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Application Note

Analysis of PFAS Extractables in Mixed Cellulose Esters (MCE) Syringe Filters and Filter Membranes

Abstract

PFAS test methods require highly sensitive instrumentation and low limits of detection. Contamination from fluorinated extractables in syringe filters and other consumables used in PFAS workflows can interfere with analysis. We evaluated levels of PFAS extractable contaminants from Mixed Cellulose Esters (MCE) filters using EPA 537.1. We also determined PFAS recoveries using C-13 labeled standards. Our results suggest that Millex® MCE syringe filters and Millipore® MCE cut disc filter membranes are appropriate for use in PFAS test methods.

Introduction

Perfluoroalkyl Substances (PFAS) represent a class of chemicals utilized in a variety of industries. Unfortunately, broad use has led to their persistent accumulation in environmental matrices. Further, mounting evidence of the negative health impacts of PFAS combined with regulations evolving at an unprecedented pace pose a significant analytical challenge.

Current regulations necessitate the use of PFAS test methods with very low limits of detection. The ubiquity of PFAS in the environment increases concern that consumables such as collection bottles, tubing components, syringe filters, and others could be sources of contamination, interfering with high sensitivity analysis. Therefore, it is crucial minimize contamination by using consumables that do not contain fluorinated materials. Toward this end, we evaluated levels of PFAS extractable contaminants in Mixed Cellulose Esters (MCE) syringe filters and cut disc filter membranes using EPA 537.1. We also determined recovery of PFAS analytes using C-13 labeled standards. Our data show that Millex® MCE syringe filters and Millipore® MCE cut disc filter membranes exhibit low levels of PFAS extractables below reporting and minimum detection limits while sustaining high recovery of PFAS analytes, making them suitable for use in PFAS testing workflows.

Materials and Methods

The following filtration devices were tested for PFAS extractables in triplicate:

- Millex® MCE syringe filter devices with pore size ratings of 0.2 µm, 0.45 µm, and 0.8 µm
- Millipore® MCE cut disc filter membranes with pore size ratings of 0.45 µm, 0.8 µm, and 5 µm in Swinnex® filter holders

An overview of the modified method EPA 537.1 is detailed in **Figure 1**, with LC-MS/MS conditions in **Table 1.** Testing was conducted in collaboration with SGS North America at their Orlando, FL facility. Briefly, a 250 mL PFAS-free DI water sample was spiked with surrogates. An internal standard spike of 0.08 ppb was used for QC blanks. To determine if sample filtration media contributes to PFAS contamination, the entire sample was passed through the filter and into a Styrene Divinylbenzene (SDVB) SPE cartridge. Sample bottles and tubes were rinsed with basic methanol and also passed through the filter and into the cartridge. The entire sample was then subjected to SPE and concentrated to 1 mL in 96:4% (v/v) methanol:water prior to LC-MS/MS analysis using a C18 column. Analysis was performed using internal standards; C-13 labeled standards were used in this study.

Modified EPA 537.1

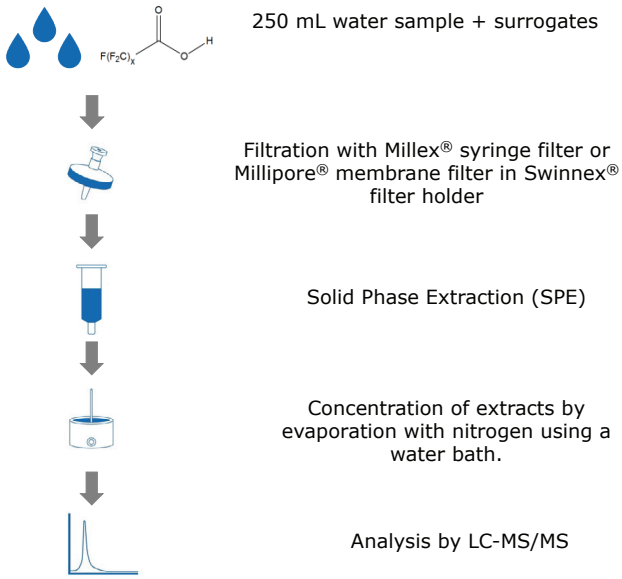

Figure 1. Workflow for analysis of PFAS extractables from MCE filters using modified EPA 537.1.

Table 1. LC and MS/MS conditions used in this study.

Column	C18, 100 x 2.1 mm ID, 2.7 µm superficially porous particles			
Mobile phase	[A] DI Water, 0.1% (v/v) acetic acid; [B] Methanol (MeOH), 0.1% (v/v) acetic acid			
Gradient	**Time (min)**	**A%**	**B%**	**Flow (mL/min)**
	0–0.0	65%	35%	0.4
	0–7.0	0%	100%	0.4
	7.0–10.0	0%	100%	0.7
	10.0–11.0	0%	100%	0.7
	11.0–15.0	65%	35%	0.4
Column temp	50.0 °C			
Injection volume	3–5 µL autosampler injection			
Sample	SPE eluate concentrated to 1 mL methanol: water, 96:4% (v/v)			
MS/MS conditions	**Parameter**			**Value**
	Gas Temp (°C)			250
	Gas Flow (L/min)			10
	Nebulizer (psi)			50
	Sheath Gas Heater			275
	Sheath Gas Flow (L/min)			10
	V Charging			600
	Ionization Mode			Neg ESI
	Collision Cell Gas (PSI)			40
	Collision Cell Gas			UHP N2

Results

Levels of PFAS contaminants

There were no detectable PFAS contaminants above the Reporting Limit (RL) or the Minimum Detection Limit (MDL) in any of the MCE filter products tested using modified EPA 537.1 (**Table 2**). These results suggest that Millex® MCE syringe filters and Millipore® MCE cut disc filter membranes are reliable and appropriate to utilize for filtration in the analysis of these PFAS compounds.

Table 2. Analyte detection by compound, performed in triplicate. ND= Not Detected; MCE = Mixed Cellulose Esters

Compound	Millex® syringe filters			Millipore® cut disc filter membranes		
	0.2 µm MCE	0.45 µm MCE	0.8 µm MCE	0.45 µm MCE	0.8 µm MCE	5 µm MCE
Perfluoroalkyl carboxylic acids (PFCAs)						
PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUnDA, PFDoDA, PFTrDA, PFTeDA	ND	ND	ND	ND	ND	ND
Perfluorosulfonic acids (PFSAs)						
PFBS, PFPeS, PFHxS, PFHpS, PFOS, PFNS, PFDS	ND	ND	ND	ND	ND	ND
Perfluorooctanesulfonamides/ Perfluoroctanesulfonamidoacetic acids						
PFOSA, MeFOSAA, EtFOSAA	ND	ND	ND	ND	ND	ND
Fluorotelomer sulfonates						
4:2 FTS/4:2 FTSA, 6:2 FTS/6:2 FTSA, 8:2 FTS/8:2 FTSA	ND	ND	ND	ND	ND	ND
Next-generation PFAS analytes						
HFPO-DA (GenX), ADONA, 9Cl-PF3ONS (F-53B Major), 11Cl-PF3OUdS (F-53B Minor)	ND	ND	ND	ND	ND	ND

PFAS recovery

Previous studies showed excellent recovery of C-13 labeled PFAS compounds after filtration using Millex® polyethersulfone (PES) syringe filters. Thus, we determined the recovery of C-13 labeled PFAS compounds using 0.22 µm and 0.45 µm Millex® MCE syringe filters (**Figures 2-3**) as well as 0.45 µm Millipore® MCE cut disc filter membranes (**Figure 4**) and compared them to recoveries of C-13 labeled PFAS compounds filtered using Millex® PES syringe filters and Millipore® PES cut disc filter membranes with the same pore size ratings. The recoveries of all C-13 labeled standards were within the acceptable QC range for the method[1] when using both MCE and PES filters. However, recovery varied slightly based on

[1]Recoveries for Millex® MCE and PES syringe filters and Millipore® MCE cut disc filter membranes were determined according to the modified EPA 537.1. The QC limits varied from compound to compound, from 30-150%.

both filter material and compound (**Figures 2-4**). For example, 0.45 µm PES filters in both syringe filter and cut disc filter membrane format demonstrated generally higher recoveries of nearly all tested PFAS compounds compared to MCE. The largest difference in recovery for any condition was with PFOSA, where PES showed between 108-120% recovery versus MCE which was considerably lower (still within the QC range), between 36-60% recovery.

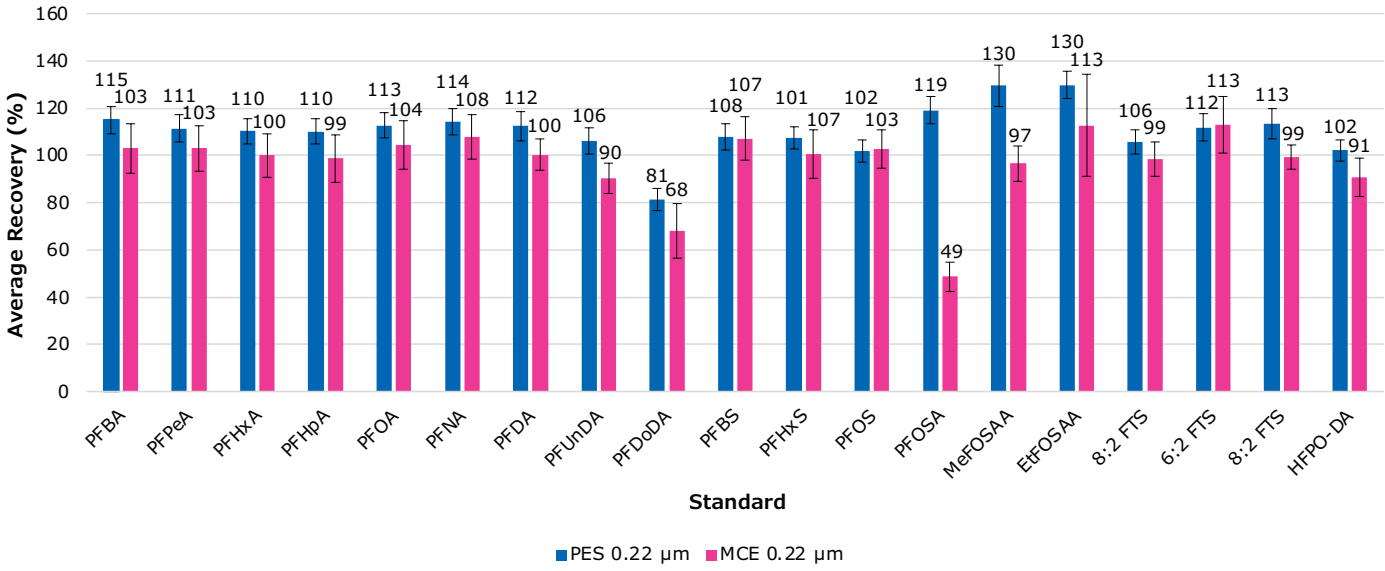

Figure 2. The average percent recovery of C-13 labeled PFAS standards after filtration with 0.22 µm MCE and PES Millex® syringe filter devices (mean ± standard deviation, n=3 replicates).

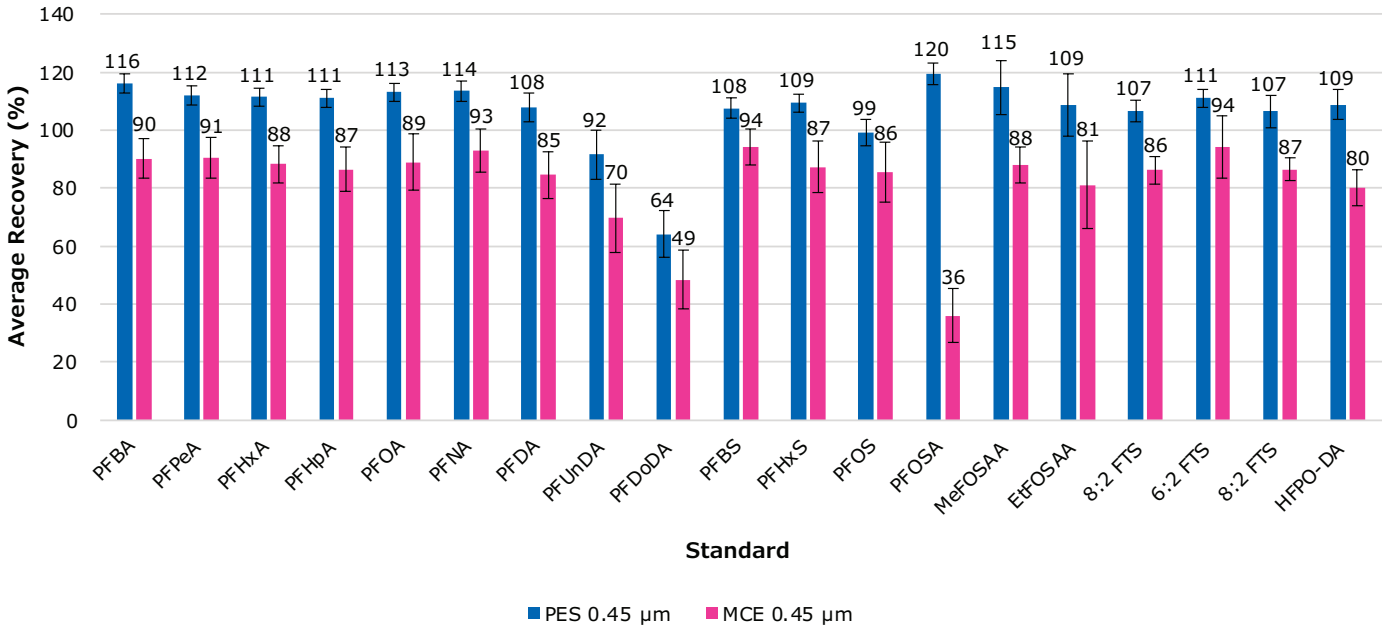

Figure 3. The average percent recovery of C-13 labeled PFAS standards after filtration with 0.45 µm MCE and PES Millex® syringe filter devices (mean ± standard deviation, n=3 replicates).

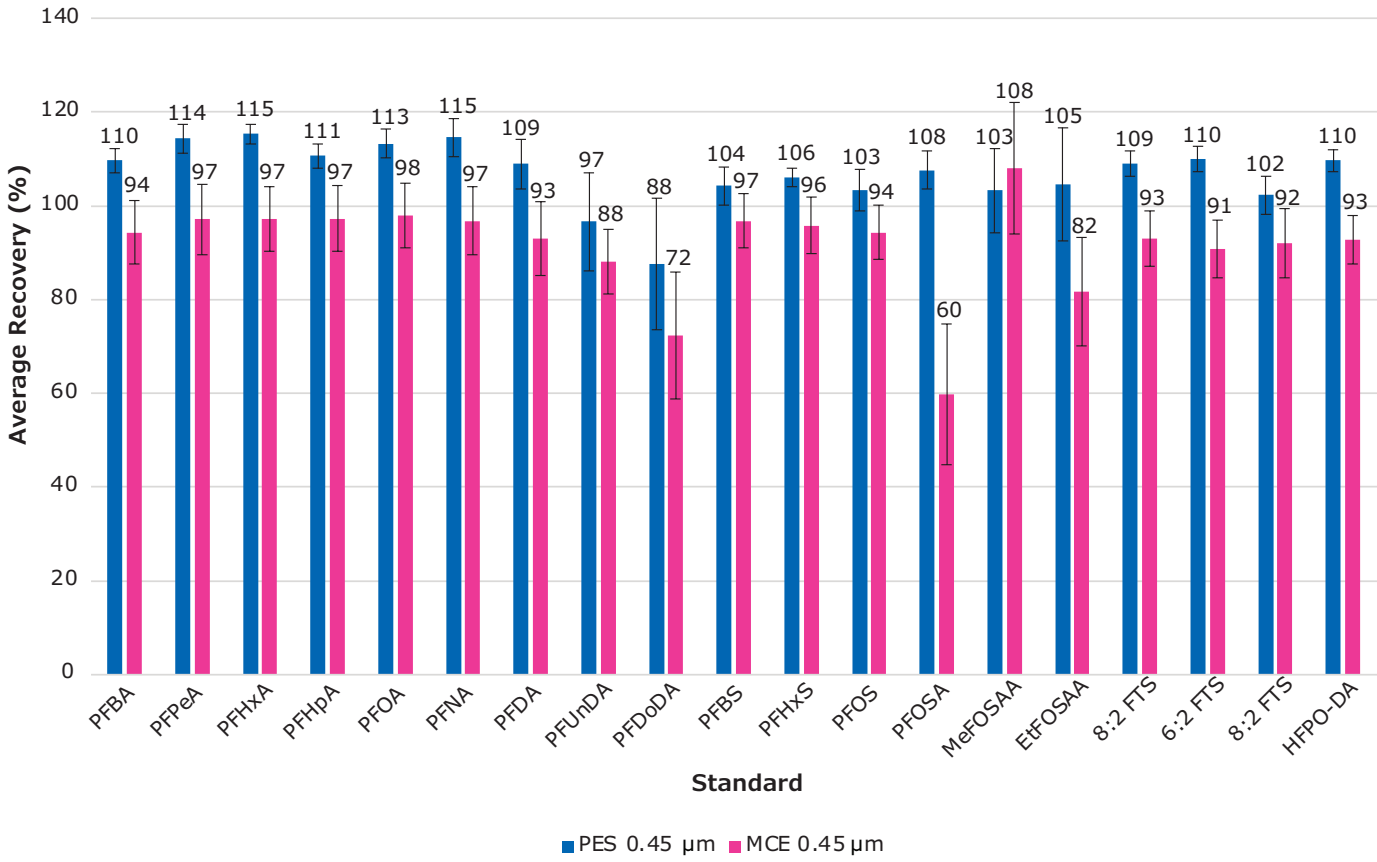

Figure 4. The average percent recovery of C-13 labeled PFAS standards after filtration with 0.45 µm MCE and PES Millipore® cut disc filter membranes (mean ± standard deviation, n=3 replicates).

We further compared recoveries of PFAS6 compounds using MCE and PES filters. PFAS6 refers to a group of six PFAS compounds [PFOS, PFOA, PFHxS, PFNA, HFPO-DA (GenX), and PFBS] for which the U.S. Environmental Protection Agency (EPA) has set Maximum Contaminant Levels (MCLs) in drinking water through the National Primary Drinking Water Regulation (NPDWR) framework. Under the tested conditions, 0.22 µm Millex® PES and MCE syringe filters exhibited comparable performance in terms of PFAS6 recoveries. 0.45 µm PES Millex® syringe filter devices exhibited higher PFAS6 recoveries compared to 0.45 µm MCE Millex® syringe filter devices. 0.45 µm PES and MCE cut disc filter membranes exhibited comparable PFAS6 recoveries. Data are summarized in **Table 3**.

Table 3. PFAS6 recovery using Millex® MCE syringe filters, Millex® PES syringe filters, Millipore® MCE cut disc filter membranes and Millipore® PES cut disc filter membranes.

Membrane	PFAS6 Compound Recovery (mean ± standard deviation n=3 replicates)					
	PFOA	PFNA	PFBS	PFHxS	PFOS	HFPO-DA
MCE syringe filter, 0.22 µm	104 ± 10	108 ± 9	107 ± 9	101 ± 10	103 ± 8	91 ± 8
MCE syringe filter 0.45 µm	89 ± 10	93 ± 7	94 ± 6	87 ± 9	86 ± 10	80 ± 6
MCE cut disc filter, 0.45 µm	98 ± 7	97 ± 7	97 ± 6	96 ± 6	94 ± 6	93 ± 5
PES syringe filter, 0.22 µm	113 ± 5	114 ± 6	108 ± 6	107 ± 5	102 ± 5	102 ± 5
PES syringe filter 0.45 µm	113 ± 3	114 ± 3	108 ± 3	109 ± 3	99 ± 5	109 ± 5
PES cut disc filter, 0.45 µm	113 ± 3	115 ± 4	104 ± 4	106 ± 2	103 ± 5	110 ± 2

Our data also showed little effect of tested pore size ratings on PFAS recovery using both Millex® MCE syringe filters and Millipore® cut disc filter membranes for the select PFAS6 compounds (**Figure 5**-**6**). However, for syringe filters, there may be a slightly negative relationship in recovery with increasing pore size, which may be caused by changes in diffusional gradients of small molecules within filter pores and would require further investigation. Similar trends were seen for all the C-13 labeled standards.

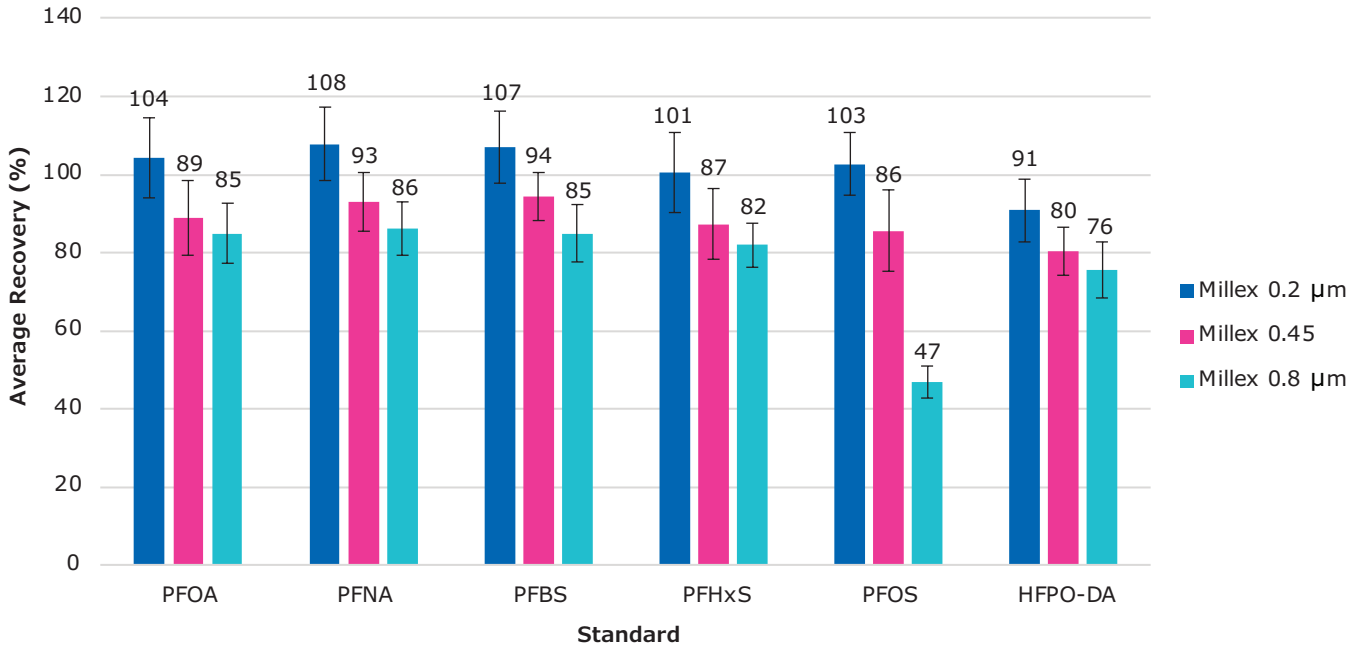

Figure 5. Average recoveries of PFAS standards using 0.2 µm, 0.45 µm, and 0.8 µm Millex® MCE syringe filters.

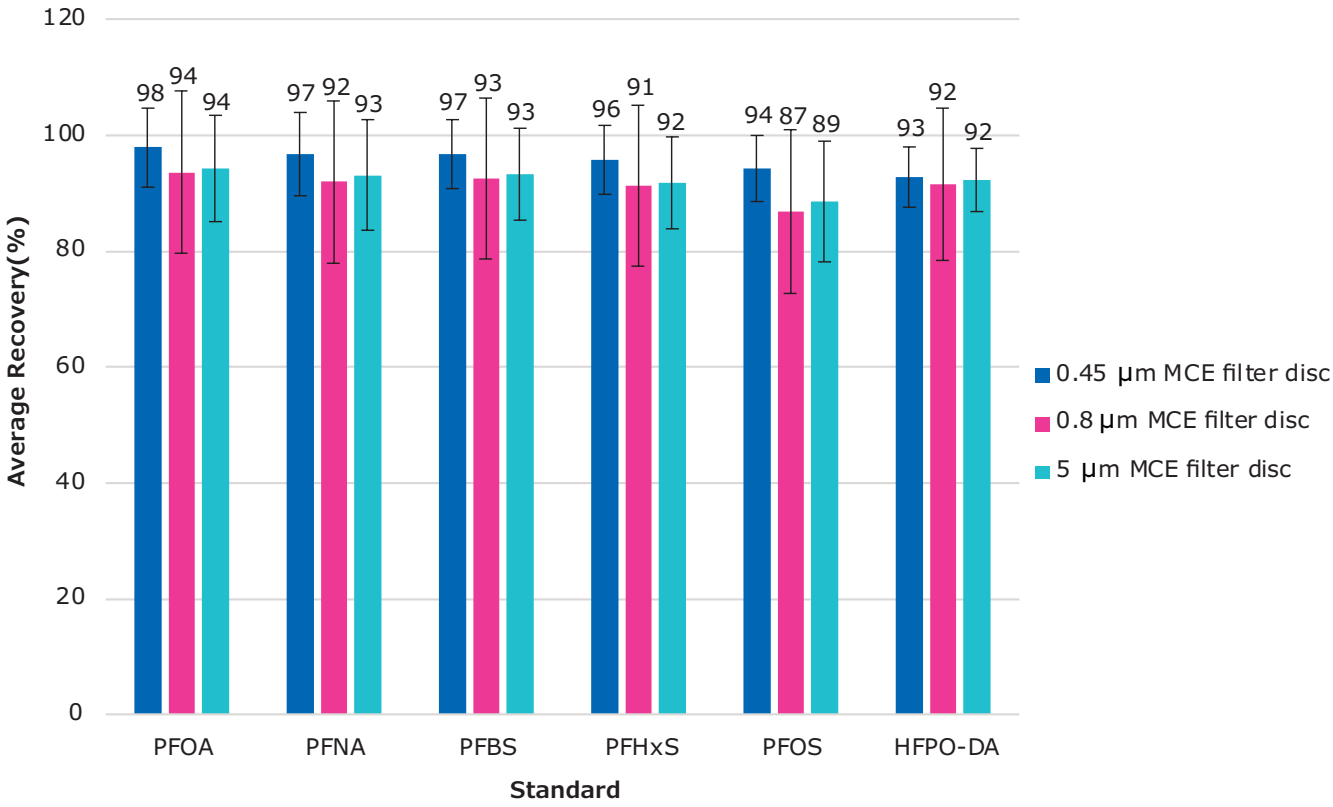

Figure 6. Average recoveries of PFAS standards using 0.45 µm, 0.8 µm, and 5 µm Millipore® MCE cut disc filter membranes.

Conclusions

There are many considerations when selecting a filter for a particular analytical method. Filters should be compatible with sample and chemical components, exhibit low levels of extractables that might interfere with data interpretation, and yield high recovery for the analyte-of-interest. For PFAS methods, it is necessary to carefully consider filter material, pore size, format, manufacturer, and method parameters for the best filter performance. Our data show that mixed cellulose esters (MCE) Millex® syringe filters and Millipore® cut disc filter membranes are suitable for use in PFAS test methods.

Recommended Products

Millex® Syringe Filters

Product	Pore size	Diameter	Product Number
Millex® MCE syringe filters	0.22 µm	25 mm	**SLGS025**
	0.45 µm	25 mm	**SLHA025**
	0.8 µm	25 mm	**SLAA025**

Millipore® Cut Disc Filter Membranes

Product	Pore size	Diameter	Product Number
Millipore® MCE Cut Disc Filter Membranes	0.22 µm	13 mm	**GSWP01300**
		25 mm	**GSWP02500**
		47 mm	**GSWP04700**
		90 mm	**GSWP09000**
	0.45 µm	13 mm	**HAWP01300**
		25 mm	**HAWP02500**
		47 mm	**HAWP04700**
		90 mm	**HAWP09000**
	0.8 µm	13 mm	**AAWP01300**
		25 mm	**AAWP02500**
		47 mm	**AAWP04700**
		90 mm	**AAWP09000**
	5.0 µm	13 mm	**SMWP01300**
		25 mm	**SMWP02500**
		47 mm	**SMWP04700**
		90 mm	**SMWP09025**
Millipore® PES Cut Disc Filter Membranes	0.22 µm	25 mm	**GPWP02500**
		47 mm	**GPWP04700**
		90 mm	**GPWP09050**
	0.45 µm	25 mm	**HPWP02500**
		47 mm	**HPWP04700**
		90 mm	**HPWP09050**

Filter Holders

Image	Product	Product Number
	Swinnex® Filter Holder, 13 mm diameter	**SX0001300**
	Swinnex® Filter Holder, 25 mm diameter	**SX0002500**
	Swinnex® Filter Holder, 47 mm diameter	**SX0004700**
	Millicup™-FLEX, 47 mm Disposable Filtration Unit	**MCFLX4710**
	Millipore® All-Glass Filter Holder Kit • 47 mm, Glass frit membrane support, 300 mL funnel	**XX1514700**
	Millipore® All-Glass Filter Holder Kit • 47 mm, Glass frit membrane support, 500 mL funnel	**XX5514700**
	Millipore® All-Glass Filter Holder Kit • 90 mm, Glass frit membrane support, 1 L Funnel	**XX1019022**

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