A Simple Method for Evaluating Filtration Membranes

Establishing a Correlation Between the Apparent Filtration Area and the Effective Filtration Area

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Membrane filters can be evaluated for the amount of fluid that can be filtered per unit area of filter media (throughput) at the bench scale using disposable or small membrane discs in reusable filtration devices (test housings). These devices typically range in diameter from 25 mm to 90 mm, with 47 mm being the most commonly used. A laboratory often has more than one type of 47-mm disc housing for testing membrane filters. Even though two different test housings may accept the same diameter membranes and seal the membranes with the same size O-ring, they may produce significantly different throughput test results. The primary cause for variability in throughput is differences in the effective filtration area (EFA) of membranes evaluated in the test housings. Even though the O-ring seals may be the same size and therefore provide the same apparent filtration area (AFA), the EFAs of the different housings could range from

about 10 cm^2 to about 13.5 cm^2, depending on the type of support screen used. When the downstream side of a membrane contacts the solid surface of its support screen, fluid will not flow through that portion of the membrane.

Here I describe and document a simple approach for maximizing EFA to ensure it is equal to the AFA. Although the tests described here were conducted using CUNO membranes, this approach is generic for membrane filters and housings available from other suppliers.

TEST HOUSING

Although test housings of many sizes exist, the focus of this paper is on 47-mm disc housings. The principles described here are applicable to housings of other sizes as well. A typical 47-mm test housing comprises a housing base, support screen, and housing top. The support screen is placed in the housing base, and the membrane is placed on top of the support screen. The O-ring is placed on top of the membrane, and the top of the housing is secured to the bottom with either a seal nut or sealing bolts (Photo 1).

Both housings in Photo 1 accept a 47-mm diameter membrane. Both use the same size O-ring. When a membrane is installed in each housing and sealed with the O-ring, the housings exhibit the same AFA (Photo 2). However, the EFAs of membranes installed in these housings are not the same.

Examples of bench-scale test housings
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Because the EFA is the area of the filter through which fluid can actually flow, liquid filtered by a membrane in direct contact with a metal screen can flow only through the perforated area of the screen. Photo 3 shows a close-up of the perforated areas of the two screens used in this testing and also shown in Photo 1. The diameter of the perforated area of the Type 2 screen is about 4.1 cm, whereas the diameter of the perforated area of the Type 1 screen is about 3.5 cm. The 0.6-cm difference in the two diameters results in a ~37% difference in perforated area. Even though the AFA of the two housings shown in Photo 1 is the same (based on the sealing O-ring inside diameter), the cutaway membranes (Photos 4A and B) clearly show the difference in EFAs resulting from differences in the perforated areas of the two metal screens. The amount of perforated area of a support screen is the primary characteristic of the screen having the greatest effect on the throughput. This characteristic was evaluated in this study.
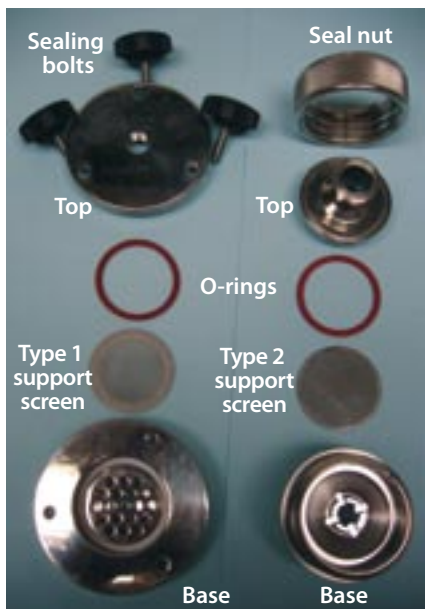
PRODUCT FOCUS: BIOPHARMACEUTICALS

PROCESS FOCUS: MEMBRANE FILTRATION

WHO SHOULD READ: PROCESS DEVELOPMENT AND MANUFACTURING

KEYWORDS: MEMBRANE FILTRATION, THROUGHPUT, HOUSING DESIGN

LEVEL: INTERMEDIATE

Photo 1: Examples of 47-mm housings

Photo 2: AFA comparison of different housings

Photo 3: Comparison of support screen perforated area

Photo 4: (A) Type 1 support screen and (B) Type 2 support screen

Photo 5: Type 1 support screen

Photo 6: Type 2 support screen

Other secondary characteristics of the support screen (such as the size, shape, and pattern of the holes) also can have an effect on the throughput. Fluid flow will take the path of least resistance. Screens with different secondary characteristics will create different flow paths through a membrane structure. The effect of those secondary characteristics on throughput is determined by how the internal flow dynamics of the fluid are affected as it passes through the internal structure of the membrane.
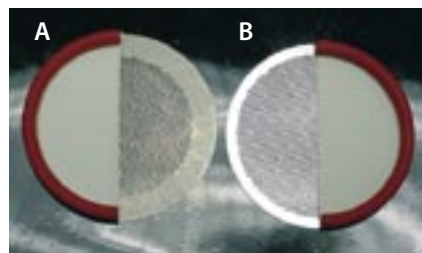
The support screens selected for this testing not only have different perforated areas, but also different size, shape, and hole patterns as can be seen in Photos 5 and 6. These secondary characteristics may account for some of the differences in throughput between screens at the different test pressures. However, the data suggest that the effect was small, and quantifying the effect of these secondary characteristics on throughput was outside the scope of this testing.
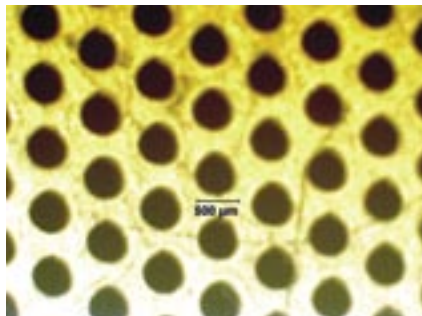
MATERIALS AND METHODS

Membrane Supports: Two types of 316-L stainless steel support screens were used in the testing. The perforations in the Type 1 support screen were uniform diameter perforations (Photo 5). The perforation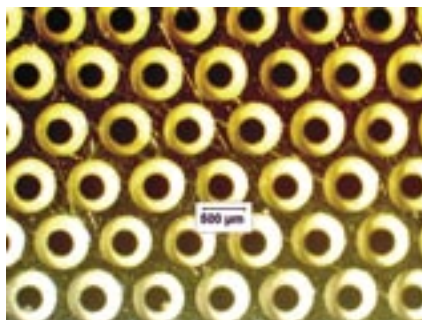s in the Type 2 support screen were through conical holes (Photo 6). The direction of flow was from the large diameter of the cone to its smaller diameter. The conical hole design maintains screen rigidity while maximizing open area for flow. The outside diameter (OD) of the perforated area in the Type 1 screen was well inside the sealing diameter of the O-ring used to seal the test membrane (Photo 4A). The outside diameter (OD) of the perforations in the Type 2 screen matched the sealing diameter of the O-ring used to seal the test membrane (Photo 4B). The Type 1 screen represents a low available filtration area screen, and the Type 2 screen represents one at or near maximum available filtration area.

Methods: Test methods for measuring the impact of using a mesh drainage layer (MDL) between the membrane test disc and the metal support screen were an abridged flow decay (AFD) test and a bubble point (BP) test. The AFD evaluated the impact of the MDL on throughput capacity (membrane life), and the BP test evaluated the MDL's impact on the ability to seal the membrane in the test housing.

In each AFD test, the test housing was connected to a pressure reservoir. An air pocket trapped at the surface of the membrane can prevent fluid from flowing through the portion of the membrane in contact with the air, which would lead to inaccurate results.

To permit bleeding the air from the upstream side of the test filter, a vent valve was installed on the upstream side of the test housing. To facilitate the venting process, shut-off valves were installed upstream and downstream of the test housing. The downstream shut-off valve was closed. The upstream shut-off valve and vent valve were opened. Sufficient pressure was applied to the pressure reservoir to fill the upstream side with test fluid. When a steady stream of fluid emerged from the vent valve, the vent valve was closed. The pressure reservoir was pressurized to the desired test pressure. The downstream valve was opened and a stopwatch started as soon as flow from the test housing was observed.

The cumulative throughput was measured and recorded every minute

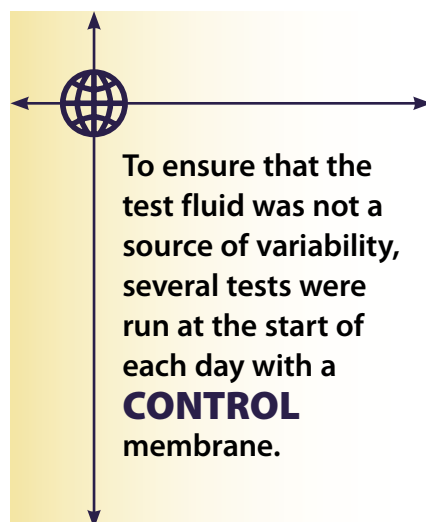| Table 1: AFD average throughput (liters/13.5 cm² at 90% plugging)										
Test Pressure (psid)	PSA020 no MDL Baseline		PSA020 Type A MDL		PSA020 Type B MDL		PSA020 Type C1 MDL		PSA020 Type C2 MDL	
	Type 1 Support	Type 2 Support	Type 1 Support	Type 2 Support	Type 1 Support	Type 2 Support	Type 1 Support	Type 2 Support	Type 1 Support	Type 2 Support
7.5	.212	.297	.267	.279	.153	.198	.252	.261	.243	.257
	—	—	(26%)	(-6%)	(−28%)	(−33%)	(19%)	(−12%)	(14.6%)	(−13.5%)
15	.216	.297	.284	.302	.189	.180	.279	.288	.266	.275
	—	—	(31.5%)	(1.7%)	(−12.5%)	(−39.4%)	(29%)	(−3.0%)	(23%)	(−7.4%)
20	.239	.297	.284	.320	.203	.203	.306	.297	.311	.284
	—	—	(18.8%)	(7.7%)	(−15.1%)	(−31.6%)	(28%)	(0%)	(30.1%)	(−4.4%)
Average Throughput	0.222	0.297	0.278	0.300	0.181	0.197	0.279	0.282	0.273	0.272

for 10 minutes. The data were used to calculate the throughput for the test disc to 90% plugging: defined as the point at which the flux through the disc dropped to 10% of its initial value.

In the BP test, the membrane was installed and sealed in the test housing. The housing was connected to a pressure reservoir. A small-ID flexible hose was attached to the outlet of the test housing, and the end of that hose was placed in a beaker.

To obtain accurate BP test results, a membrane must be thoroughly wet with water. To wet the membrane, the pressure reservoir was filled with 500 mL of 0.2-µm filtered deionized water, the reservoir was sealed, and 10 psi of air pressure was applied to the reservoir. After the water was passed through the membrane, air pressure was applied to the membrane in small increasing increments while bubbling from the flexible hose submersed in the beaker of water was observed. The pressure at which the bubbling from the outlet hose transitioned from an intermittent to a steady stream was recorded as the bubble point.

Data Analysis: The data from the AFD test were used to predict the throughput for the test disc to 90% plugging. Each throughput value represents the average of three separate tests. The data were used to

To ensure that the test fluid was not a source of variability, several tests were run at the start of each day with a CONTROL membrane.

determine whether the various MDLs affected the throughput volumes for each type of membrane–screen combination when compared with the throughput for those membrane–screen combinations with no MDL. The change in throughput volumes for screens using MDLs compared with the screens with no MDL was calculated on the basis of a percent increase or decrease. In addition, the effect of test pressure on throughput volume was determined.

Test Fluid: A solution of molasses at a concentration of 0.5 g/mL dissolved in water was used as the test fluid. Molasses is a complex carbohydrate, which when dissolved in water at a suitable concentration, will gradually

plug the pores of a microporous membrane. Batches of test solution could be consistently reproduced each day for testing.

Test Filters and MDLs: The membranes used were CUNO PSA020 (0.2 µm) sterilizing grade nylon 6,6 membranes. Three types of nonwoven polypropylene MDLs were tested: two spunbonded nonwoven materials, one 8-mil thick (10^{-3} inch = 1 mil) and labeled Type A, and the other, a calendered web, 4-mil thick, labeled Type B. We also tested a third type of nonwoven material 10-mil thick, made by an extrusion process and exhibiting a sidedness (one side smooth and the other ribbed). The 10-mil-thick material was labeled Type C1 when tested with the smooth side against the downstream side of the test membrane and labeled Type C2 when tested with the ribbed side against the downstream side of the membrane.

RESULTS AND DISCUSSION

Testing was conducted over several days, with a new batch of test fluid prepared each day. To ensure that the test fluid was not a source of variability, several tests were run at the start of each day with a control membrane. Control tests were run at the same pressure, with the same test screen and with no MDL. The test fluid was acceptable for use if the average throughput value was within 5% of the control throughput values for the first day of testing.

The AFD tests were conducted at three different test pressures: 7.5 psi (0.5 bar), 15 psi (1 bar), and 20 psi (1.4 bar). The testing was conducted

Table 2: PSA020 bubble point (psi)				
PSA020 No MDL	PSA020 Type A MDL	PSA020 Type B MDL	PSA020 Type C1 MDL	PSA020 Type C2 MDL
42.0	42.0	42.0	42.0	42.0
42.0	43.0	42.0	43.0	42.0
42.0	42.0	43.0	42.0	42.0

> **The use of a proper MDL will permit maximum use of the sealed membrane area in a test housing when performing FILTRATION tests and significantly reduce variations in throughput induced by different support screens.**

in triplicate with CUNO Zetapor PSA020 nylon 6,6 membranes. Within each set of triplicate tests, the variation between the throughputs (not shown) was 4–6%. However, tests conducted with the Type B MDL showed significantly greater variation within a set, as high as 65%. The cause of this variation and reduction in throughput is possibly the result of the calendaring process used to manufacture the Type B MDL. In the calendaring process of thermal bonding of nonwovens, fibers are formed into a web and then pressed between two rolls under pressure and at elevated temperatures. It is believed that the process flattens the fibers on both sides. The flattened fibers blind off both the holes in the metal screen and the pores on the downstream surface of the membrane.

Table 1 presents throughput values, in liters, to 90% plugging. The tests conducted with no MDL between the test membrane and the metal support screen are the baseline values used for comparison. The difference between the throughput value for each MDL and metal screen combination and the baseline value for the corresponding screen type and test pressure was used to calculate the percent change shown in parentheses.

The first tests established the baseline throughput values for each

type of screen without an MDL installed. On average, across the range of test pressures examined, the throughput for the Type 2 screen was about 34% greater than the throughput for the Type 1 screen. The greater throughput observed for the Type 2 screen correlates directly with the larger perforated area of the Type 2 screen.

On average, across the range of test pressures we evaluated, the Types A, C1, and C2 MDLs increased the throughput of the Type 1 screen by approximately 25% when compared with the baseline throughput values. By contrast, the Types A, C1, and C2 MDLs had little positive, or a slight negative effect on the throughput for the Type 2 screen when compared with the baseline throughput values.

The majority of throughput achieved with a membrane filter is achieved at a differential pressure less than 20 psid. For the tests conducted at 7.5 and 15 psid, the Type A MDL produced a greater average throughput for the Type 1 screen than either the Type C1 or C2 MDLs; and the Type A MDL had a significantly lower negative effect on throughput than either the Type C1 or C2 MDL.

In every test the Type B MDL reduced the throughput with both Type 1 and Type 2 support screens by a significant amount when compared with testing these screens without MDL. Determining the reason this occurred is beyond the scope of this study. However, the test results using the Type B MDL support the importance of proper MDL selection for membrane filter throughout the studies. This is especially true if an MDL is not provided with the membrane test discs.

BP tests were conducted to determine whether the increases in throughput observed in this study were the result of the membrane being improperly sealed when the MDL was used. Table 2 shows the results of those tests. The BP tests were conducted using the Type 1 support screen. As shown, the test results were within 1 psi of each other. Results indicated that the MDL had no

negative impact on sealing the membrane.

ACHIEVING MAXIMUM USE
The use of an improper MDL not only reduces membrane throughput, but can also cause large variations in the throughputs obtained with the same type of membrane and challenge solution. By contrast, the use of a proper MDL will
- permit maximum use of the sealed membrane area in a test housing when performing filtration tests
- permit calculating the EFA based on the sealing diameter of the O-ring used to seal the membrane.
- significantly reduce variations in throughput induced by different support screens
- not interfere with the proper sealing of membrane discs in the test housing.

REFERENCE
1 Meltzer TH, Green H. Flow and Pressure, Filter Sizing, and Filter System Design. *Advances in Parenteral Sciences 3: Filtration in the Pharmaceutical Industry.* Robinson JR, Ed. Marcel Dekker, Inc.: New York, NY, 1987; 421–422.

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