Membrane Filter Choice for Preparing PFAS Testing Samples

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***Scan** for more PFAS data!*

Introduction

Highly sensitive, LC-MS/MS-based analytical methods for measuring perfluoroalkylated substances (PFAS) in complex matrices, such as wastewater, are becoming more prevalent in today's regulatory landscape. To improve data quality and preserve both instrument and column lifetime, analysts should carefully consider their sample preparation steps.

Filtration with a disc-type membrane filter or a syringe filter (**Fig. 1A-B**) is a simple, cost-effective way to clean up samples and mobile phases[1], and has been increasingly included in PFAS analytical methods for capture of particulates in liquid and air matrices (selected methods in **Table 1**).

Fig. 1: A, Syringe filters; **B,** disc-type membrane filters

With consumables like filters, major concerns about their use are: **1**, contamination of samples and **2**, loss of analytes due to unanticipated binding[2]; however, there are **many considerations** for choosing the right filter for a particular analytical method.

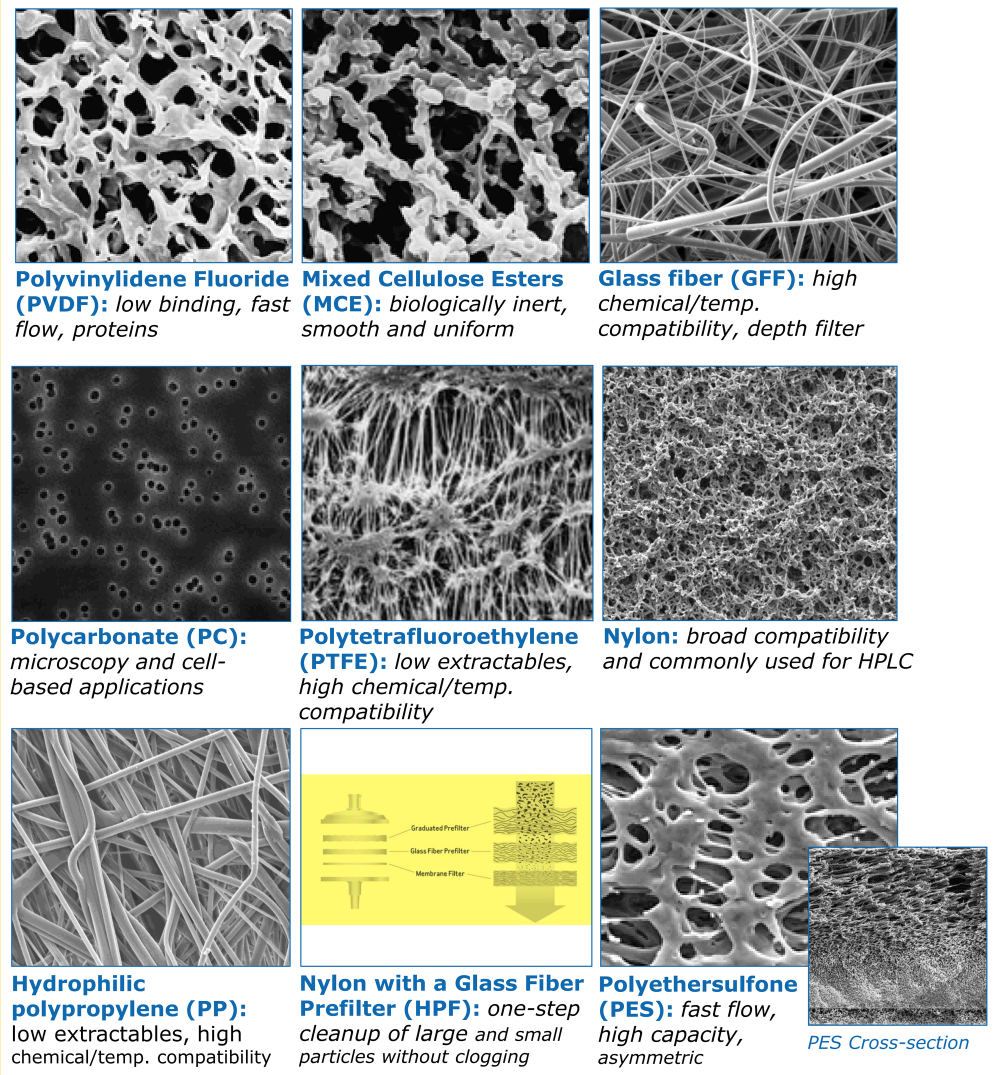
Table 1: Selected PFAS analytical methods that require filters

Method(s)	Matrix/Matrices	Sample Preparation	Analytical Method
ASTM D7968-17a	Environmental solids	Solvent extraction, **filtration**	LC-MS/MS
ASTM D7979-19	Water matrix (no drinking water)	Solvent extraction, **filtration**	LC-MS/MS
FDA C-010.03	Foods	QuEChERS, **filtration**	LC-MS/MS
OTM-45 (OTM-50)	Stationary sources (Air emissions)	Sampling train: **filtration** (particulates); Impingers (gaseous)	LC-MS/MS (GC-MS/MS)
ASTM D8535-23	Soil, Biosolids	Solvent extraction, **filtration**	LC-MS/MS
EPA 1633	Aqueous, soil, biosolids, sediment, tissue	SPE, **filtration**	LC-MS/MS
ASTM 8421-22	Aqueous matrices	Cosolvation, **filtration**	LC-MS/MS

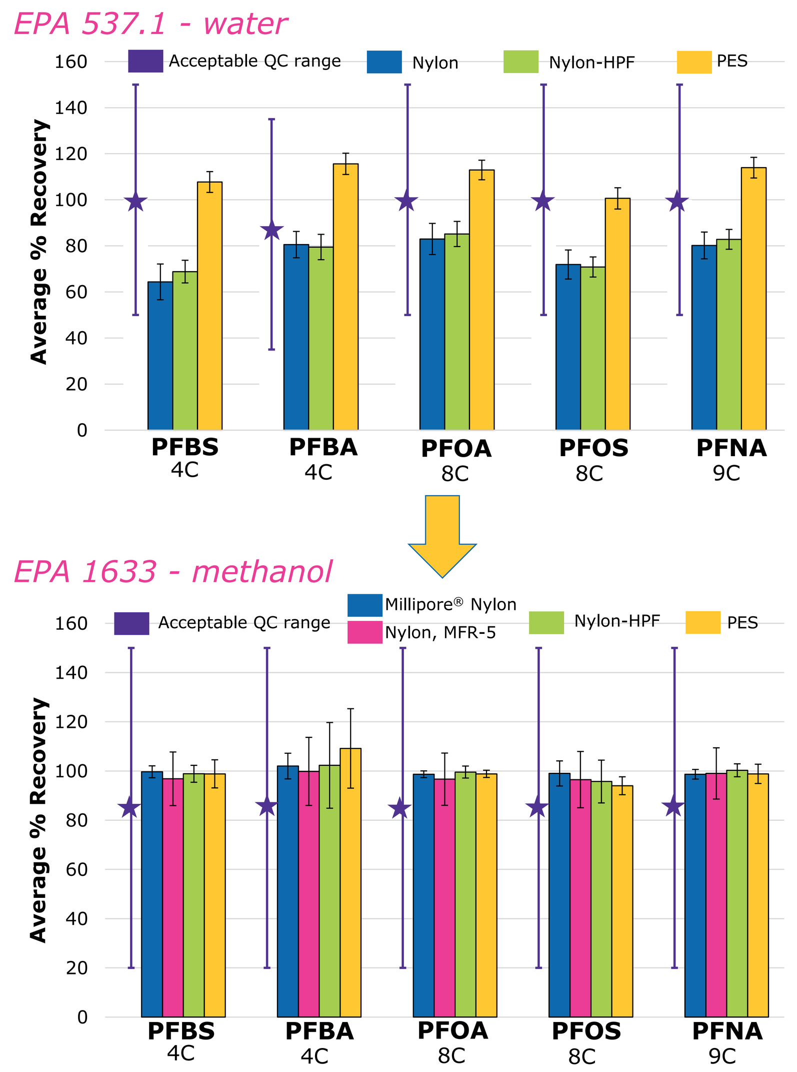
The **goal of this series of studies** was to draw parallels between the performance of membrane filters in PFAS analytical methods and their physicochemical characteristics, in order to recommend the best filters to use depending on the method parameters.

Consideration #1: Filter Material

There are many types of filter materials

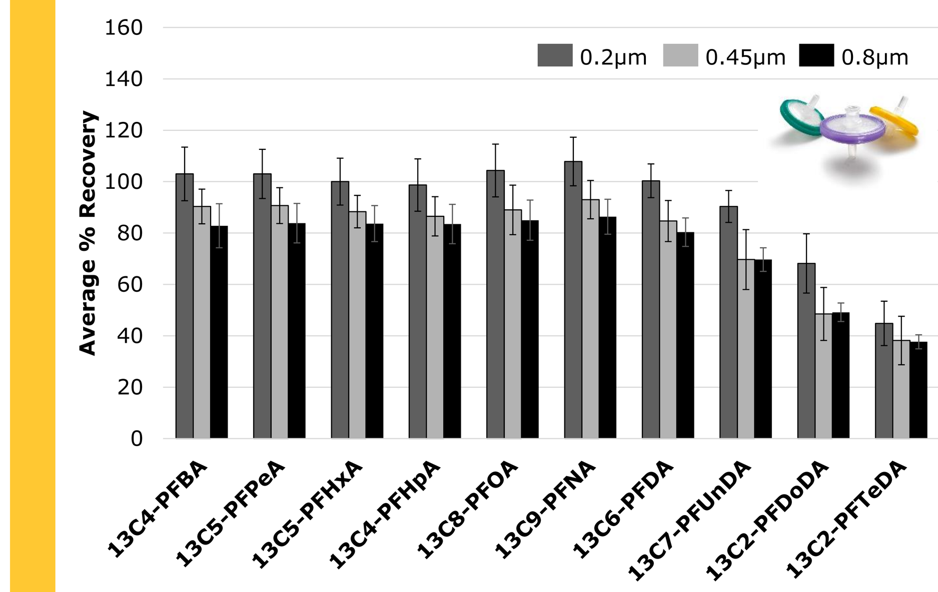


Filter material influences PFAS binding



Consideration #2: Pore Size

Smaller pore sizes may lead to higher recovery



Recovery of PFCAs by MCE of various pore sizes

There may be a slightly negative relationship in recovery with increasing pore size, from 0.2 to 0.8µm, in water.

This trend was seen up through 5.0µm pores and in both methanol and water (*not shown*) and may be caused by changes in diffusional gradients of small molecules within filter pores.

However, not all syringe filters with the same pore size retain particles in the same way

MFR	Material	% Retention
MilliporeSigma	Nylon	100.0 ± 0.10
	PES	69.4 ± 28.1
	PP	57.1 ± 3.5
MFR-5	Nylon	98.0 ± 1.1
	PP	25.3 ± 0.90
MFR-3	PP	7.9 ± 1.1
MFR-2	RC	15.8 ± 2.2

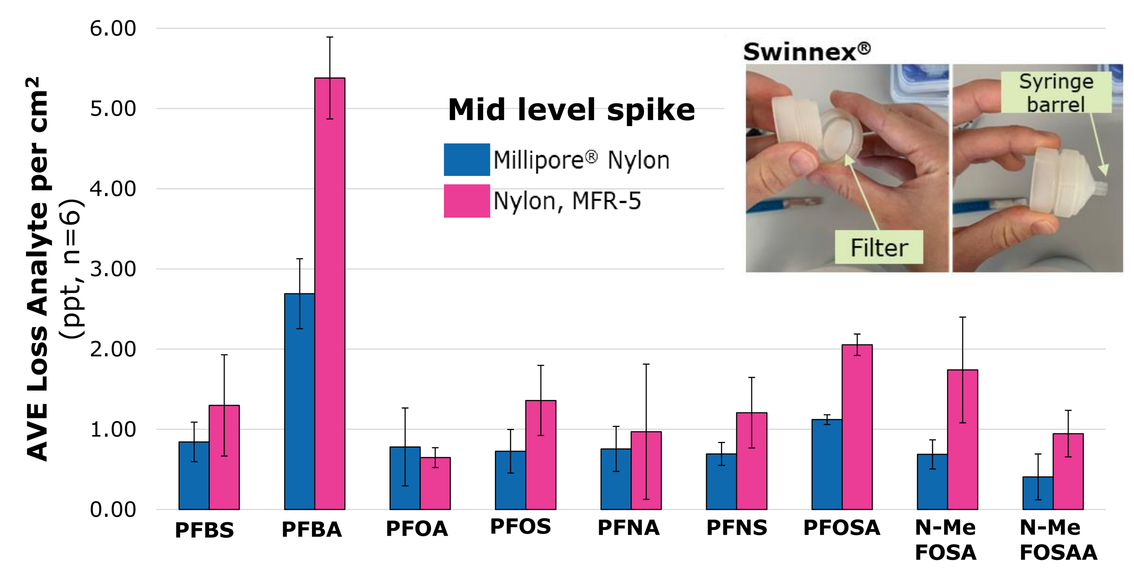
Table 2: Percent retention of 0.24 µm diameter polystyrene beads (0.05% solution) by various 0.2 µm pore size syringe filters

Not all syringe filters perform the same way

Why?

- Pore size definition and type of testing
- Force involved in pressure-based filtration devices
- Manufacturing approaches and lot-to-lot variation
- Chemical interactions with particles or aggregation at the surface

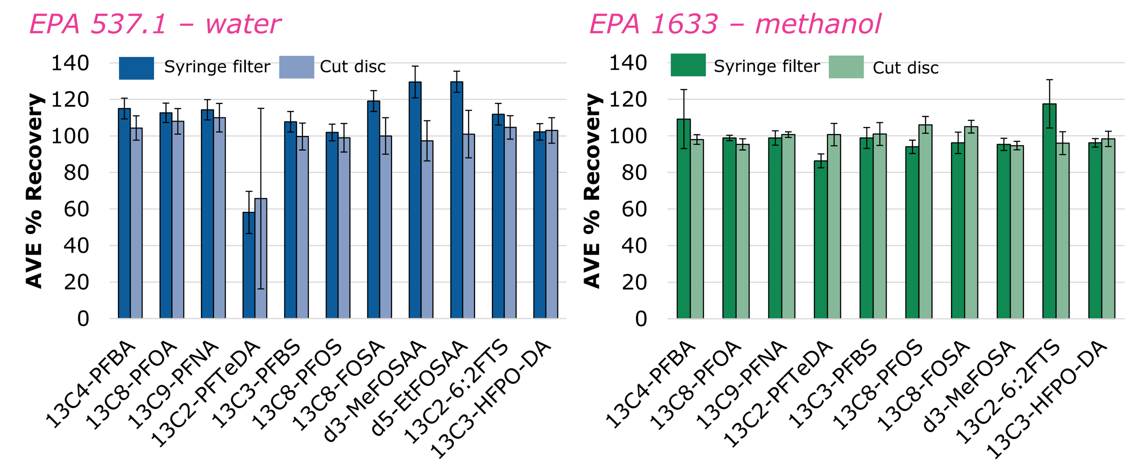
Consideration #3: Diameter & Format



Effect of Filter Diameter on Recovery of PFAS: 25mm vs. 33mm

For nearly every compound, a Millipore® nylon (33mm diameter) filter demonstrated better average recovery per cm² versus a device of 25mm. Thus, larger filtration areas do not necessarily lead to more analyte loss.

Syringe filters and cut discs perform similarly

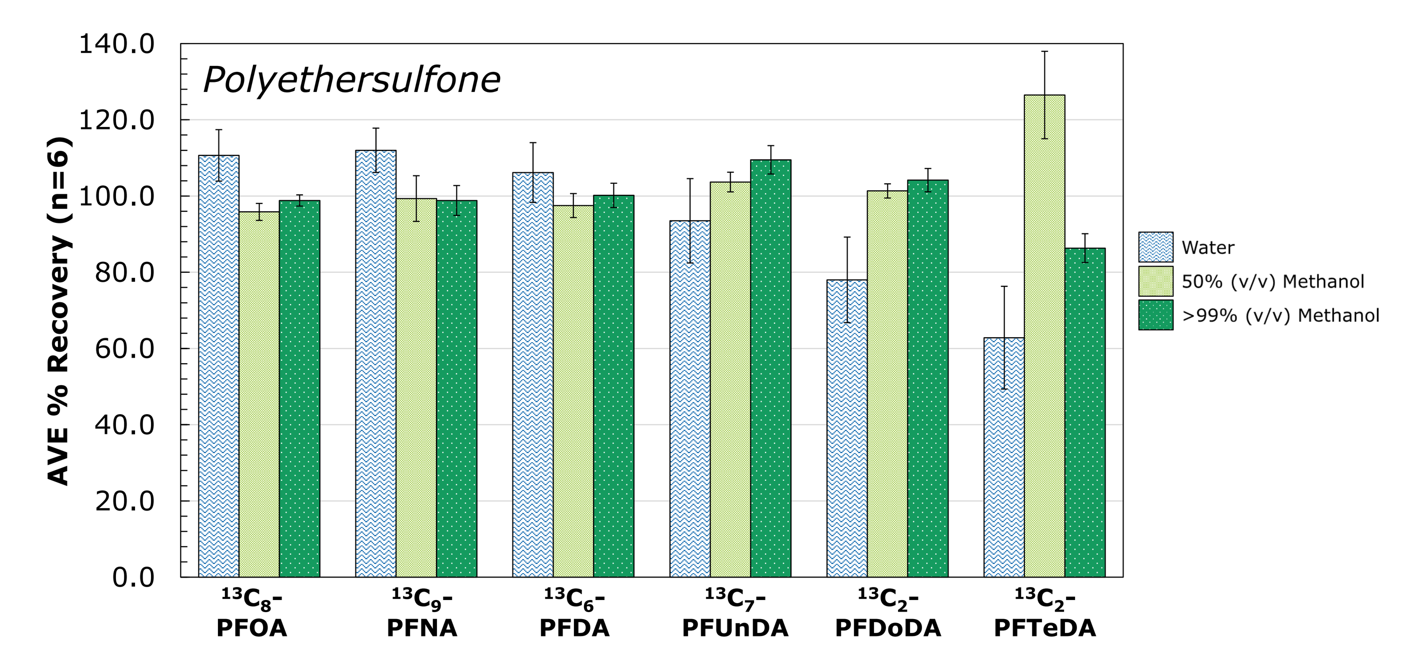


Effect of Filter Format on Recovery of PFAS: Disc-type vs. syringe filter

Recovery performance of 0.2µm PES is similar for most PFAS compounds in both disc-type and syringe filter, regardless of solvent used (*left: water; right: methanol*)

Consideration #4: Solvent

ASTM 8421 demonstrates that PFAS Recovery increases with methanol content for many filter types



Adding even a small amount of solvent increases recovery dramatically, even for non-polar filter types. PES recovery of long chain PFCAs was the highest in a mixture of polar and non-polar solvents.

Excellent recovery of even the longest chain PFCAs in 50% (v/v) methanol (via ASTM 8421) seen for all filter types. Similar results observed for philic polypropylene.

Summary & Conclusions

Filtration is a simple and cost-effective way to prepare samples for LC-MS/MS analysis. However, for PFAS methods, it is necessary to carefully consider filter type, pore size, format, manufacturer and method parameters for the best filter performance.

References

[1] Lozeau, L.D. & Dube, M., *Analytix Reporter*, **2024**, *16*, 28-31

[2] Lath, S., *et al. Chemosphere*, **2019**, *222*, 671-8.

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