Comparative Evaluation of Filtration and Imaging Properties of Filter Membranes for Microplastic Capture and Analysis

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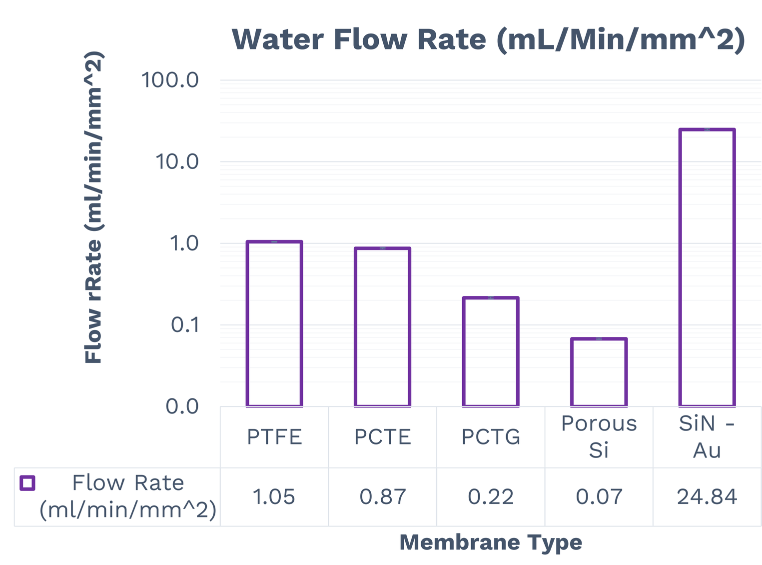
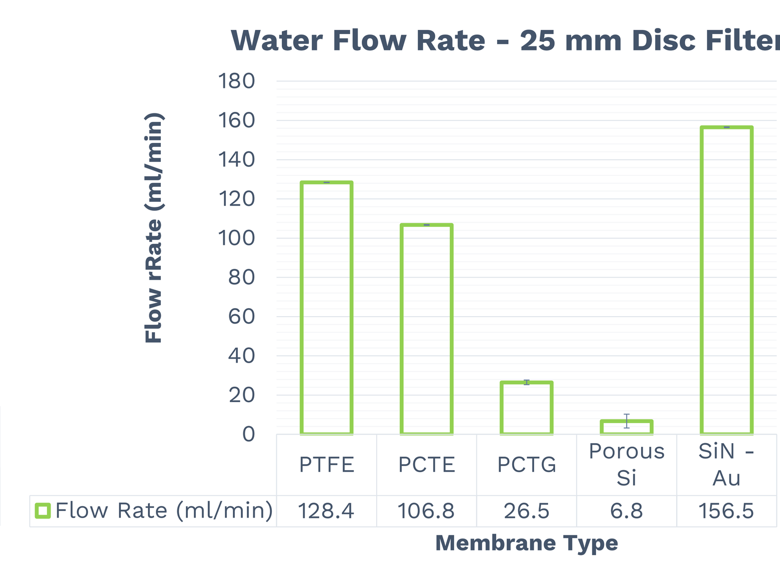
Introduction

Pollution by microplastics (MPs) in drinking, fresh, and ocean waters, as well as food and beverages, is a growing problem that is now well-recognized in both the popular media and scientific literature. Concerning levels of MPs have been found in food and local waters, as well as in human tissues. Several government regulatory bodies have, or are in the process of, implementing mandatory MP drinking water testing. Consequently, there are needs for greater understanding of the performance characteristics of common MP analytical methods and for standardizing methods and reporting.

Here, we report on the comparative evaluation of the filtration and imaging properties of five filter membranes capable of MP capture and analysis. This study was undertaken as part of an inter laboratory methods evaluation study coordinated by the Southern California Coastal Water Research Project. We compared track-etched polycarbonate +/- gold coating (PCTE and PCTG), polytetrafluoroethylene (PTFE) porous silicon (PS) and gold-coated microslit silicon nitride membranes (MSSN-Au). We further demonstrate use of MSSN filters to monitor MP entrainment along a municipal drinking water delivery network from the producing plant to a point of use drinking fountain at a local University.

Clean Water Flux Analysis



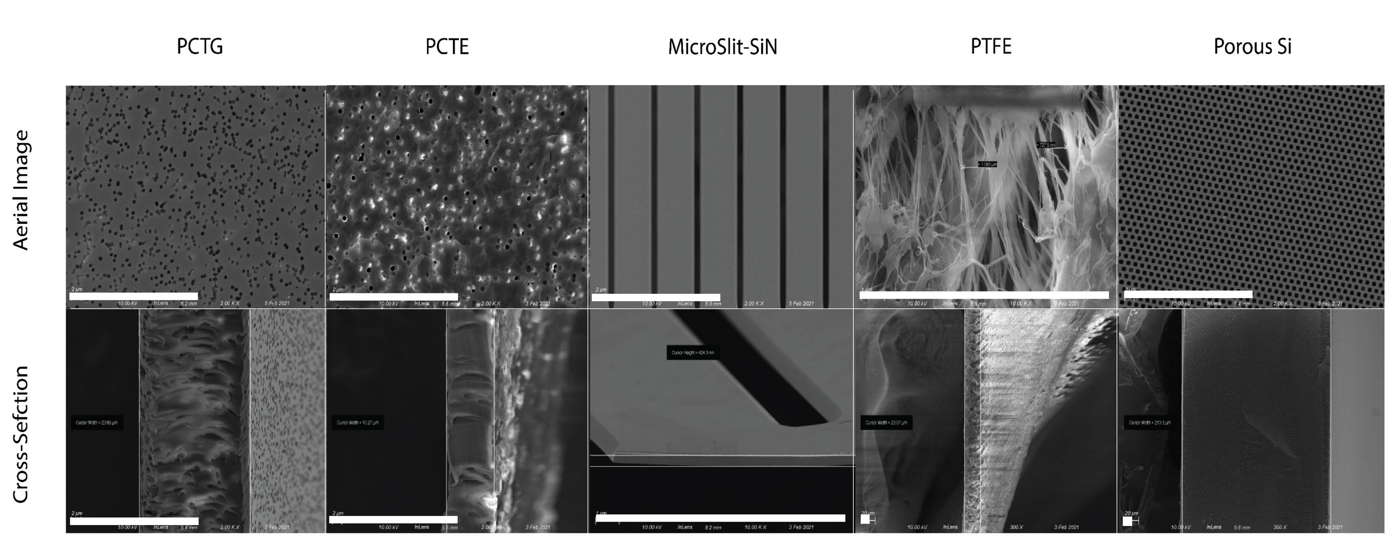


- Polymeric Membranes (PTFE, PCTE) offer similar flow rates for the same surface area
- Addition of gold to PCTE (i.e., PCTEG) greatly reduces the water flow rate (~5x)
- PS filters are impractically slow (> 2 hrs. for 1 L of clean water)
- MSSN-Au filters (25 mm disc format) demonstrated the fastest filtration times (>17% faster than PTFE) using substantially less filter area (6.3 vs 70.9 mm²)

1 Liter Water Flux Time (minutes)

PTFE	PCTE	PCTG	Porous Si	SiN - Au
7.8	9.4	37.8	148.0	6.4

Filter Characteristics

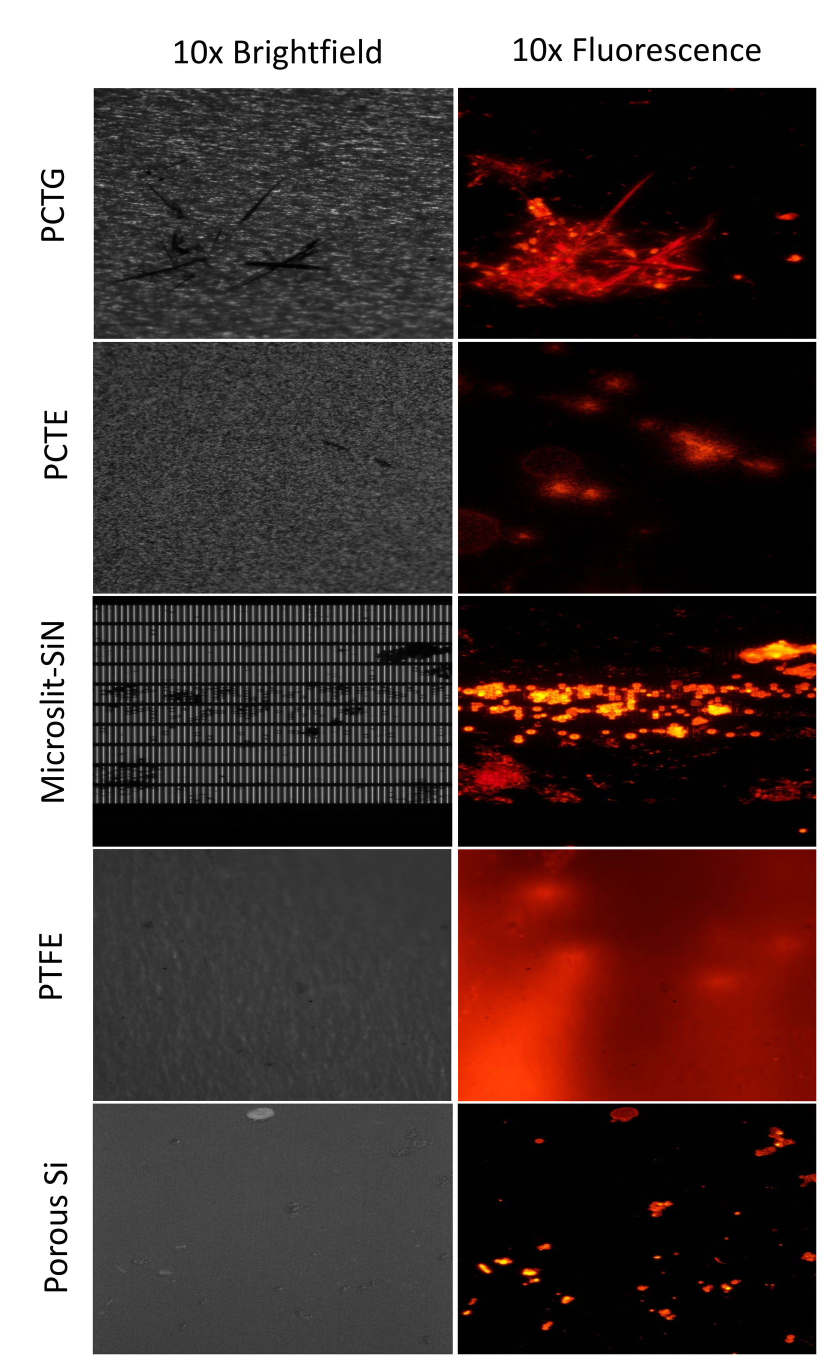


Note: White scale bars denote 10.0 micron

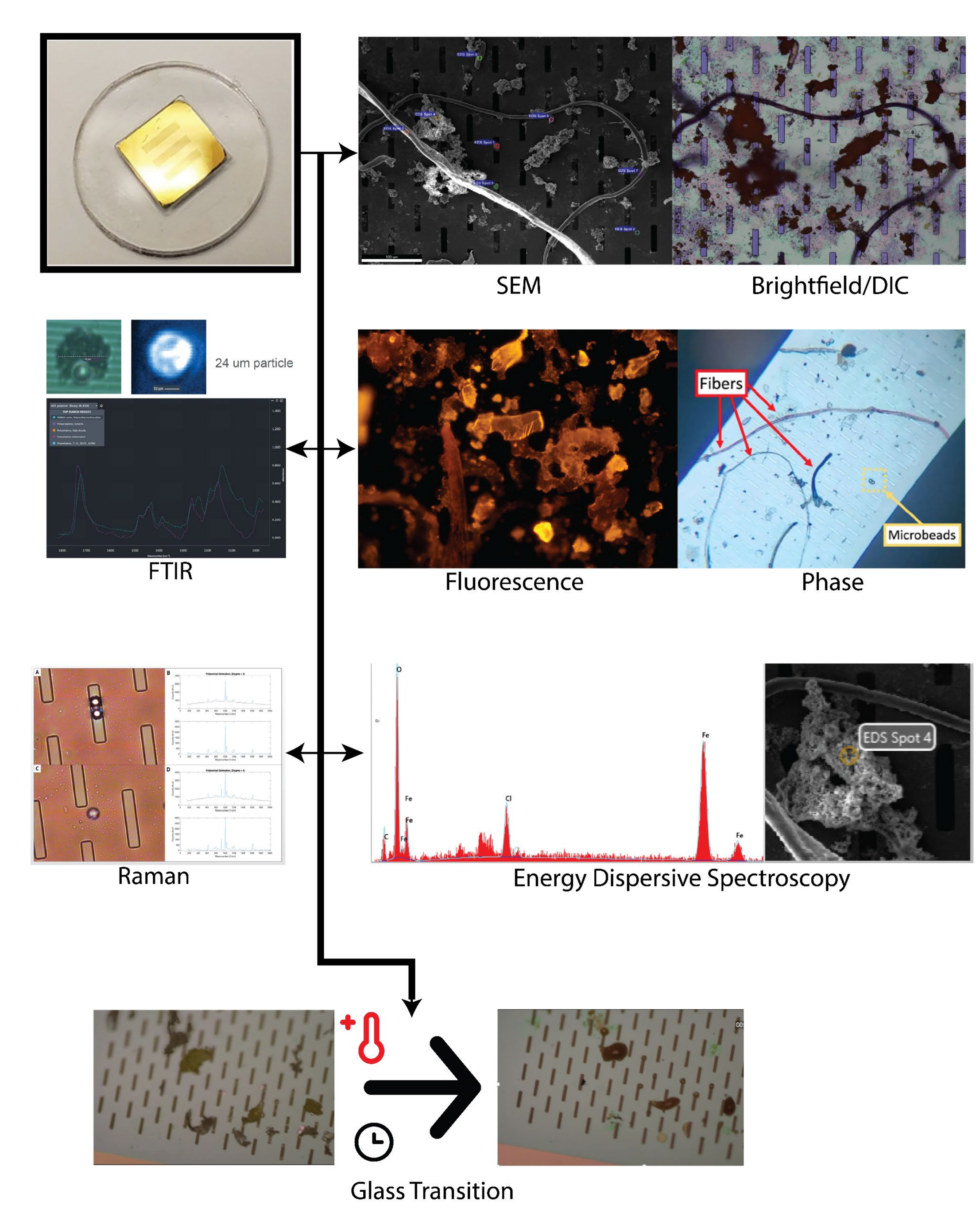
Filter Type	Vendor	CAT	Lot	Composition	Cutoff (µm)	Porosity (%)	Thick. (µm)	Price (USD)
PTFE	Advantec	H100A025A	90227640	Polytetrafluoroethylene - hydrophilic treated	1.0	-	35	$2.67
PCTE	Sterlitech	PCT1025100	M-180227	Polycarbonate - Track Etched	1.0	16%	11	$0.91
PCTG	Sterlitech	1270007	TPA.PC.20.01.1.8	Polycarbonate - Track Etched w/ 40 nm Au	0.8	15%	9	$23.84
Porous SI	Smart Membranes / Thermo Fisher	950789-W16	-	Macroporous Silicon Lift-Off Membrane	1.0	60%	220	$30.00
MSSN-AU	SiMPore	FD25-1.0-NC	4882	Silicon Nitride, 120nm Au Coated	1.0	16%	0.4	$16.00

- PTFE Background fluorescence* prevented use of higher gain to improve contrast
- PS filter data not available due to impractically slow sample processing and incompatibility with phase microscopy
- Metal coating of polymeric filters tends to improve microscopy results with considerable increase in sample processing durations and filter cost
- Solid state materials (e.g., PS, Al oxide, etc.) currently in use for IR and Raman improve fluorescent microscopy results but with substantial decrease in sample processing rate and considerable expense

Representative Microscopy



Analytical Optionality of SiN Membranes

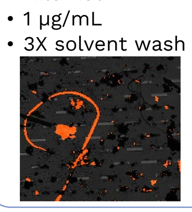


Capture & Analysis Workflow

Sample Filtration
(10 Minutes)
- Vacuum or peristaltic pumping-based filtration apparatus
- Low sample volume (0.1- 1 L)

Dissolution*
(90 Minutes)
- Digest Biologicals & Other Contaminants if required
- SDS/Tris/BME
- Hot Water Rinse
- Membranes are alternatively suitable for peroxide or KOH digests

Staining
(10 Minutes)
- Improves MP contrast relative to other particles when using fluorescence microscopy
- Nile Red
- 1 µg/mL
- 3X solvent wash

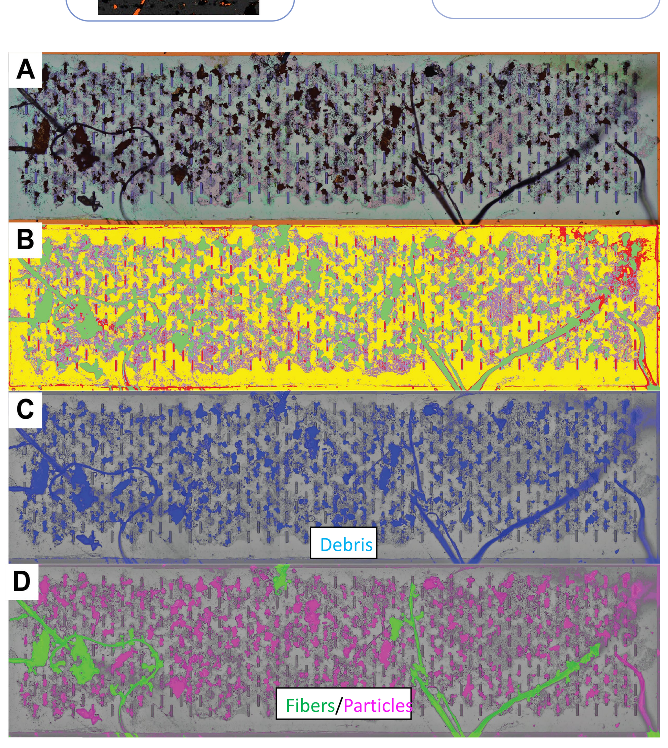


Imaging & Analysis
(10 Minutes)
- Fluorescent Microscopy** & Image Analysis
- Fluorescent particles tabulated by mass and quantity
- Filter Images [A] are processed via edge segmentation processes [B-C] to differentiate particles from fibers [D]

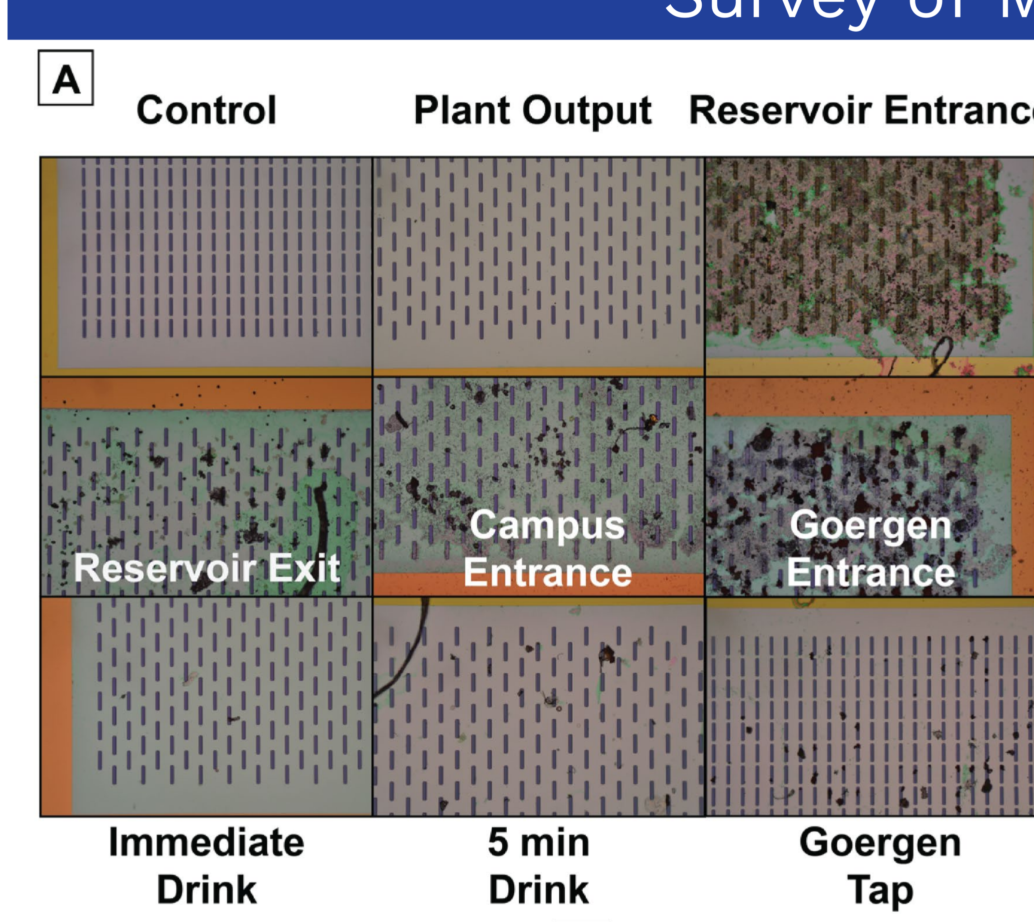
* Not required for tap water samples, ref: Madejski et al, 2020. Sustainability, 12: 10655.
** Fluorescent Microscope Required

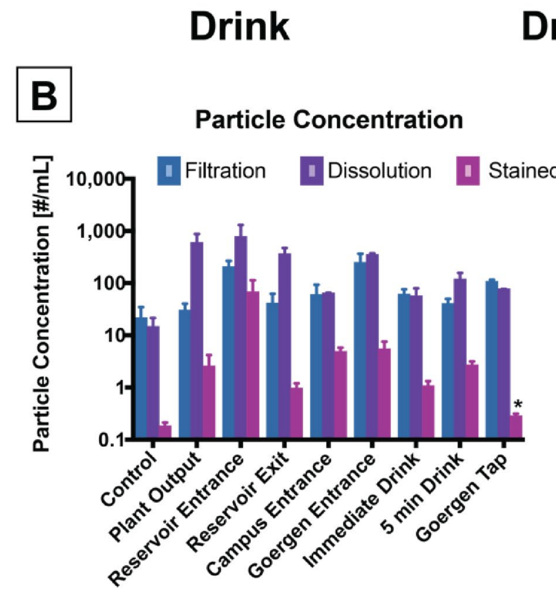
Capture and Analyze Workflow Key Performance Attributes

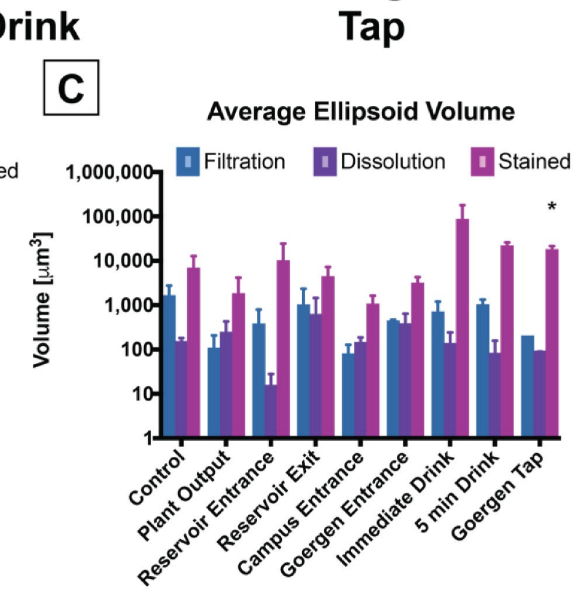
Time to Result (TAT)	90 minutes
Sensitivity	1 particle / mL
Resolution	1 – 100 microns
Sample Dissolution	Clean Water : No Env. & Wastewater Samples: Yes
Per Test Cost	$16 (filter)
Required Instrumentation	Vacuum Filtration Apparatus, Fluorescent Microscope w/Camera
Availability	Beta units and SOP available for evaluation



Survey of Municipal Water [1]







Particulate quantification along the water transport route. (A) Representative images of the captured particulate are shown (10× objective magnification, 8 µm wide MSSN filters). (B) Particle Concentration normalized to the volume of water filtered. (C) Average volume of a particle calculated from minor and major axis of image projection. N = 3 replicates, 9–36 images/replicate for dissolution and filtration stages, 1–2 whole field images for stained stage (N = 2 for asterisk [*]). Error bars are the standard error of the mean.

- We used MSSN filters to survey the MP concentration along a municipal water route (Hemlock Lake, NY).
- Based on consuming 500 mL of water a day, a consumer at a final delivery site (a drinking fountain on the University of Rochester campus) would consume 496 MP particles/day and 3–7 µg of MP upon immediate use.
- If unfiltered, the pipes directly below the drinking fountain indicate 5.0 particles/mL (2522 particles/day) and 0.5–1.2 µg of MP load.
- Simple filtration appears to be effective in reducing the debris load, as there is a ~50% reduction in the amount of particulate at the drinking fountain immediately compared to the building's source. However, more of these particulates appear to be plastic.
- Many of the observed particles in pipes before the drinking fountain are rust and sand from the environment in which the water resides.

Conclusions

- Polymeric filters generally offered good flow rates for clean water samples at reasonable cost, but at the expense of poor overall microscopy performance, owing to their heterogeneous internal structure (visible by SEM) and their intrinsic chemical composition.
- Non-polymeric MSSN filters lacking appreciable internal structure offer higher filtration rates than polymeric filters and demonstrate microscopy performance like comparable solid-state filters.
- The utility of MSSN filters for MP surveillance across a water distribution has been demonstrated using a simple capture and on-filter analysis workflow
- Future plans include expanding the survey to other available filter types and broader samples, as well as perform on-filter sample digestion to align more closely with current and future optimized analytical workflows for microplastics analysis.

Acknowledgements

[1] - Gregory R. Madejski,, S. Danial Ahmad, Jonathan Musgrave, Jonathan Flax, Joseph G. Madejski, David A. Rowley, Lisa A. DeLouise, Andrew J. Berger, Wayne H. Knox, and James L. McGrath, "Silicon Nanomembrane Filtration and Imaging for the Evaluation of Microplastic Entrainment Along a Municipal Water Delivery Route", Sustainability 2020, 12(24), 10655; https://doi.org/10.3390/su122410655

Conflicts of Interest: Gregory Madejski is a cofounder of Parverio Inc. James Roussie is a co-founder of SiMPore Inc.

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