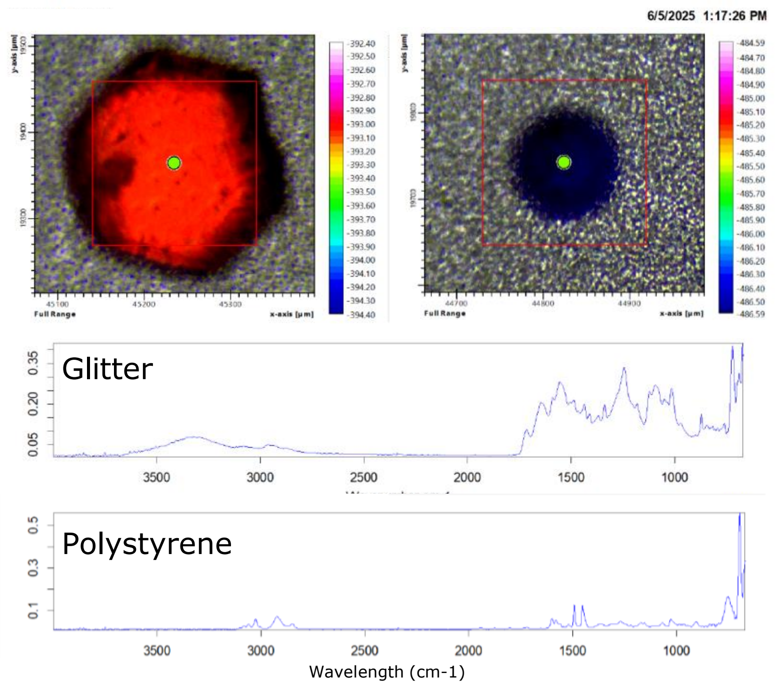
- ✗ Okay in ATR, but always gives a PC background in reflection
- ✗ Smooth surface and flexibility causes movement of particles



Gregory Weiss, Kathryn Garbuzinski (**2025**)

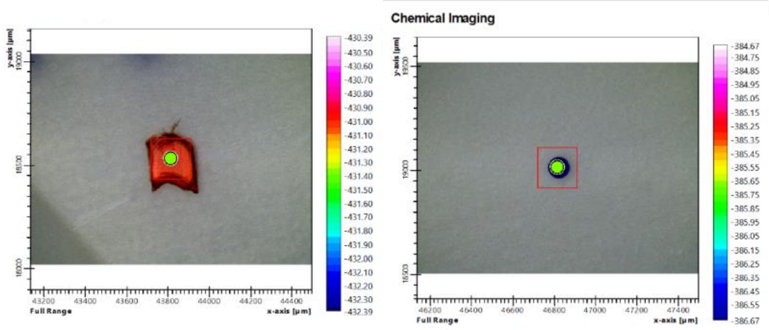
# Microplastics analysis via FTIR Microscopy
## Testing different membrane substrates in ATR and Reflection

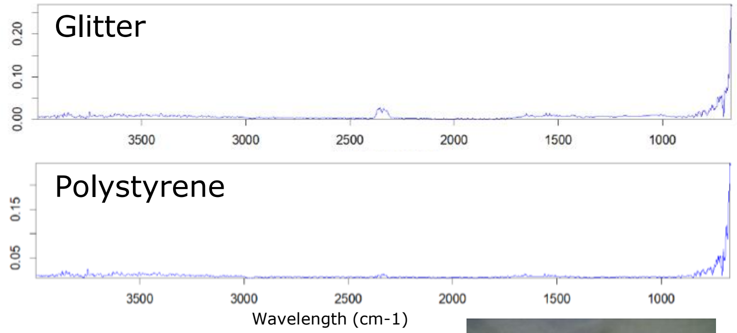
### Silver



- ✓ Good matches in ATR & Reflection
- ✓ Library match **58%** PET and **93%** PS matches
- ✗ Membrane is wrinkly after wetting and drying; hard to focus

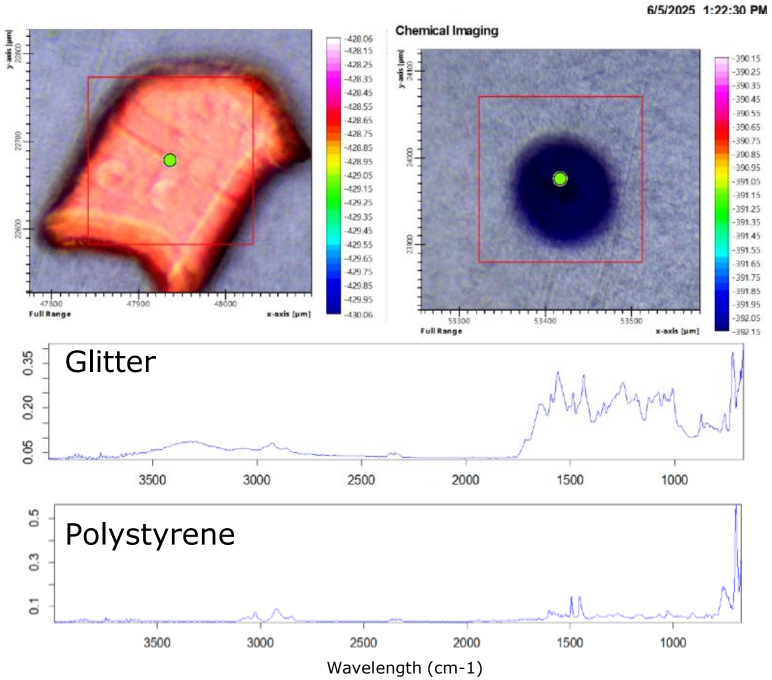
### Aluminum oxide



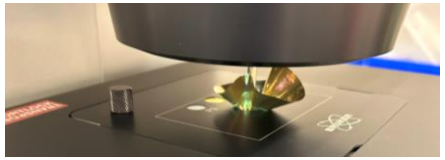


- ✗ No matches observed for glitter or PS beads
- ✗ Smooth surface & extremely breakable membrane causes movement of the particles (even when backed)

### Polycarbonate (opaque & clear)



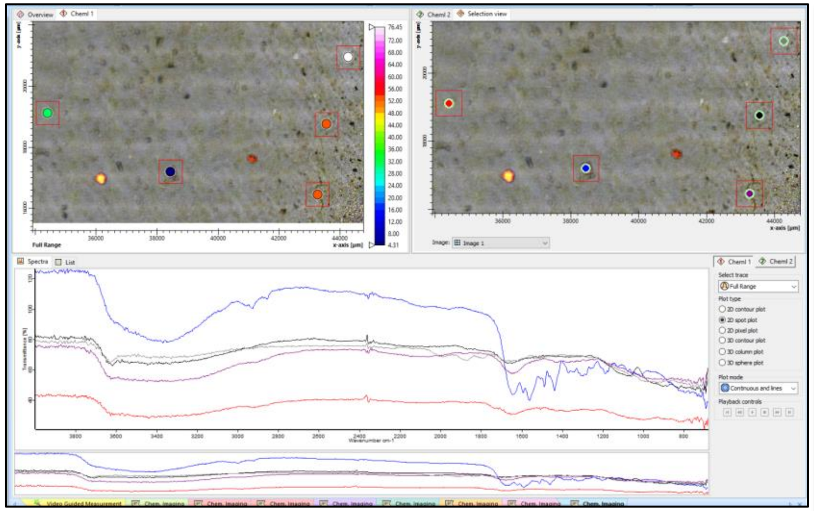
- ✗ Okay in ATR, but always gives a PC background in reflection
- ✗ Smooth surface and flexibility causes movement of particles



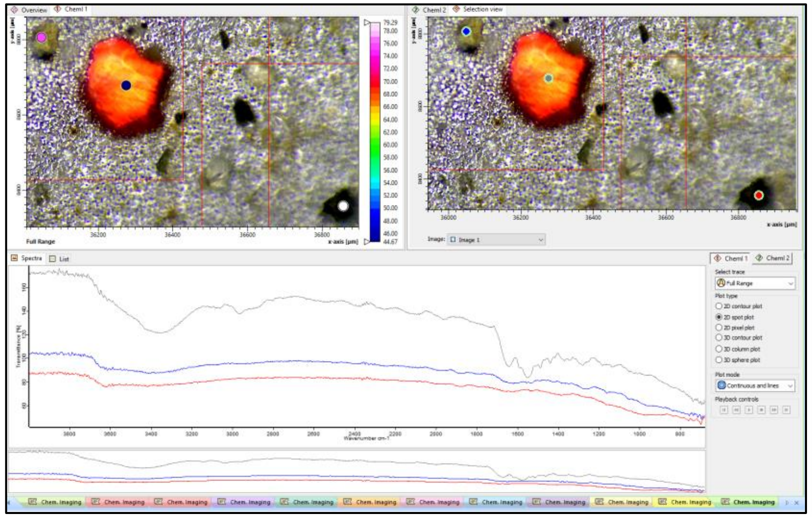
# Microplastics analysis via FTIR Microscopy

## Using silver membranes to measure beach sand samples

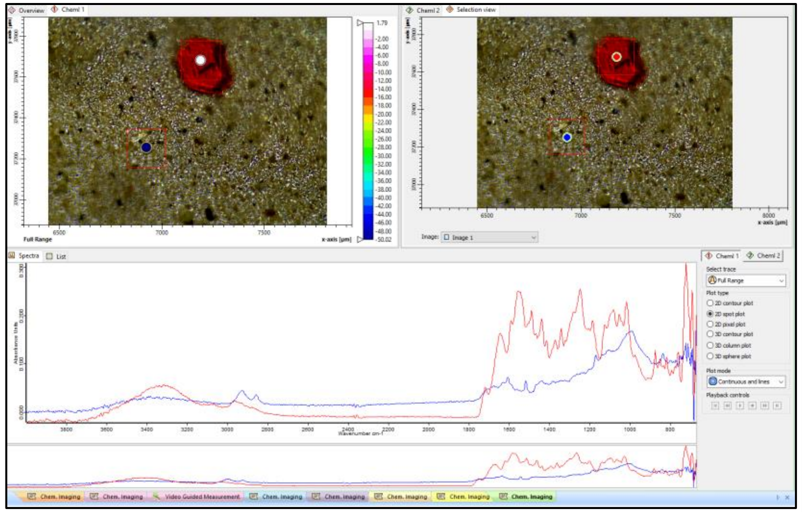
### Crane’s Beach



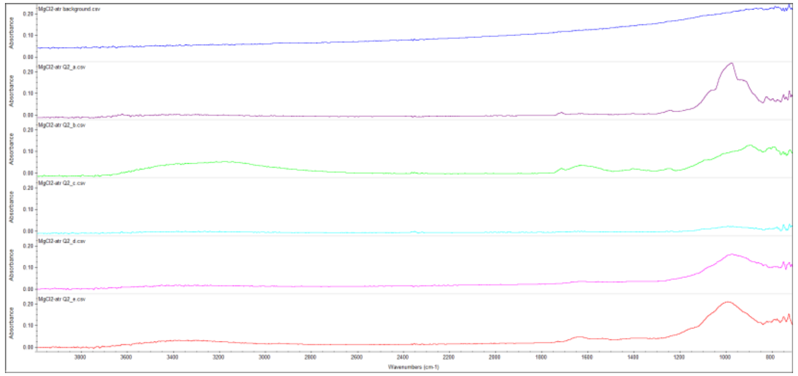
**Matches** to acrylics, polyolefins



### Newfound Lake

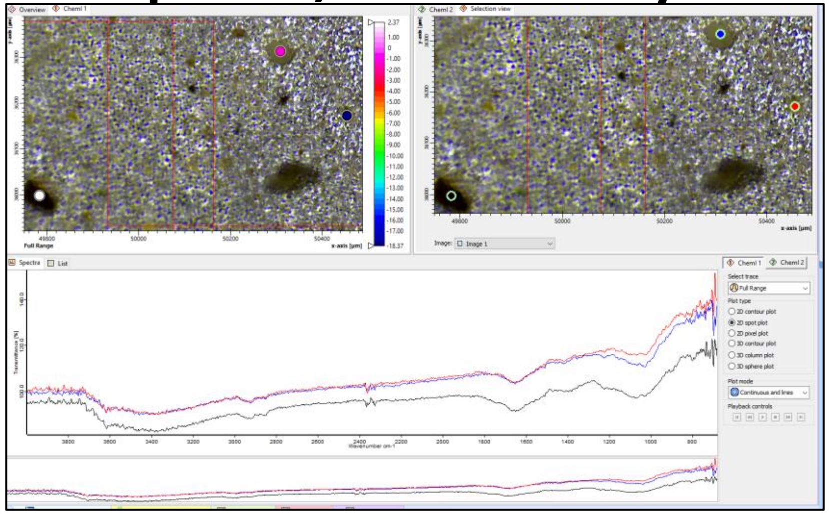


**Matches** to rubber and black rubber, ATR & reflection

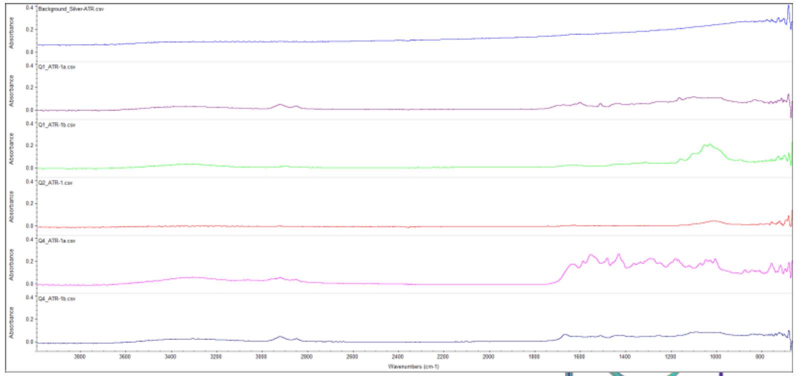


**Example scans in Reflection mode**

### Mattapoisett/Buzzard’s Bay



**Various matches** to synthetic resin, polymer additives, PAMAM, pigments, surfactants

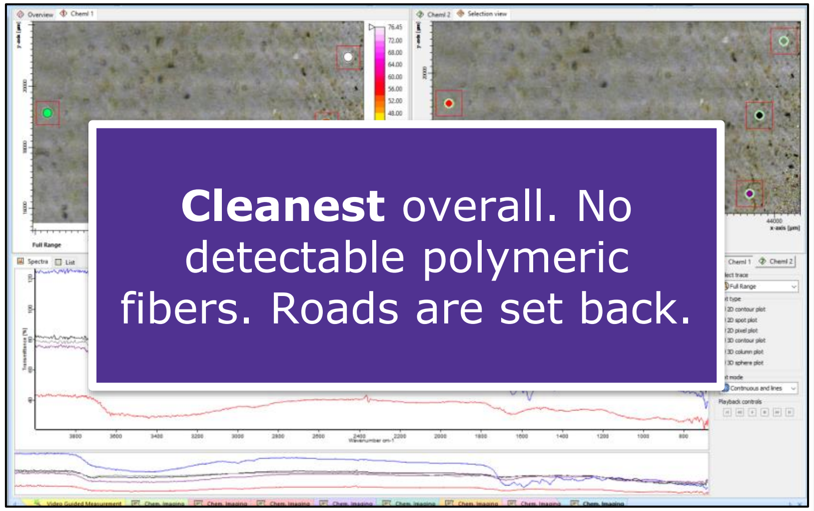


**Example scans in ATR mode**

# Microplastics analysis via FTIR Microscopy

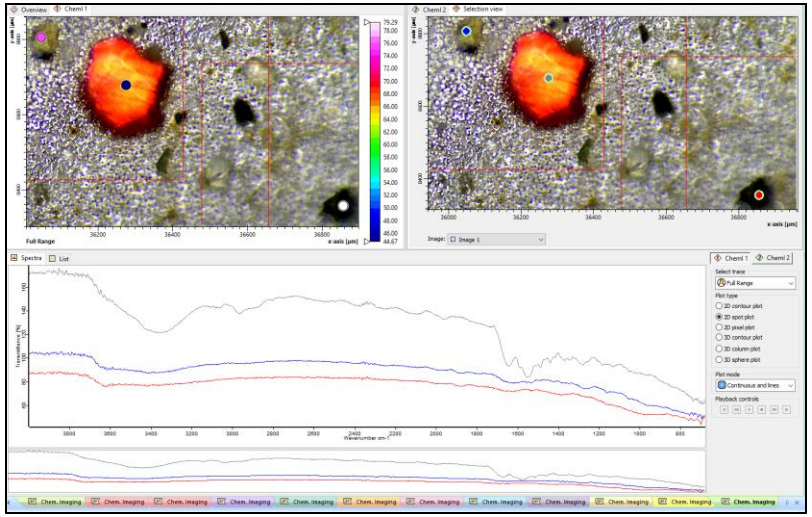
## Using silver membranes to measure beach sand samples

### Crane's Beach

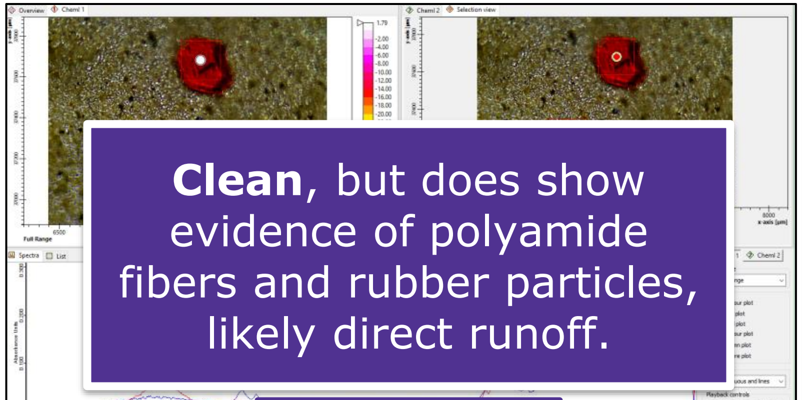


**Cleanest** overall. No detectable polymeric fibers. Roads are set back.

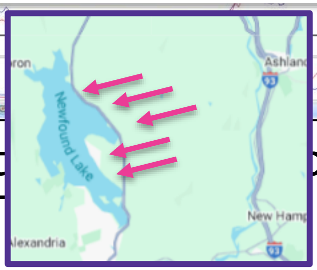
**Matches** to acrylics, polyolefins



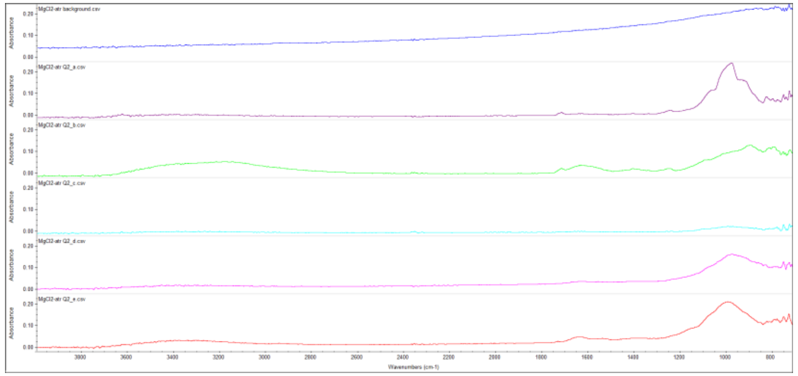
### Newfound Lake



**Clean**, but does show evidence of polyamide fibers and rubber particles, likely direct runoff.

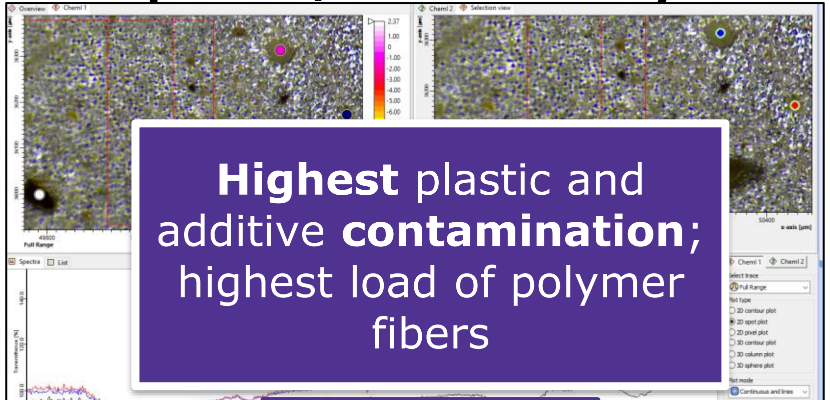


**Matches** t black
rubber, AT



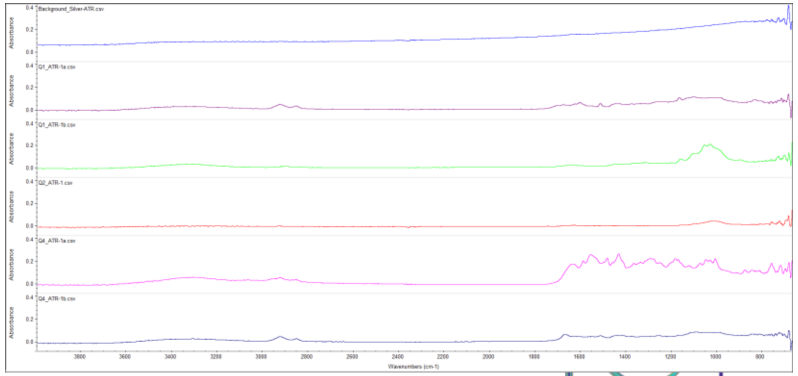
**Example scans in Reflection mode**

### Mattapoisett/Buzzard's Bay



**Highest** plastic and additive **contamination**; highest load of polymer fibers

**Various m** etic
resin, polyr AMAM,
pigments, s



**Example scans in ATR mode**

Gregory Weiss, Kathryn Garbuzinski (**2025**)

MilliporeSigma, **2024**. *Membrane filters for microplastics analysis*.

# Microplastics characterization

## Guidelines for choosing the right membrane

🟢 Recommended
🟤 Recommended, with caveats
🔴 Not recommended

| **Technique/Application** | **Recommended Millipore® Membrane Filter(s)** | | | | | | |
|---|---|---|---|---|---|---|---|
| | Glass fiber (GFF) | Quartz fiber (QFF) | Polycarbonate (PC) | Mixed cellulose ester (MCE) | Polypropylene (PP) | Aluminum oxide ($Al_2O_3$) | Silver |
| Production of MAG water | 🟢 | 🟢 | 🟢 | 🟢 | N.T. | N.T. | N.T |
| Visual analysis | 🟢 | 🟢 | 🟢 | 🟢 | 🟢 | N.T. | 🟢 |
| Nile Red Fluorescence | 🟢 | 🟢 | 🟢* | 🟢* | 🔴 | 🔴 | 🟢 |
| Drying & Handling | 🟢 | 🟢 | 🟤 | 🟢 | 🟢 | 🔴 | 🟤 |
| Improvement on handling | 🟢 | 🟢 | 🟢 | 🟢 | N.T | 🔴 | N.T |
| Oil flotation | 🟢 | 🟢 | 🔴 | 🔴 | 🟢 | N.T. | N.T |
| Salt Separation | 🟢 | 🟢 | 🟢 | 🟢 | N.T. | 🔴 | N.T. |
| Chem. digestion/30% $H_2O_2$ | 🟢 | 🟢 | 🟤 | 🟢 | N.T. | 🟤 | N.T |
| Chem. digestion/Fenton Rxn | 🟢 | 🟢 | 🟤 | 🔴 | N.T. | 🟤 | N.T |
| Chem. digestion/KOH | 🟢 | 🟢 | 🟤 | 🔴 | N.T. | 🟤 | N.T |
| Chem. Digestion + Salt (NaI) | 🟢 | 🟢 | 🟢 | 🟤 | 🔴 | 🟤 | 🟢 |
| Spectroscopy | 🔴 | 🔴 | 🟤 | 🔴 | 🔴 | 🟤 | 🟢 |
| Pyrolysis-GC/MS | 🟢 | 🟢 | 🔴 | 🔴 | 🔴 | 🔴 | N.T |

- Microplastics methods are being developed, with focus on certain matrices and portions of workflow (sampling, etc.)
- **Many technical hurdles** in collecting and analyzing microplastics, **cut discs are almost always required**
- Through this study our team **expanded the "recommended membranes by method"**

# Thank you

Curious2024 Future Insight™--Microplastics Hackathon, Mainz Germany (10-11 JUL 2024)

**Maricar** Dube
**Amy** Laws
**Ryan** Amara
**Mayra** Jimenez
**Kevin** Sydlowski
**Vivek** Joshi
**Taylor** Reynolds

**Dave** Brewster
**Janet** Smith
**Yue** Li
**Julia** McCarthy
**Maria** Portie
**Gregory** Weiss
**Kathryn** Garbuzinski

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Analytical Intern – Innovation, Strategy and Portfolio Management