Millipore®
Preparation, Separation,
Filtration & Monitoring Products

Application Note

Characterization Of 0.2 µm And 0.45 µm Hydrophilic And Hydrophobic Polypropylene Membrane Filters

Description

Polypropylene (PP) filters are white in color and used for the retention of particles on the filter surface. They are constructed of 100% virgin polypropylene media, which is a strong and flexible material compatible with most organic solvents and contains no fluorinated compounds.

Applications

- Aqueous and organic solvent filtration
- Suitable for PFAS methods that require filtration
- Clarification of a range of environmental, food and beverage samples prior to analysis
- Gas filtration & air venting (hydrophobic polypropylene membrane filter)
- High air particle retention for air monitoring (hydrophobic membrane filter)

Features

- Broad chemical compatibility
- Low extractables
- Good thermal stability
- High liquid flow rates
- Hydrophobic membranes available in 0.2 µm and 0.45 µm pore sizes, and 25, 47, 90 mm diameters
- Hydrophilic membranes do not need pre-wetting for aqueous samples and solvents. They are available in 0.2 µm and 0.45 µm pore sizes, and 13, 25, 47, 90 mm diameters.

Technical Data - Summary

Characteristic	Method	Result
Extractables	HPLC (UV detector)	No extractables detected
PFAS Extractables	LC-MS/MS Modified EPA 537.1 LC-MS/MS Modified EPA 1633	No PFAS extractables detected
Particle Retention (Aqueous)	UV-Vis	Particle retention of 0.2 µm and 0.45 µm hydrophilic and hydrophobic membranes determined for polystyrene beads of 0.28 µm, 0.4 µm and 0.784 µm diameters
Particle Retention (Air)	Particle Counter	Greater than or equal to 99% retention of 0.1-5 µm airborne particles by 0.2 µm and 0.45 µm hydrophobic PP membranes
Protein Binding	Measurement of radiolabeled IgG binding (µg IgG bound per cm^2 of membrane)	Hydrophobic PP, 0.2 µm: 128.3 ± 12.7 Hydrophobic PP, 0.45 µm: 98.4 ± 8.99 Hydrophilic PP, 0.2 µm:47.4 ± 2.02 Hydrophilic PP, 0.45 µm: 44.4 ± 2.21
Heat Stability	90°C for 1 hour	No visual changes after heating; Little to no variation in flow rate; No difference in ease of handling

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Measurement of extractables in 0.2 µm and 0.45 µm polypropylene membrane filters

Introduction

Hydrophilic PTFE (polytetrafluoroethylene) membrane is the best option for (U)HPLC and LC-MS sample and mobile phase filtration because of its broad chemical compatibility and low extractables. However, the recent concerns on poly- and perfluoroalkylated substances (PFAS) have made it necessary for analytical methods to be free of any possible fluorinated contaminants, including membrane filters used for sample and solvent preparations. For applications that require non-fluorinated membranes for sample and solvent filtration, we show that hydrophilic and hydrophobic polypropylene (PP) membrane filters have low extractables profiles similar to PTFE membranes and thus are an appropriate alternative for sample preparation in HPLC, UHPLC, and LC-MS/MS, including PFAS-related workflows.

Method Overview

Polypropylene and PTFE filters were soaked for two hours under the following conditions:

Extracting Medium	Membrane pre-heating	Notes
70 % v/v acetonitrile/water	No	Acetonitrile is a commonly used mobile phase
Methanol	No	Methanol is a commonly used mobile phase
Methanol	120° C for 1 hr then cooled to room temperature before soak	The maximum filter operating temperature for OTM-45 (PFAS testing in air) is 120° C
Acidic methanol (0.1 % v/v formic acid in methanol)	No	0.1% formic acid in methanol is a common mobile phase
Basic methanol (5 % v/v ammonium hydroxide in methanol)	No	Filtration step in EPA draft 1633 (PFAS testing) has a basic methanol matrix; OTM-45 (PFAS testing) uses basic methanol as extracting medium

Extracts from the 2-hour soak were collected and analyzed by HPLC.

HPLC column	Acquity UPLC® BEH HILIC, 1.7 µm, 50 x 2.1 mm ID		
Mobile Phase	[A] 0.1% formic acid in Milli-Q water [B] 0.1% formic acid in LC-MS grade acetonitrile		
	Time (min)	**A %**	**B %**
	0.0	100	0
	2.0	0	100
	2.6	0	100
	3.2	100	0
	4.0	100	0
Flow rate (mL/min)	0.42		
HPLC instrument	Waters Xevo G2-S QToF		
Detector	PDA		

Results

The overall extractables results are summarized in Table 1, where a checkmark indicates that no significant extractables were detected under the extraction conditions specified and the method used to analyze them (HPLC with UV detector).

Extraction Condition	Membrane Filter					
	Hydrophilic			Hydrophobic		
	0.2 µm PP	0.45 µm PP	0.2 µm PTFE	0.2 µm PP	0.45 µm PP	0.2 µm PTFE
Acetonitrile	✓	✓	✓	✓	✓	✓
Methanol	✓	✓	✓	✓	✓	✓
Methanol/ pre-heated membrane	✓	✓	✓	✓	✓	✓
Acidic Methanol	✓	✓	✓	✓	✓	✓
Basic Methanol	✓	✓	✓	✓	✓	✓

Table 1: Summary of extractables levels for polypropylene (PP) and PTFE membranes. Checkmarks indicate low-level extractables.

The HPLC extractables profiles for hydrophilic and hydrophobic polypropylene and PTFE membranes with 70 % (v/v) acetonitrile as the extraction medium are shown in **Figure 1**. Membranes tested under the other extraction conditions (methanol, acidic and basic methanol, methanol with pre-heated membrane) have similar profiles (not shown).

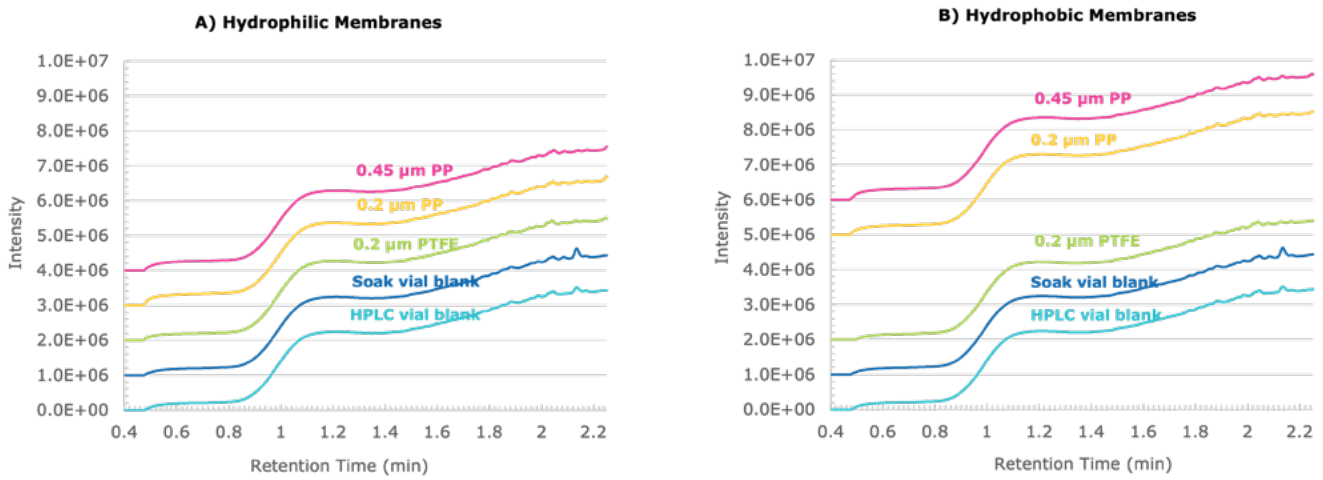


Figure 1: Representative extractables profiles (UV diode array) of (A) hydrophilic and (B) hydrophobic PP and PTFE membranes in 70 % (v/v) acetonitrile in Milli-Q® water.

Extractables would show up as peaks that are not present in blanks. Based on the HPLC extractables profiles, the polypropylene membranes do not have significant chemical extractables. They performed similarly to the PTFE membranes, indicating they may be used in filtering samples and mobile phases for (U)HPLC and LC-MS/MS, especially for PFAS-related analytical methods.

Measurement of PFAS Extractables in 0.2 µm and 0.45 µm polypropylene membrane filters

Introduction

Concern over poly- and perfluoroalkylated substances (PFAS) contamination continues to grow. To obtain accurate data, PFAS testing using advanced instrumentation techniques such as LC-MS/MS requires that instruments, chemicals, and labware are free of any possible PFAS contaminants, including the membrane filters that are used for sample and solvent preparations. In this work, modified EPA 537.1 and EPA Draft 1633 methods were used to show that there are no detectable PFAS contaminants in any of the polypropylene cut disc membrane filters tested.

Method Overview

Figure 2 describes the testing procedure for measuring PFAS extractables in polypropylene membrane filters. Both hydrophobic and hydrophilic 0.2 µm and 0.45 µm polypropylene membrane filters were tested using modified EPA 537.1, and only hydrophilic membranes were tested using modified EPA Draft 1633. Details of the experiments can be found in our **Technical Article**.

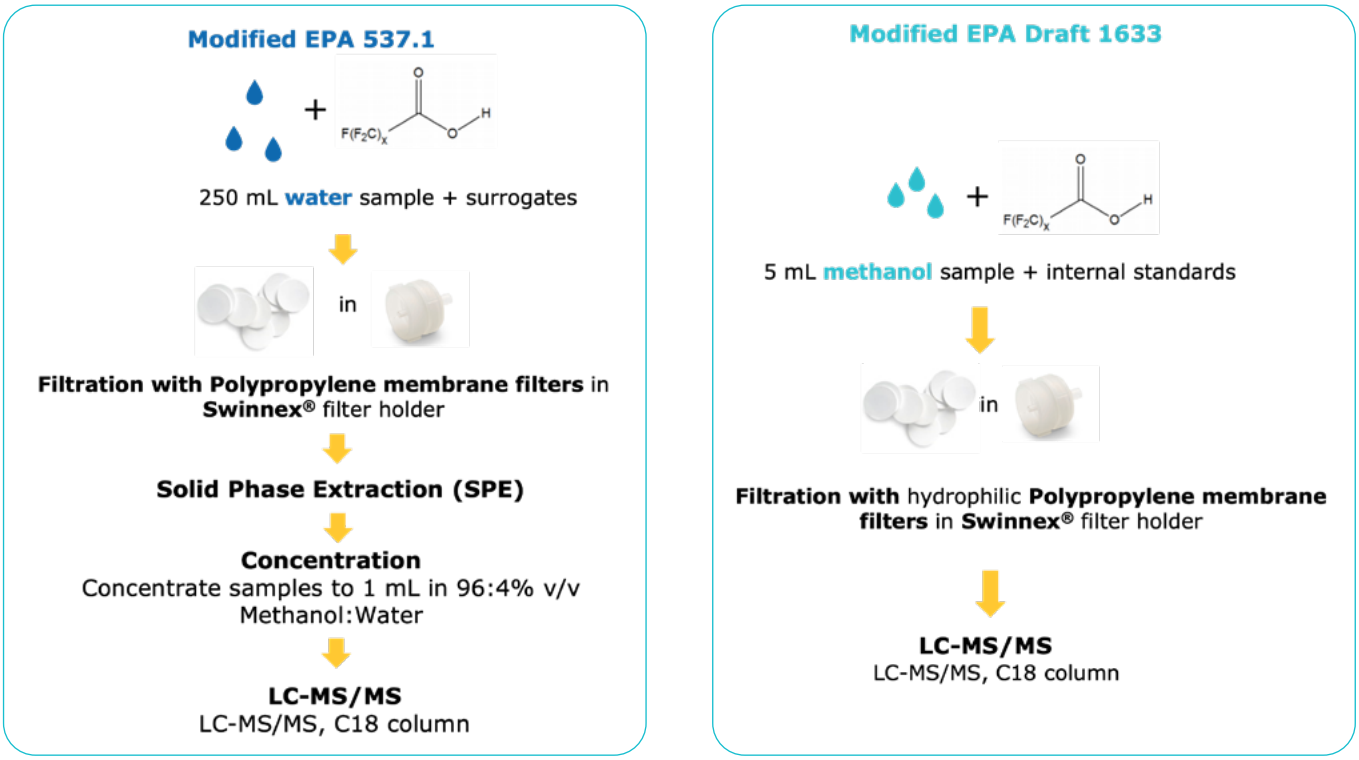


Figure 2. Schematic of the modified EPA 537.1 and modified EPA Draft 1633 method that were used to measure PFAS extractables in polypropylene membrane filters.

Assembly of polypropylene membrane filters in Swinnex® filter holder is shown in **Figure 3**.

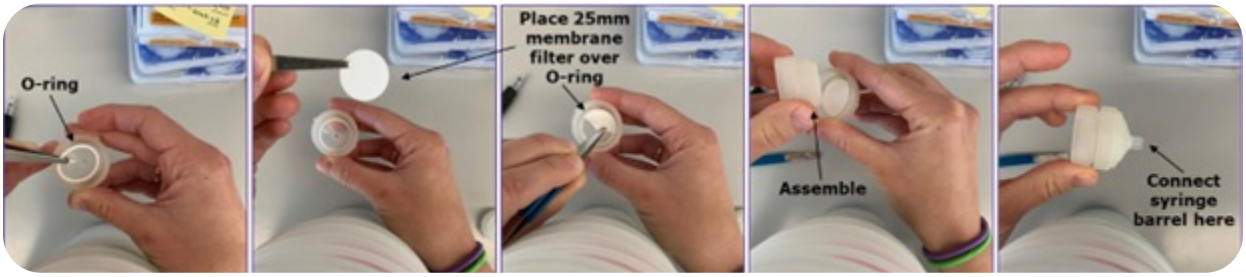


Figure 3: Assembly of a 25 mm polypropylene membrane filter in a Swinnex® filter holder.

Results

Table 2 summarizes the results for the PFAS extractables experiments. There were no detectable PFAS contaminants in any of the polypropylene cut disc membrane filters that were tested according to modified EPA 537.1 and modified EPA draft 1633. This indicates that these membranes are suitable for filtering samples and/or solvents in PFAS testing methods.

PFAS compounds	Hydrophilic PP		Hydrophobic PP
	Modified EPA 537.1	Modified EPA Draft 1633	Modified EPA 537.1
Perfluoroalkyl Carboxylic Acids	Not detected	Not detected	Not detected
Perfluoroalkyl Sulfonic Acids	Not detected	Not detected	Not detected
Fluorotelomer Sulfonic Acids	Not detected	Not detected	Not detected
Fluorotelomer Carboxylic Acids	(Not tested)	Not detected	(Not tested)
Perfluorooctane Sulfonamides	Not detected	Not detected	Not detected
Perfluorooctane Sulfonamidoacetic Acids	Not detected	Not detected	Not detected
Perfluorooctane Sulfonamido Ethanols	(Not tested)	Not detected	(Not tested)
Per- and Polyfluoroether Carboxylic Acids	Not detected	Not detected	Not detected
Per- and Polyfluoroether Sulfonic Acids	Not detected	Not detected	Not detected

Table 2: Summary of PFAS contaminants after filtration with polypropylene membrane (PP) filters in Swinnex® devices. Hydrophilic 0.2 µm and 0.45 µm polypropylene disc filters were tested using modified EPA 537.1; only 0.2 µm hydrophilic membranes were tested using modified EPA Draft 1633. (Detailed information can be found in our **Technical Article**.)

Aqueous particle retention of 0.2 µm and 0.45 µm polypropylene membrane filters

Introduction

Membrane filters used for sample and solvent filtration in analytical applications such as (U)HPLC and LC-MS retain particulate matter that can damage sensitive instrumentation and potentially impact data quality. In general, it is suggested that filters of 0.45 µm pore size are appropriate for HPLC and 0.2 µm filters are appropriate for UHPLC applications. There are many ways that pore size can be measured, such as porosimetry, bubble point, BET surface area, etc., leading to variation in actual pore sizes of polymeric membranes. Particle retention in aqueous solutions is a practical way of determining the pore size of a membrane by challenging it with labeled particles of a known diameter and characterizing the percentage that pass through and goes to the filtrate. We characterized the particle retention of hydrophilic and hydrophobic polypropylene (PP) membrane filters of different pore sizes.

Method Overview

Hydrophilic PP membranes of 0.2 µm and 0.45 µm pore sizes were challenged with 0.01 % polystyrene (PS) bead solutions in phosphate-buffered saline (PBS, pH=7.4). Three bead sizes (diameters) were tested: 0.784 µm, 0.4 µm, and 0.28 µm.

- The PP membranes were placed in Swinnex® devices (assembly described in **Figure 3**).
- 3 mL of polystyrene (PS) bead solutions were passed through the membrane filters.
- 250 µL filtrate was collected, and the absorbance was read at OD (600) using a UV-Vis spectrometer. This was compared to a 6-point calibration curve to calculate percent bead retention.

A similar study was carried out with hydrophobic polypropylene. 0.2 µm and 0.45 µm pore size PP membranes were tested in a Microcon® centrifugal device format (**Figure 4**), so that higher intrusion pressures could be applied using centrifugation at 8,000 x g.

Figure 4: Image of a Microcon® centrifugal device used for testing hydrophobic PP membrane filters

Results

Percent retention of all beads sizes is shown in **Figure 5**. Hydrophilic 0.2 µm PP demonstrated >95 % retention of both 0.4 µm and 0.784 µm beads (97.9±1.07 % and 96.2±0.87 %, respectively). However, for 0.28 µm beads, which are close in diameter to membrane pore size, percent retention was lower, at 57.1±4.90 %. This is not uncommon for fiber-based membranes, where beads can be sterically pushed through pores if the applied pressure is high enough. Further, non-specific interactions at the membrane surface, including surface tension and non-specific binding, can play a role in allowing particles of various sizes through a membrane rated at a certain pore size. This is true, for example, with nylon membranes, which tend to bind particles regardless of size leading to unexpectedly high retentions. 0.45 µm hydrophilic PP passed many 0.28 µm particles (as expected) but showed >80 % retention of both 0.4 µm and 0.784 µm (88.0±3.52 % and 82.9±8.00 %, respectively) bead sizes.

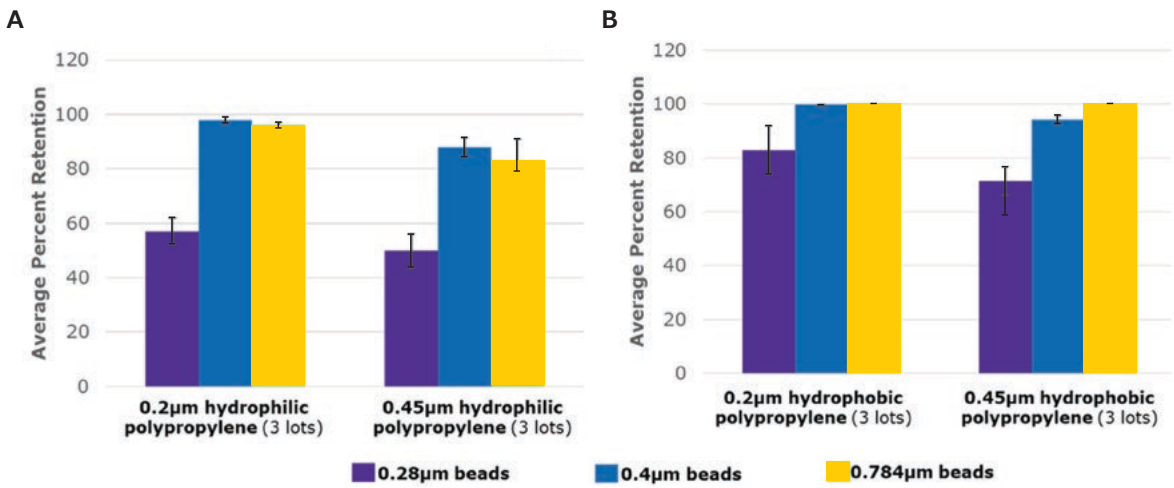


Figure 5: Average percent retention of 0.2 µm and 0.45 µm (A) hydrophilic and (B) hydrophobic PP membrane filters against 0.28 µm, 0.4 µm, and 0.784 µm beads in Swinnex® devices (for hydrophilic PP) and Microcon® devices (for hydrophobic PP). Values are mean ± standard deviation across three lots.

In general, it is expected for hydrophobic surfaces to retain more polymeric particles than hydrophilic counterparts due to hydrophobic surface interactions, and this is shown in **Figure 5**B. Both 0.2 µm and 0.45 µm membranes demonstrated >95 % retention of 0.4 µm beads (99.8±0.17 % for 0.2 µm and 94.3±1.55 % for 0.45 µm membranes) and 0.784 µm beads (100.0±0.08 % for 0.2 µm and 100.0±0.10 % for 0.45 µm membranes). The 0.2 µm hydrophobic PP showed improved retention of 0.28 µm beads (83.0±8.83 %) over its hydrophilic counterpart.

Since both hydrophilic and hydrophobic PP membranes use the same base material with different wettability, it is likely that the percent retention of PS beads is significantly influenced by surface chemistry between the membrane filter material and the particles to be retained, as well as the amount of pressure applied to the filter during the filtration process. Thus, for best results it is always good practice to be aware that retention may be different depending on the type of filter chosen, filtration devices used, and chemistry of analytes and matrices.

Air particle retention in 0.2 µm and 0.45 µm hydrophobic polypropylene membrane filters

Introduction

The ability to retain airborne particles of various sizes can be crucial for membranes that are to be used in air and industrial particle monitoring applications. Additionally, the tendency of certain membranes to retain airborne particles can be different than their tendency to retain liquid particles, due to differences in various physico-chemical properties including surface chemistry, porosity, and flow rate.

This study shows the ability of hydrophobic polypropylene (PP) membranes to retain airborne particles between 0.1 µm and 5 µm diameters.

Method Overview

0.2 µm and 0.45 µm hydrophobic PP membranes were assembled into clean, particle-free Swinnex® devices, as shown in **Figure 3**.

- 100 cfm of ambient air was passed through each assembled Swinnex® device
- Air particles with diameters of 0.1 µm-5 µm that passed through the membranes were characterized and counted by optical means using an automated Lasair® particle counting system.
- Percent retention was calculated based on baseline particle counts in ambient air passed directly through the system (with no membrane).

Results

The average percent retention of various particles in air over n=6 discs is described in Table 3. Of particular interest to air monitoring and particle capture workflows is the ability of certain membranes to retain small particles. Despite having pore sizes of 0.2 µm and 0.45 µm, respectively, hydrophobic PP membranes were able to retain 100% of airborne particles of even 0.1 µm and 0.2 µm diameters.

Membrane	Lot#	Retention of Particles					
		0.1 µm	0.2 µm	0.3 µm	0.5 µm	1 µm	5 µm
0.2 µm hydrophobic PP	Lot 1	100.0±0.00	100.0±0.00	100.0±0.00	100.0±0.00	100.0±0.00	100.0±0.00
	Lot 2	100.0±0.00	100.0±0.00	100.0±0.00	100.0±0.00	97.37 6.45	100.0±0.00
	Lot 3	100.0±0.00	100.0±0.00	100.0±0.00	100.0±0.00	100.0±0.00	100.0±0.00
0.45 µm hydrophobic PP	Lot 1	100.0±0.00	100.0±0.00	100.0±0.00	100.0±0.00	100.0±0.00	100.0±0.00
	Lot 2	99.99±0.00	100.0±0.00	99.99±0.03	100.0±0.00	100.0±0.00	100.0±0.00
	Lot 3	99.99±0.01	100.0±0.01	100.0±0.00	100.0±0.00	100.0±0.00	100.0±0.00

Table 3: The percent airborne particle retention (mean ± standard deviation of n=6 discs) for multiple lots of hydrophobic PP membranes filters

When choosing membrane materials for air monitoring applications, it is important to know the size of the airborne particles of interest and whether or not the chosen membrane filter is able to retain these sizes. In that sense, hydrophobic PP is a good choice for retaining both large and very small airborne particles.

Protein binding characteristic of 0.2 µm and 0.45 µm polypropylene membrane filters

Introduction

The general protein binding behavior of a membrane material is important for applications that require either low or high protein binding membranes. For example, avoiding protein loss due to binding to the membrane surface or interior geometry is essential when filtering a proteinaceous sample. On the other hand, high protein binding capacity is preferred in some applications, such as Western blotting. In this work, IgG binding to hydrophilic and hydrophobic polypropylene (PP) membrane filters was compared with binding to PVDF membranes.

Method Overview

- Membranes were immersed and soaked in a 1 mg/mL goat gamma-globulin solution in phosphate-buffered saline (PBS) containing 125I-(goat IgG) at a concentration of 0.1 µCi/mL*.
- The membranes were washed with PBS three times and assayed for bound radioactivity using a gamma counter.

**Hydrophobic membranes were pre-wetted in isopropanol and then rinsed with Milli-Q® water prior to addition of the tracer solution.*

Results

The level of protein binding of a membrane filter is based on its surface chemistry, thickness, amount of available surface area within the pores, and polymer adsorptive properties. Durapore® hydrophilic PVDF is often used as a low-binding comparison material due to its well-characterized superior low binding behavior, while hydrophobic Immobilon® PVDF is often used as a high-binding comparison material for its well characterized high binding capacity.

Hydrophilic PP membranes demonstrated relatively lower protein binding than the hydrophobic PP membranes (**Table 4, Figure 6**). In terms of pore size, the binding behavior of 0.2 µm and 0.45 µm polypropylene membrane filters were not considerably different.

Millipore® Membrane Type	IgG Binding (µg/cm2)
Hydrophilic PVDF, 0.22 µm	7.2 ± 0.40
Hydrophilic PP, 0.45 µm	44.4 ± 2.21
Hydrophilic PP, 0.2 µm	47.4 ± 2.02
Hydrophobic PP, 0.45 µm	98.4 ± 8.99
Hydrophobic PP, 0.2 µm	128.3 ± 12.7
Hydrophobic PVDF, 0.45 µm (Immobilon®)	310.0 ± 11.2

Table 4: IgG binding of polypropylene membranes compared with PVDF membranes filters (mean ± standard dev of n=3 replicates; 3 lots per membrane tested)

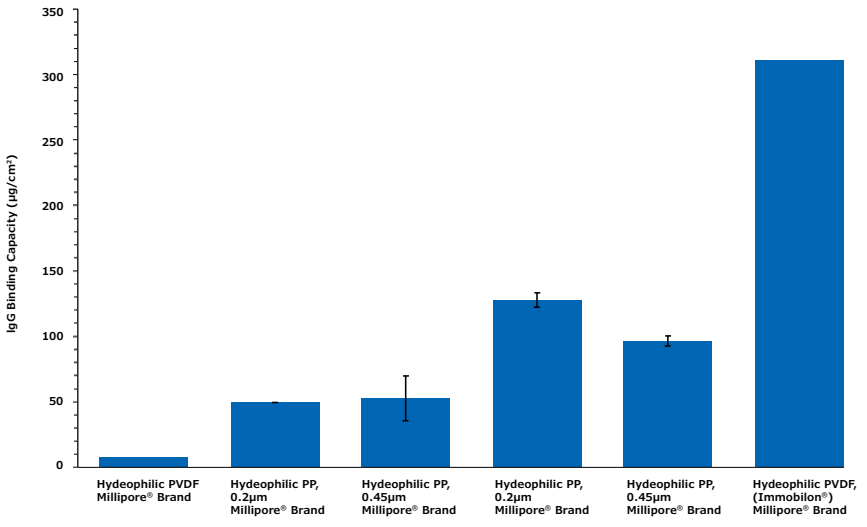


Figure 6. Graphical protein binding comparisons of PP membranes (mean ± calculated standard dev of n=3 lots; 3 replicates per lot tested) compared with high- and low-binding controls (PVDF membranes) displayed in light purple.

Stability to Heating of Polypropylene Membrane Filters

Introduction

Typically, the “stability to heating” specification of a membrane filter indicates whether visual changes (such as color and shape) or other morphological changes are observed upon exposure to elevated temperatures for a particular time. However, functional attributes of membrane filters after heat exposure may be important in cases where they are used in applications such as stack testing, where membrane filters are subjected to high temperatures as they collect analytes and particulates for subsequent analysis. Membrane characteristics such as flow rate or bubble point should be tested after exposure to elevated temperatures to ensure their usability post exposure. We assessed both visual and functional stability of hydrophilic and hydrophobic polypropylene (PP) membrane filters to heating.

Method Overview

0.2 µm and 0.45 µm hydrophobic and hydrophilic PP membranes in a glass dish were placed in an oven at 90°C for 1 hour. The membranes were then allowed to fully cool to room temperature and visual observations were recorded. Flow rate for each membrane post-heating in water (hydrophilic PP) or methanol (hydrophobic PP) was determined and compared to membranes that had never been exposed to heat.

Results

Representative images before and after heating are shown in **Figure 7**. Both hydrophilic and hydrophobic PP membrane filters maintained color and shape after heating. This implies that the membranes can be handled without additional challenges (like curling, which can be observed in other membranes) should they be used after heating.

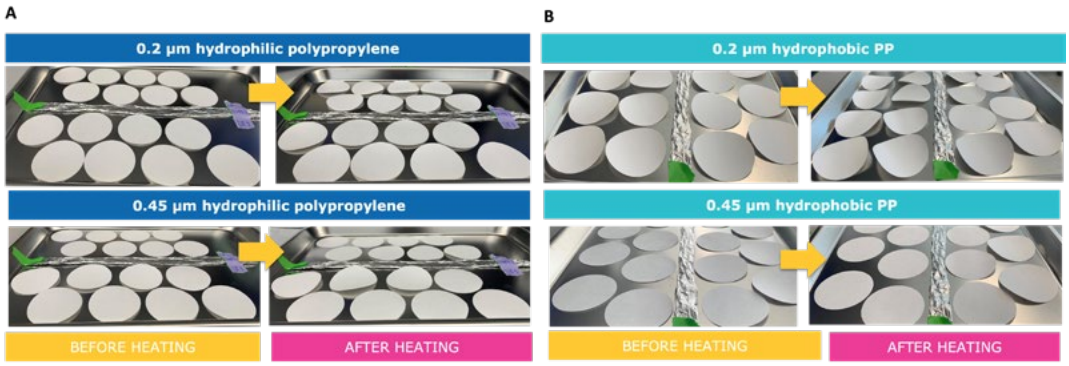


Figure 7: Representative images of (A) hydrophilic and (B) hydrophobic polypropylene membrane filters before and after heating in an oven at 90°C for 1 hour.

The flow rates of the membranes were characterized after exposure to heat. **Figure 8** shows the water flow rate for hydrophilic PP membranes, while **Figure 9** shows the methanol flow rate for hydrophobic PP membranes.

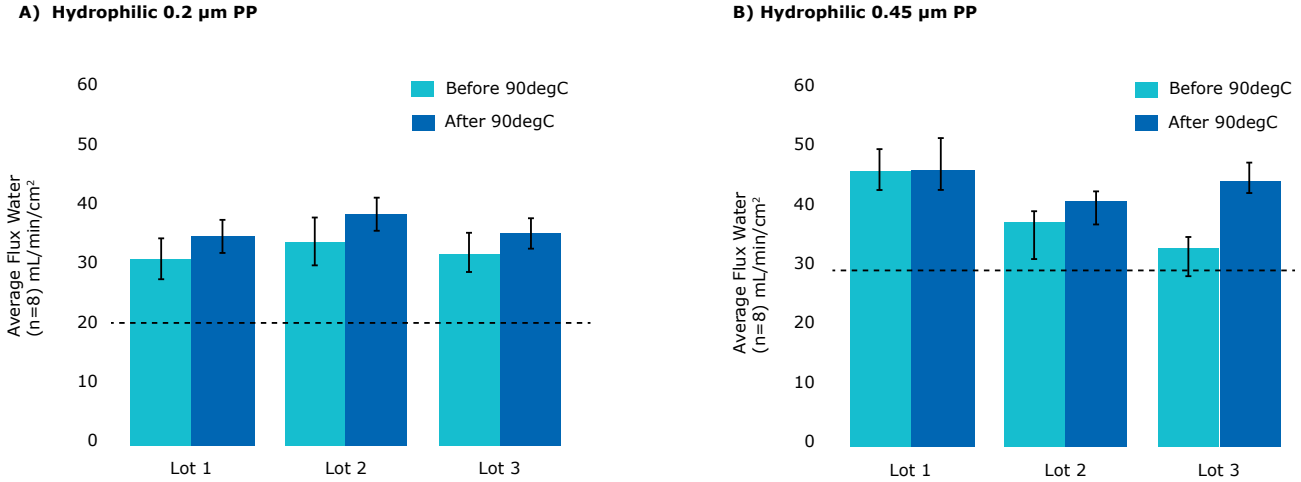
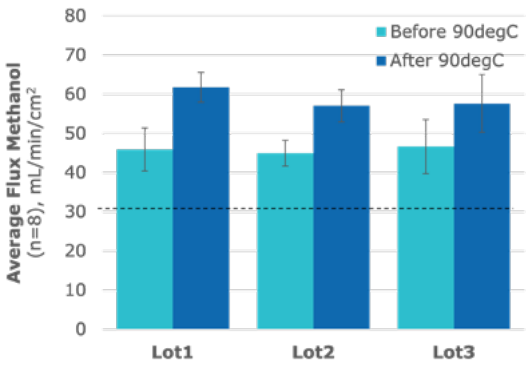


Figure 8: Comparison of the average water flow rate (mL/min/cm2) across 3 lots for (A) 0.2 µm and (B) 0.45 µm hydrophilic PP membranes before and after heating. Values represent mean ± standard deviation of n=8 membrane discs per lot.

A. Hydrophilic 0.2 µm PP



B. Hydrophilic 0.45 µm PP

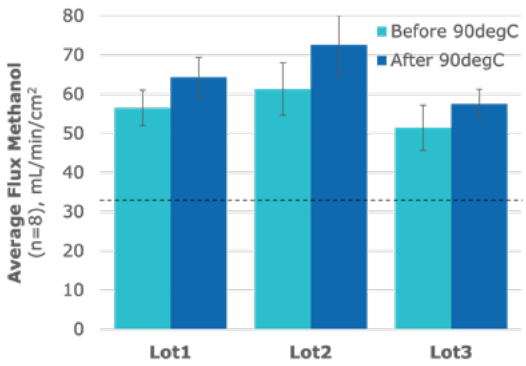


Figure 9: Comparison of the average methanol flow rate (mL/min/cm2) across 3 lots for (A) 0.2 µm hydrophobic polypropylene and (B) 0.45 µm hydrophobic polypropylene before and after heating. Values represent mean ± standard deviation of n=8 membrane discs per lot.

All hydrophilic PP membranes met and exceeded the flow rate specification of >20 mL/min/cm^2 (0.2 µm membranes) and >30 mL/min/cm^2 (0.45 µm membranes) for water flow rate. There was little variation in flow rate from before and after heating. Likewise, all hydrophobic PP membranes met and exceeded flow rate specification of >30 mL/min/cm^2 (0.2 µm membranes) and >32m L/min/cm^2 (0.45 µm membranes). Additionally, all PP membranes were easy to handle after heating. Thus, polypropylene membranes are a good choice for applications that require heat stability and functionality after exposure to heat.

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