Development of a Novel 20 µm Cut-off Microporous Silicon Nitride Membrane for Separating and Analyzing Microplastic Particles in Potable Water

Teagan Horan[1], Joshua Miller[1] Jared Carter[1], Gregory Madejski[2], Michael Deible[3] and James Roussie[1]*

1. SiMPore Inc., West Henrietta, NY 14586
2. University of Rochester, NY 14620
3. RJ Lee Group, Monroeville, PA 15146

* Corresponding Author: jroussie@simpore.com

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Abstract

Track-etched polycarbonate (PCTE) membranes have been used as a filtration standard for microplastic (MP) capture and analysis across a variety of studies.

However, the track-etch method creates pores of varying angles that complicate on-membrane particle analysis and sometimes create merged multi-pores that allow passage of MPs > 20 µm. Additionally, the membrane's overall ~3 µm thickness makes it prone to folding and wrinkling, which may complicate microscopy analysis.

To address these issues, a novel 20 µm gold-coated microporous silicon nitride (MPSN-Au) membrane was developed and compared directly against gold-coated PCTE (PCTE-Au) membranes via manual manipulation and processing time, pore characteristics, light microscopy particle analysis, and Raman/FTIR analysis.

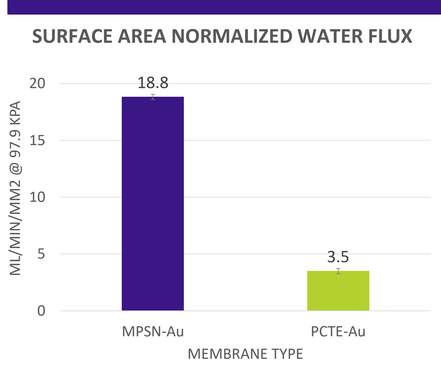
We found that on an area-normalized basis, MPSN-Au membranes offered greater gas and water flux over PCTE-Au. The regular pore geometry of MPSN-Au membranes made particle imaging and spectral analysis more consistent and easier to discriminate between captured PS particles, when compared to PCTE-Au membranes.

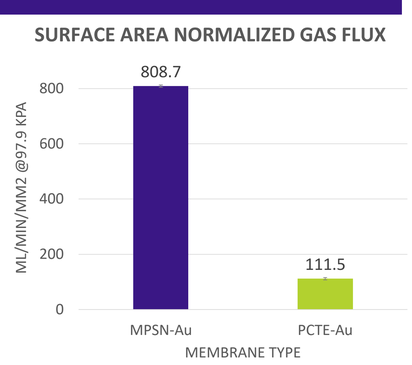
Total handling and processing time for each membrane was compared, which determined that the total processing time (including filtration, automated image acquisition, and particle counting) was 161.56% faster on average for MPSN-Au than PCTE-Au membranes.

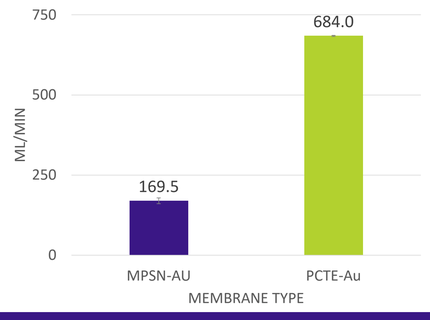
In an 8-hour workday, 85.13% more MPSN-Au membranes can be handled, processed, and imaged than PCTE-Au, which equates to 66 vs 26 total samples, respectively.

Overall, these data demonstrate the utility of MPSN-Au membranes and suggest they can significantly improve testing time-related efficiency in all aspects of normal use-case situations as compared to PCTE-Au membranes.

Water and Gas Flux





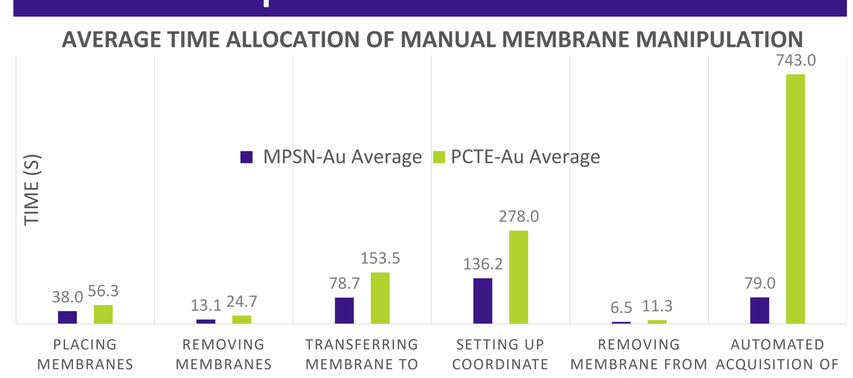


MPSN-Au membranes had a 137.34% faster water flux and a 151.54% faster gas flux when surface area was normalized.

PCTE-Au membranes experimentally filter water faster due to the difference in filtration surface area.

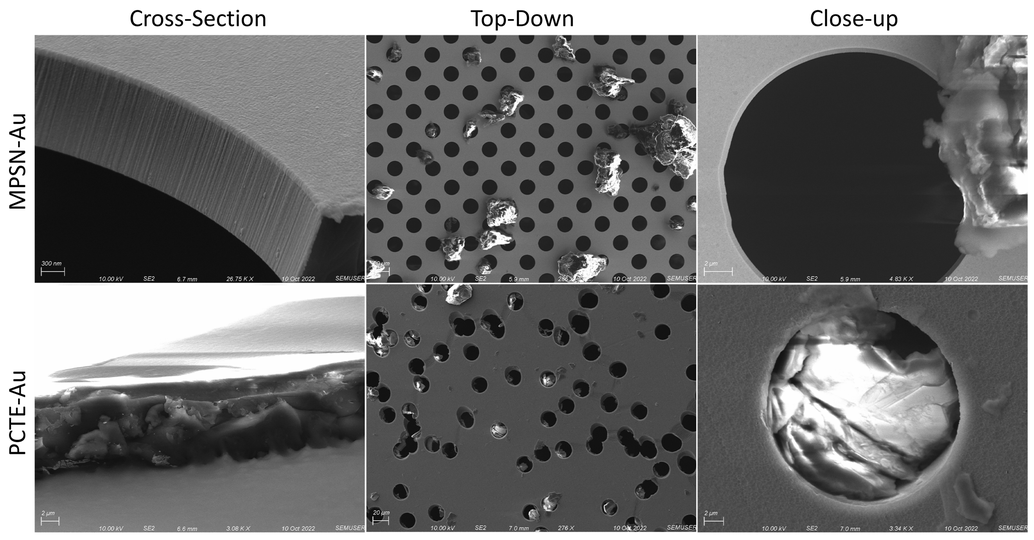
- **PCTE-Au - 78.54 mm²**
- **MPSN-Au – 9 mm²**

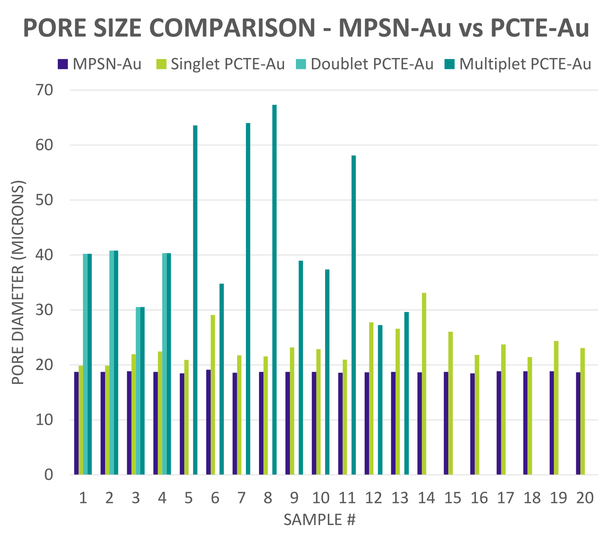
Manipulation Processes



SCAN ME -->
to see a visual demonstration of manipulation processes!

Membrane Characteristics



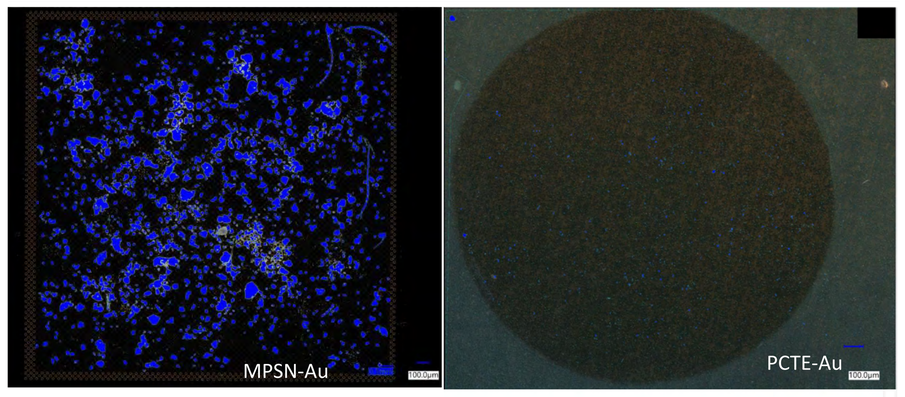


Filter type	Composition	Pore Diameter	Porosity	Thickness (µm)	Gold coating thickness (nm)	Vendor	CAT
MPSN-Au	Gold-coated silicon nitride	18.71 µm	27.7%	0.40	120	SiMPore	5065
PCTE-Au	Gold-coated polycarbonate track-etched	23.60 µm	12%	3	20	SPI Supplies	E20025-MB

Measurements of pore sizes taken from SEM images using ImageJ

- MPSN-Au - pore size was consistent (Min – 18.447 µm, Max – 19.126 µm)
- PCTE-Au - Wide variability of pores required classification into three categories– singlet, doublet, and multiplet. (Min – 19.86 µm, Max – 67.308 µm)

Microscopy Particle Counting



Algorithmic Particle Analysis of Light Microscopy Images

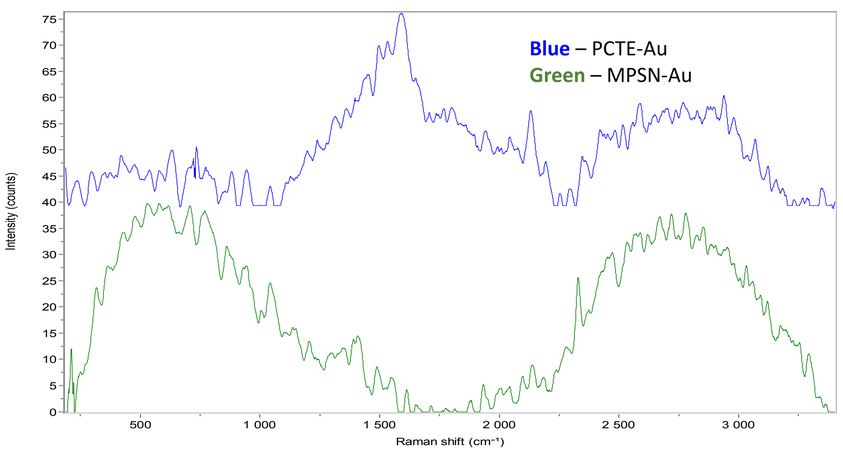
Imaging and particle analysis system - Keyence vhx-7000

- MPSN-Au – appeared to have larger overall particle sizes captured on the membrane (18.7 - 426.5 µm range).
- Resultant particle counts - 1810

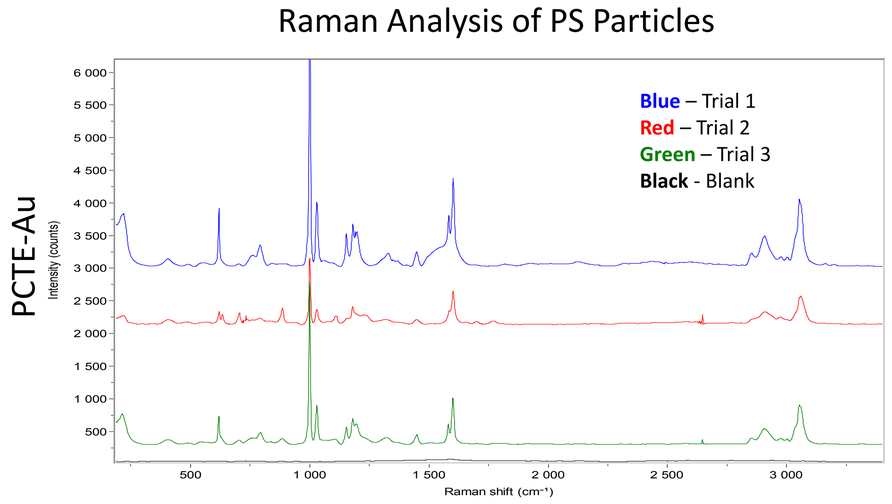
- PCTE-Au – Smaller particle sizes counted, many of which appear to pores mistaken for particles based on diameter sizes returned (2.8 – 166 µm range)
- Resultant particle counts - 9382

Raman and FTIR Analysis

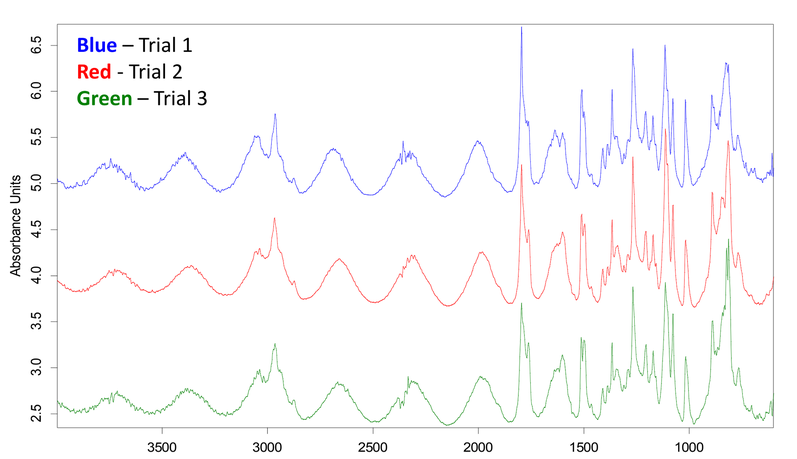
Raman Analysis of Blanks



Raman Analysis of PS Particles



FTIR Analysis of Substrate



Polystyrene sourced from from HPU's Polymer Kit 1.0, (J. Lynch, NIST). Data uploaded to OpenSpecy and coefficients of correlation analyzed:

MPSN-Au – 0.77, 0.73, 0.73
PCTE-Au – 0.75, 0.81, 0.79

Raman

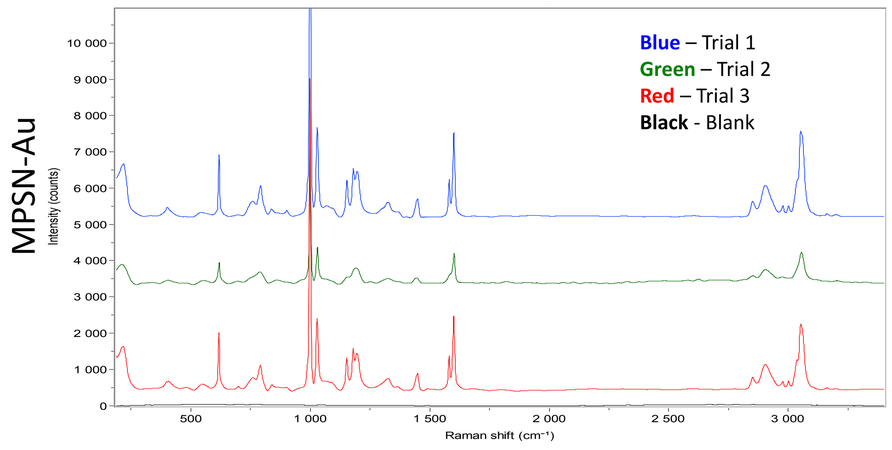
Horbia LabRAM HR with LabSpec v6 was used for all analyses.

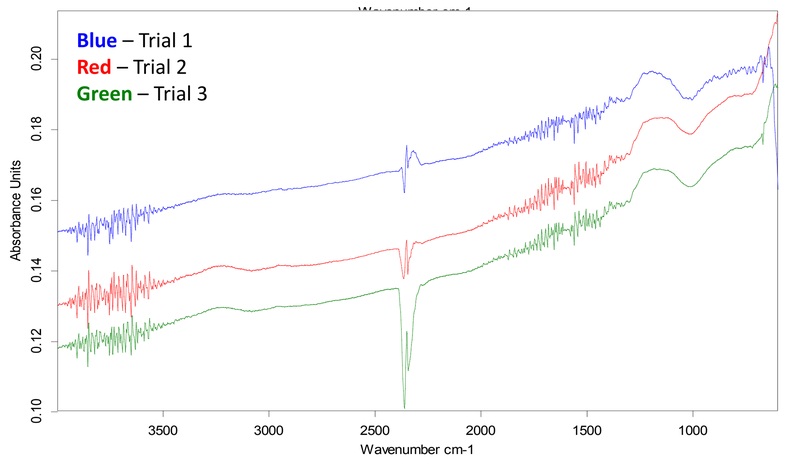
- 632 nm Laser, ~10 mW power. Spot size 1028 nm with 50x long working distance (LWD) objective.
- 600 gr/mm grating gives ~2.7 cm^{-1} resolution.

FTIR

Brucker Tensor 27 with Hyperion microscope attachment used for all analyses.

- Samples analyzed in reflection mode.
- 32 accumulations used for samples.





Conclusions

- MPSN-Au membranes offer a faster and more consistent method of manual manipulation than PCTE-Au.
  - When accounting for experimental filtration times, automated acquisition, and manual manipulation time, 85.13% more MPSN-Au membranes can be processed than PCTE-Au (66 vs 26, respectively, in an 8-hour workday)
- MPSN-Au had 137.34% and 151.54% greater surface area-normalized water and gas flux rates, respectively, versus PCTE-Au, but an experimentally 120.25% slower filtration rate due to the smaller surface area.
- MPSN-Au offers a highly consistent pore geometry, which improves particle counting methods by eye and by algorithm. PCTE-Au's inconsistent pore geometry and angle may lead automated counting systems to count pore edges as particles and misrepresent particle counts by a significant margin. However, the concentration factor of the MPSN-Au membranes may result in multilayer formation with the same filtered volume due to the smaller surface area.
- PCTE-Au has higher coefficient of correlation with returned Raman spectra by 5.24% on average.
- PCTE-Au is not suitable for FTIR analysis at this gold coating thickness – the PCTE substrate can be seen in the resultant spectra, while MPSN-Au returns a consistent and predictable background spectra.

Acknowledgements

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