Advanced Thermoplastic Membranes for Water Filtration – Phase II

Final Report

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Acronyms and Abbreviations

Term	Definition
Alg	Algicide
ALW	Artificial Lake Water
ANSI	American National Standards Institute
cm	Centimeter
CTMA	Commercial Technologies for Maintenance Activities
DOD	Department of Defense
Em	Emission
Ex	Excitation
g	Grams
gal	Gallons
GVSC	Ground Vehicle Systems Center
hp	Horsepower
Hr	Hour
IN	Industrial Netting
Int	Intermass
L	Liter
MF	Microfiltration
mg	Milligram
min	Minutes
mL	Milliliter
NCMS	National Center for Manufacturing Sciences
NDSU	North Dakota State University
NSF	National Sanitation Foundation
nm	Nanometers
ODASD-MR	Office of the Deputy Assistant Secretary of Defense, Materiel Readiness
psi	Pounds per Square Inch
RFU	Relative Fluorescence Units
RO	Reverse Osmosis
SEM	Scanning Electron Microscopy
SGW	Simulant Ground Water
TARDEC	Tank Automotive Research, Development and Engineering Center
TMP	Transmembrane Pressure
UF	Ultrafiltration
U.S.	United States

1. Executive Summary

Water scarcity has become a recent trend in news worldwide and thus resulted in multiple research paths to treating water to make it potable for human consumption. From the minimal amount of potable water in the world, which is less than 1%, with the majority of that percentage in the glaciers that are rapidly melting into our oceans driving down the amount of fresh water on our planet. Besides the minimal amount of fresh water, manmade disasters, as well our rising world population, water scarcity will continue to grow in what geographical locations become water scarce. Water scarcity is also not dependent on the country's financial status since China and the United States (U.S.), the two biggest economies, are having more areas becoming water scarce because of more stress being applied to already overstressed water sources. To overcome these problems of water scarcity, efforts to treat dirty water and generations of potable water from water sources previously not considered is at the forefront of research in filtration. Potable water is not only a civilian concern, as generating potable water from different water sources across the globe is also a military issue as generating potable water will be needed when operating forward operating bases. The Department of Defense (DOD) has significant cost projections when operating globally and generating potable water from maintenance to expenses that occur naturally with water filtration. Generating a portable, low-pressure, and automated filtration system incorporating a thermoplastic spiral wound filter with a long lifespan will decrease costs when generating potable water from various water sources encountered across the globe. Besides forward operating bases, this system could also be utilized for civilian areas affected by natural disasters rendering people without drinkable water for days and sometimes weeks depending on the severity of the natural disaster.

Potable water is a civilian and military issue that will continue to evolve until different sources of water are utilized as well as sustainable water management is utilized globally. For most of the world's population who lack reliable sources of potable water a mobile, small scale filtration system would bring possibilities of producing potable water from the dirty water sources used for water daily. A simple low-pressure filtration system that is capable of transportation to treat ground and surface waters would allow people the opportunity to potable water that is currently unavailable to vast populations around the globe. This system will also reduce costs of producing potable water for forward operating bases that vary depending on the geographical location of the base. The filtration system will also need to be capable of filtering different water sources to accommodate various locations while also being low cost. To accommodate the low cost the filters will need to have long lifespans which will be a direct correlation to fouling time as well as colonization of bacteria present in ground and surface water. Upon generation of a low-cost filtration system designed to treat various types of ground and surface water an enhanced filter design will further reduce costs while directly increasing the efficiency of the filtration system. Finally, to minimize costs even further automation of this filtration system will be crucial to eliminate the need for operators to constantly run the filtration system.

To accomplish the generation of potable water from ground water and surface water an enhanced thermoplastic spiral wound filter will be designed for incorporation into the filtration system. The filtration system will be designed to be portable so that it can be utilized in various areas and can be deployed with ease for military and civilian usage. The filtration system will need to function with low pressure to minimize expenses as well as being operational in isolated areas that is common for natural disasters as well as military operations. To further reduce the costs of this filtration system a simplistic design will needed to be incorporated to accommodate isolated areas, as well as automation will be needed to eliminate the need of advanced operators to run the system. The membrane utilized in the spiral wound filter will need to be optimized for maximum amount of flux while also minimizing the pore size to restrict solids and dissolved substances from passing through. This membrane will also need to be resistant to bacterial colonization as this will directly decrease the flux of the membrane utilized and decrease the lifespan of this filter. Finally, the spiral wound filter will need to be optimized for cleaning by backwashing and cleaners to increase the lifespan of the filter reducing the costs of the filter. Producing a spiral wound filter with antifouling properties incorporated into a simplistic, automated filtration system will provide potable water to isolated areas around the globe as well as areas affected by natural disasters within the continental U.S. at a lower cost than most current filtration systems.

For membrane optimization PPG constructed different formulas to maximize water flux as well as bacterial resistance properties of the Microfiltration (MF) and Ultrafiltration (UF) membranes. Different amounts of silica were utilized to maximize the water flux of the membranes produced as well as different coatings to reduce biofouling from bacteria common in ground and surface waters. The water flux tests were conducted at PPG while the antifouling studies were conducted at North Dakota State University (NDSU) for the different membranes produced by PPG. These tests were conducted on bench scales with repeated trials to validate results amongst different membranes tested before construction into larger scale filters. For filter optimization multiple components of the spiral wound filter were analyzed for maximum water flux with geometry, size, and thickness varying amongst the feed spacers and permeate tubes studied during this phase. NDSU also conducted biofouling tests on the various feed spacers being studied for use in our filter design with one manufacturer clearly outperforming the others in bacterial colonization. For the construction of the filter design different membranes as well as varying leaf numbers of the spiral wound filters were studied as well. When designing the filter smaller scale filters were produced initially which started with 2514 filter series then from the trends discovered filter designs were scaled up to 2540 and finally 4040 filters which did not involve varying leaf counts like the first two filter sizes. Upon completion of the smaller scale filters the 4040 series groundwater testing was completed on four different designs to study the effects of fouling over 72 hours of the different designs utilized. For system optimization InnoH2O Solutions, LLC (InnoH2O) was subcontracted to offer a system that was a simplistic design that would be portable as well as an automation of the system. They redesigned the system that was utilized in Phase I and reworked plumbing and layout of the system to minimize space used and reduce pressure drops across the system. Upgrades were also completed on the pump utilized as well as rerouting of lines back directly to the tank to further reduce pressure drops and improve the overall efficiency of the system. A standard filter design was utilized to compare the different skids at PPG and Ground Vehicle Systems Center (GVSC) to ensure universal results with no clear design and data differences amongst the two systems fabricated. Cleaning procedures were studied in Phase I and will continue to be studied for the various sources of water being treated throughout the phases of this project. Lastly, operating conditions were studied to ensure maximum lifespans of the filter designs and to ensure the least energy intensive conditions to minimize the cost of this filtration system.

Funding for the collaborative effort was secured through the National Center for Manufacturing Sciences (NCMS) Commercial Technologies for Maintenance Activities (CTMA) Program and the Office of the Deputy Assistant Secretary of Defense, Materiel Readiness (ODASD-MR).

1.1 Results

For membrane, PPG conducted different membrane formulations to balance the water flux and antifouling properties. PPG selected three membranes, PPG standard UF membrane, PPG MF membrane and PPG newly developing antifouling membrane for the bacteria antifouling study. NDSU conducted the antifouling studying. A consistent, repeated trend of decreased *Pseudomonas fluorescens* (P. *fluorescens*) surface colonization of the UF membrane relative to the MF membrane was observed from each replicated experiment, ranging from 8%-25% with a mean reduction of 19% across the four independent assessments. In terms of membrane surface fouling, the antifouling 833-1915 membrane accumulated 23% less P. *fluorescens* than the UF PPMK087-08 membrane after 24 hours of recirculated feed culture exposure.

For feed spacer, NDSU conducted bacteria antifouling for nine types of feed spacers provided by PPG. They analyzed in conjunction with UF membrane PPMK087-08. Despite a higher degree of bacterial fouling accumulation overall, the general trend in terms of relative P. *fluorescens* colonization obtained accorded well with the comparative fouling trend. The 9x9 43 mil treated, algicide (Alg) treated 44 mil Intermass (Int), and non-Alg treated 44 mil Int variants were shown to be the most effective at mitigating microbial biofouling on the UF membrane when exposed to continuous recirculating flow of Artificial Lake Water (ALW) spiked with bacteria for 24 hours, achieving a 29%-43%, 24%-39%, and 10%-28% reduction, respectively, relative to the six other candidate feed spacer variants.

For the prototype optimization, PPG optimized the design start with 2514, 2540 and then scale up and validate with 4040 designs. When developing the 2514 filters a number of leaves and different permeate carriers were studied. Variations in permeate carrier were studied and a 24% increase in mean permeability with the change to a more porous 12 mil permeate carrier versus standard 12 mil carrier. Number of leaves in each filter was studied with a 71% increase by increasing membrane leaves from two to four, 26% increase from four to six, and a 34% decrease in mean permeability when increasing membrane leaves from six to eight. From the 2514 filter results the number of leaves was studied further as well as different membranes were studied for scale up. The number of leaves and the correlation to the specific permeability of the filters provided the same results observed from the 2514 filter design. When leaves were increased from two to four leaves a 48% increase was observed in mean specific permeability and a 1.85% increase was observed when leave count was increased from four to six in 2540 filter elements. Upon completion of the 2540 filter deigns four different filter designs were created with variations in feed spacers and membrane to analyze results with a larger filter. PPG fabricated four designs of prototype 4040 membrane elements, 101 (A, C, D, and G), 102 (D, E, G, and H), 103 (A, F, G, and H) and 104 (D, E, G, and H). InnoH2O evaluated groundwater filtration performance of the prototype filters. Eight runs were completed by installing two spiral wound membranes into the InnoH2O test skid at a time. InnoH2O constructed the test skid for delivery to GVSC utilizing an electric diaphragm pump to simplify design for backwashing procedure while minimizing the use of external air sources. The skid utilized also had upgrades from the previous phase which increased the potential for spiral wound membrane filters. All four filter designs were studied with the 21-MB-102 filter series performing the best overall amongst all other designs. The 102 series had a 9.2% increase in normalized flux and a 3.7% decrease in pressure drop compared to the 21-MB-101 series, the next closest filter design. The 102 series performed best in the groundwater study as it was 8.33% more resistant to initial fouling than the other filter designs with the 104 series the most resistant to fouling over 72 hours but had the lowest normalized flux of all designs.

1.2 Benefits

U.S. Army, Marine Corps, and Special Operations all need reliable, easy to use and cost-effective systems for producing potable water from groundwater. This project has accelerated the development of thermoplastic membrane technology for groundwater use.

Thermoplastic membrane technology can provide advantages for groundwater use due to the ability to modify porosity and incorporate inorganic fillers to deliver high flux rates. This can result in a high percentage influent stream recapture. Thermoplastic membranes also have shown the ability to operate longer without fouling, reducing the cost for replacements to municipalities, maintenance operations and facilities as well as reducing the waste stream. Thermoplastic membranes offer durability and can be cleaned by backwashing to restore flux, thereby providing long lifetimes and reliability in the field.

A laboratory scale crossflow testing methodology was developed at NDSU to quantify salt water (i.e., marine) bacteria fouling on flat membrane and feed spacer combinations provided by PPG.

Benefits to the General Public

A successful project will accelerate the development of a robust filtration system with significantly reduced operator complexity that is based upon thermoplastic membrane technology. The less complex the apparatus, the easier it will be to produce and maintain, which will result in lower operating costs. The thermoplastic membrane technology is applicable to defense, domestic, and global applications, typically for, but not limited to, seawater desalination. Other applications would include:

- Groundwater Remediation Filtration
- Process Water Filtration
- Public Utilities and Municipal Water Filtration
- Pulp and Paper Manufacturing Filtration

The units can be scaled and deployed to any operational situation requiring immediate water filtration, including natural disasters, drought conditions, or infrastructure failures. Cheaper and easier to deploy systems would allow more units available more quickly in time of need (disaster relief) which would translate directly to more people served or saved. Using such membrane element systems for municipal, maintenance, and industrial applications will enable more cost-effective water development and conservation worldwide. More cost-effective systems would result in more capacity and more jobs at water-treatment plants and/or more jobs at production facilities and their suppliers.

Benefits to the DOD

Water filtration has been a research area for the Tank Automotive Research, Development and Engineering Center (TARDEC) Force Projection Directorate for over 12 years. TARDEC has a test stand used to integrate new filtration technology. Over the years, many new technologies have been integrated into this system. The complexities of these systems have made them difficult to practicably implement. One issue has revolved around the flux. What that means is the usable flow of the water. In past technologies the flux was a major concern because not enough water was available for the needed usages. The other technical issue is of fouling. In past technologies dirt and other pollutants were not being filtered out for the water to be potable. Having water that can be used and consumed easily and safely is essential for the hardworking warfighters and is a critical component of the entire maintenance enterprise. This project will allow for the development of systems with ease of use in "ready to integrate" components for TARDEC system evaluation.

Additional benefits to the U.S. Army/DOD include:

- Reduced maintenance costs
- Larger volumes of potable water for deployed or remote troops
- Simpler, quicker, and easier to deploy and set up units or systems
- Longer lifecycle times
- Enhanced readiness due to reduced unit set up

1.3 Recommendations

Prototype Design

The best available feed spacer will be further evaluated in Phase III with variants from Int built into prototype filters utilizing the coated antifouling membrane as well as the standard UF membrane utilized in 21-MB-102 filter design. Available permeate carriers will also be further investigated to increase the normalized flux of our prototype filters. Membrane optimization will also be conducted with the antifouling membrane as it more selective and was most resistant to the fouling to the groundwater testing.

UF Skid Operation

Since the 4040 filters all resulted in lower than 80% recovery the prototype design as well as skid parameters will be investigated to increase the recovery of our 4040 filters. InnoH2O will be contacted for assistance with any other improvements that may be feasible to increase the recovery of our filters in the filtration system. Further development, focusing on both cartridge design and system design optimization, is required to achieve higher recovery rates with this technology.

Antifouling UF Membrane

Since the antifouling membrane showed the best results with overall fouling in the groundwater, which will increase the lifetime of filters utilizing this membrane, further investigation into the fouling mechanisms will need to be understood to maximize its potential. Also, the coated antifouling membrane utilized in the prototype 4040 filters resulted in the lowest normalized flux investigation into increasing the flux of this membrane will be imperative to increasing the potential for this membrane to be utilized in 4040 filters. Different silica loading will be utilized to increase the flux of this membrane which will aid in increasing the recovery of our prototype filter designs.

1.4 Technology Transition

Based on the results of the 4040-groundwater simulant testing conducted by InnoH2O, in conjunction with biofouling data from NDSU, PPG constructed six of each from designs 21-MB-102 and 21-MB-104, both utilizing 12-leaf construction with the prototype antifouling membrane used in the latter design. While this demonstrated a lower flux in testing 27.42 GPM after six hours of groundwater testing it fouled at a lower rate and may have potential for longer run cycles and better recovery of performance after cleaning.

1.5 Invention Disclosure

Invention Disclosure Report(s):

DD882 Sent to NCMS ☐
No Inventions (Negative Report) ☒

1.6 Project Partners

- PPG Industries, Inc.
- North Dakota State University (NDSU)
- InnoH2O
- National Center for Manufacturing Sciences (NCMS)

2. Introduction

2.1 Background

Reliable supplies of potable water may be the most pressing international issue facing the world today. Without clean water, communities are decimated by disease, farming and subsistence operations cease, children cannot go to school, and dehydration leads to systemic bodily shut down. The quality of life in those regions is extremely low. Even in areas of the U.S. the drinking water distribution system is antiquated and, in some places, contaminated. By 2025, almost every country will have some amount of water stress or shortage (from BCC Research Report: The Global Market for Membrane MF). An estimated 80% of the world's population lives in areas with threats to water security. Since only 1% of the world's water is available for human use, efforts to increase water availability must include both reuse and generation of new potable water sources. This is a problem for both native populations as well as U.S. agencies doing business in these regions. The economic and environmental impacts of polluted water reach far beyond quenching thirst but influence every aspect of productive life. By reliably producing clean water communities could grow economically, the populations would live longer, healthier lives, and pull themselves out of poverty. Investments could be made in areas with clean water that would bring jobs and new residents.

Globally, most people who lack clean water access live in remote, hard-to-reach places and use polluted lakes and rivers as water sources. These locations and sources could benefit from mobile, on-site purification technology over current Best Available Technology systems that have large footprints and require highly skilled operators.

In addition to current, ongoing clean water problems, there are frequent natural and man-made disasters for which global disaster relief efforts provide clean drinking water. In expeditionary missions, workers must either carry water with them or use technology to produce it in situ. Water is projected to be 30-40% of the daily sustainment requirement, and in situations of water scarcity, only a 6-8% water deficit (4-6 quarts) renders a person completely ineffective. Even maintenance, repair, and overhaul efforts, both domestic and expeditionary, require copious amounts of clean water. Whether the situation is a global, humanitarian problem or a DOD-specific mission, portable water treatment technology helps preserve life and decrease the logistical difficulties and costs of missions.

Reverse Osmosis (RO) is effective at cleaning water for potable use. Current potable use membrane filtration systems use an RO-based process, even when using low salinity fresh water sources, such as surface or ground waters. Existing RO processes are complicated entailing significant operating energy requirements, and the expensive and sensitive RO membranes require a pre-filtration process. Additionally, the hollow fiber membrane filtration systems used for pre-filtration of RO potable water production require complex process control valves, instrumentation, and programmable logic controllers that require significant training and maintenance expenditures to operate in the field, making the process cumbersome. Even with this complexity, hollow fiber membranes can still be sensitive and unreliable. The alternative, however, is also unsatisfactory. Single-use filtration cartridges are simple to maintain, but require labor to change, which adds to transportation and logistical costs.

The DOD in particular has significant force projection costs, depot maintenance, and operational expenses associated with potable water. That makes the DOD the ideal testbed for developing new water treatment technologies.

2.2 Purpose

A need exists for development of a low-pressure thermoplastic filtration membrane module specially designed to treat fresh water sources for safe and reliable potable use, without the need for high-pressure RO. The filter membrane, module, and system design would be optimized for high performance in the presence of foulants common to surface and ground waters.

In particular, the complexity of water filtration systems needs to be simplified through the design of an improved automated filtration system. This system would be simple to operate and maintain by taking advantage of current performance advancements in thermoplastic spiral wound membrane filter development.

The objective of this project is to develop an easy to use, robust filtration system. In support of the filtration system, the thermoplastic filtration membrane performance will be characterized for treatment of low-salinity fresh waters for potable use. The project will identify mechanisms of membrane fouling and optimize filter and system design for surface and ground water applications. As a goal of the project, an automated filtration system will be delivered to TARDEC for testing, validation, training, and field pilot trials.

2.3 Scope/Approach

A collaborative effort will be used to test and demonstrate an improved water filtration membrane module. The approach to be deployed embraces both government and industry participants. Industry will provide research, development, and testing. The government will provide functional and tacit knowledge, value opportunity definition and capture. The solution will begin with the system(s) deployed by the U.S. Army. Gaps in requirements versus system capabilities will be closed through modeling and testing.

This initiative will use a phased approach, managed in a way that delivers proof that the solution meets objectives and successfully demonstrates the ability to perform the functional requirements within the U.S. Army. Specific emphasis will be placed on the following:

- System Development (end-to-end design)
- Control Platform Development (s/w process control)
- System Testing/Evaluation
- Membrane Optimization
- Filter Performance Evaluation
- Prototype Delivery
- Fouling Assessment

2.4 Project Tasks and Deliverables

2.4.1 Project Tasks

Task 1.1: System Development

- Design an end-to-end filtration system based on thermoplastic spiral wound membrane filters, with a goal of reducing complexity, increasing ease of use, and allowing for robust automated operation.
- Design input to incorporate system modifications and operations tailored to related thermo-plastic subsystem work, and to be validated on a bench scale before system construction.
- Optimize automation hardware to consider operator usability and maintainability in the field.
- Design the system with shutdown sequences to allow for proper storage of the membrane elements in the unit for long periods of time.

Task 1.2: Control Platform Development

- Develop process control software for integration into the filtration system that will include online monitoring, analysis, and control of devices for the filtration system, enabling enhanced performance and reliability of the system with minimal operator input.
- Develop control platform that will monitor filtration performance indicators in real-time, and automatically adjust operating parameters to optimize system efficiency and prevent premature decline in performance.

Task 1.3: System Testing/Evaluation

- Deliver the system to TARDEC for evaluation.
- Train TARDEC in system use and demonstrate system performance.
- Support TARDEC during evaluation of the membrane-based filtration system.
- Filtration Membrane Development.

Task 2.1: Membrane Optimization

- Optimize filter membrane, module, and system design to treat fresh water sources.
- Components to be optimized for high performance in the presence of foulants common to surface and ground waters by evaluating flux rates, chemical resistance, reliability, and cleanability/maintainability.
- System design recommendations to be validated at bench scale.

Task 2.2: Filter Performance Evaluation

- Fabricate prototype filters and evaluate in lab-scale equipment.
- Conduct chemical analysis on the filtration samples before and after testing.
- Filtration performance of the prototype filters using fresh water sources will be characterized using an independent laboratory for items like bacteria reduction.

Task 2.4: Prototype Delivery

- The final prototype filtration element design will be delivered to TARDEC and evaluated for compliance with National Sanitation Foundation (NSF) 61 for potable water use.

Task 2.5: Fouling Assessment

- Determine fouling mechanisms and effects on membrane performance by commonly found organic compounds, causes of biofouling, and solids fouling by silts and clays.
- Determine mechanisms of membrane scaling by dissolved inorganic compounds such as iron, manganese, calcium, and magnesium.

2.4.2 Deliverables

Key deliverables that were provided include:

- Build and ship to TARDEC a membrane-based filtration system, including system use documentation, and filtration cartridge spares sufficient for six months of testing, based on the expected operation or service life, plus one additional set.
- Report on fouling mechanisms and filter design characteristics for treatment of low salinity fresh waters for potable use.
- Monthly Status Reports
- Quarterly Reports
- CTMA Final Report

3. Project Narrative

3.1 Filtration Membrane Development

3.1.1 Flat Sheet Membrane Optimization and Preparation

In this session, different flat sheet membranes were evaluated with filtration performance and fouling property. The small pilot trail was conducted. The membrane was scaled up for protocol cartridge development.

3.1.1.1 Flat Sheet Membrane Pilot Trial to Optimize Flux

A pilot trial was performed to mill and classify high surface area silica to produce a particle size distribution that is more amenable to producing a thin film. The middle oil absorption and high absorption with different oil absorption of silica were selected for experimental test. Different silica loading was tested in small pilot extrusion. Six prototype membranes (five experimental and one control) made in the PPG pilot laboratory were extracted. The detail is shown in Table 1. The membranes were tested with dead end flux at 50 pounds per square inch (psi). The results are shown in Figure 1. Samples 1670, 1671, 1674 all showed a significant improvement in flux over control membrane formulation. The results clearly indicate that the membrane flux increases with the increase of silica loading and silica high oil absorption properties. Therefore, the membrane with increased silica loading was selected for scale up as the next step.

Table 1. Membrane Flat Sheet with Different Silica Type and Silica Loading

Sample	Silica type	Silica loading	Polymer
833-1669	Standard	Standard	Standard
833-1670	Standard	High	UHMW-PE
833-1671	HOA	High	UHMW-PE
833-1672	MOA	Med-High	UHMW-PE
833-1673	MOA	High	UHMW-PE
833-1674	MOA/HOA blend	High	UHMW-PE

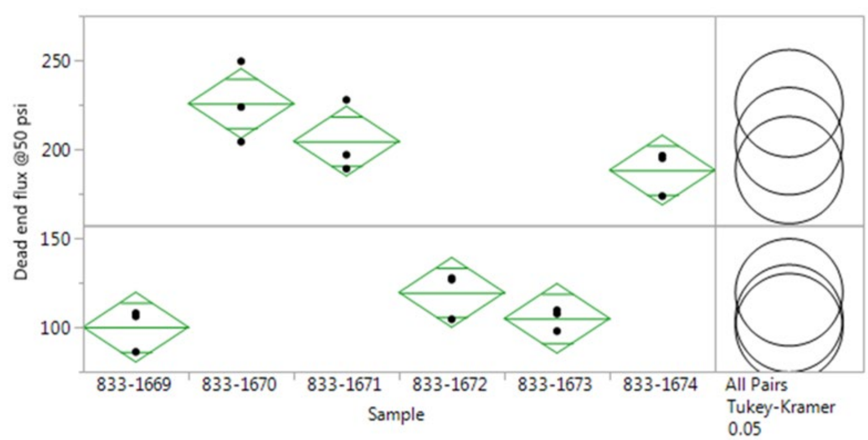


Figure 1. Dead-End Flux at 50 psi of Different Flat Sheets of Membrane

3.1.1.2 Different Membranes for Antifouling Study

Three types of membranes are investigated for the antifouling properties listed in Table 2. PPMK-087-08 is a commercially available UF membrane with standard silica loading and is utilized as a control membrane in experiments. 769-6412 is a commercially available MF membrane with standard silica loading, the membrane has higher water permeate due to large pore size distribution. The last membrane, a coated antifouling membrane 833-1915, is a developing new membrane with improve of silica loading, the membrane is also treated with PPG proprietary coating to improve surface fouling properties. The membrane is developed by PPG and will be used for the Phase III study.

Table 2. Properties of Three Different Membranes Being Utilized in Prototype Filter Construction

Membrane ID	Dead End Flux at 50 psi	Gurley (seconds)	Thickness (millimeters)
PPMK-087-08 (UF)	498.306 (g/min)	199.7	4.15
769-6412 (MF)	2438.26 (g/min)	35.1	7.3
833-1915	142.84 (g/min)	267.8	5.64

3.1.2 Microbial Biofouling Assessments

The primary objective for the microbial biofouling assessment task conducted at NDSU was to quantify freshwater bacteria fouling on flat membrane sheets and feed spacer materials provided by PPG to identify optimally performing combinations that effectively mitigate microbial biofouling. Data gleaned from these bench-scale experiments at NDSU aided PPG in determining which membrane and feed spacer technologies to integrate into spiral wound filter cartridges fabricated for advanced skid-based assessments of surface and ground water filtration effectiveness.

The rapid assessment of microbial biofouling on the surface of PPG membranes and feed spacer materials subjected to surface and ground water simulant feed sources was achieved utilizing a laboratory scale cross-flow testing methodology originally developed at NDSU during the first phase of this project to quantify salt water bacteria fouling on flat membrane sheets. An illustration of the testing setup is provided in Figure 2a, which comprised four cross-flow cells (CF042, Sterlitech) connected in parallel to a four-channel peristaltic pump to continuously deliver, in recirculating mode without permeation, simulated surface and ground water feed sources spiked with the freshwater bacterium, P. *fluorescens* (10^7 cells/mL), at a flow rate 10 mL/min. A hotplate stirrer was utilized to maintain a constant temperature of 28°C ± 1°C and mixing rate of 150 rpm within the glass feed culture vessel. After reaching the desired duration of feed culture exposure (24 hour), the flow was terminated and the membranes and feed spacers removed for quantification of bacteria surface colonization via conversion of the cell viability indicator dye resazurin to resorufin by attached cells; measured via fluorescence at Excitation/Emission (Ex/Em) = 550/583 nm. In select instances, prior to resazurin cell viability quantification, each cross-flow cell was detached from the peristaltic pump and reconnected to a pressurized tank of deionized water to measure permeate flux under 5 psi of supply pressure for 1 minute (Figure 3).

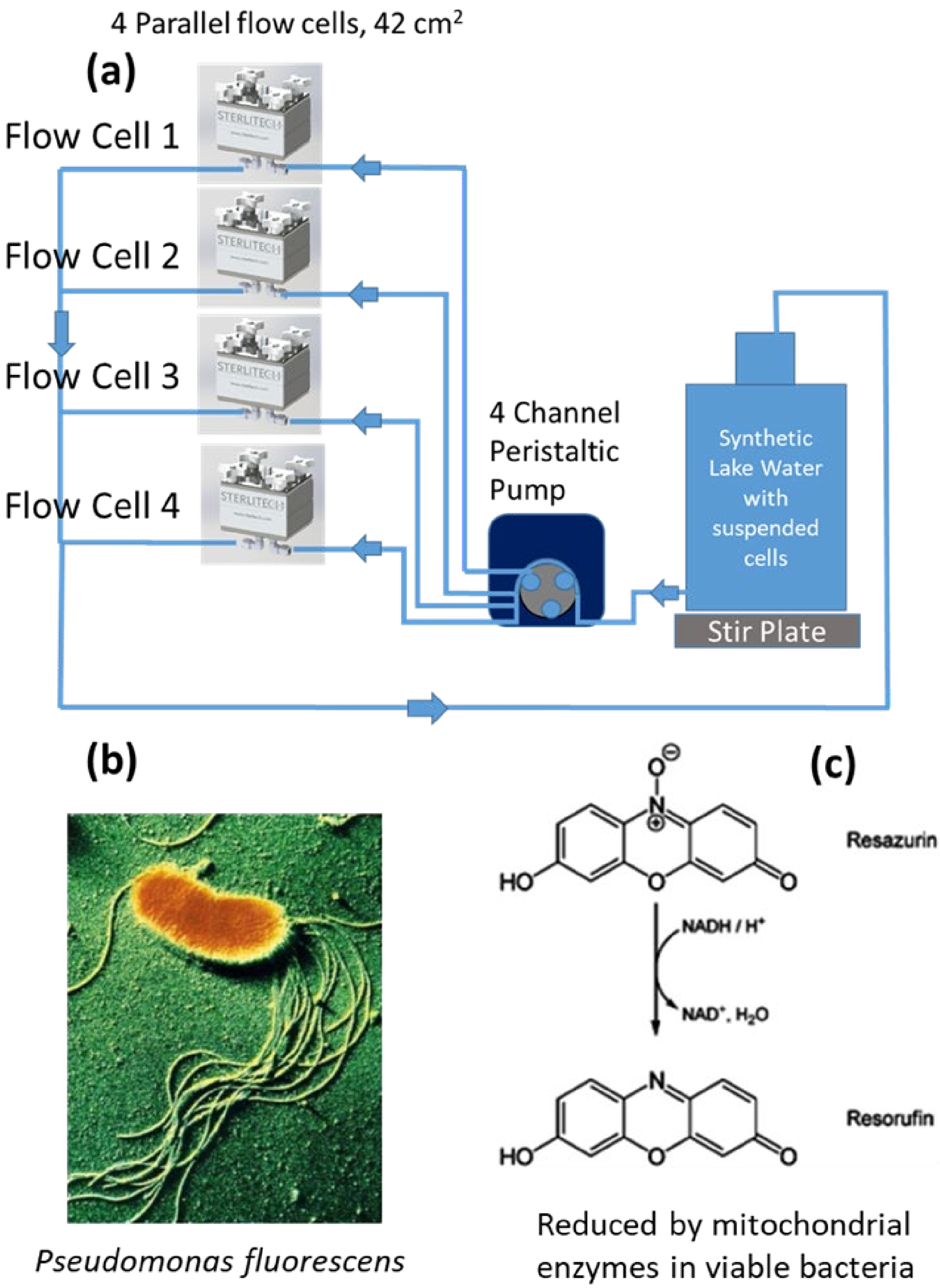


Figure 2. **Microbial Biofouling Assessments**
(a) Illustration of laboratory scale cross-flow testing setup at NDSU to characterize bacterial biofouling on flat membrane sheets and feed spacers. (b) Scanning Electron Microscopy (SEM) image of Gram-negative freshwater bacterium P.s fluorescens (https://web.mst.edu/~djwesten/Mo/BIO221_2009/P_fluorescens.html). (c) conversion of resazurin compound by viable bacteria to resorufin product quantified via fluorescence (Ex = 550 nm ; Em = 583 nm).

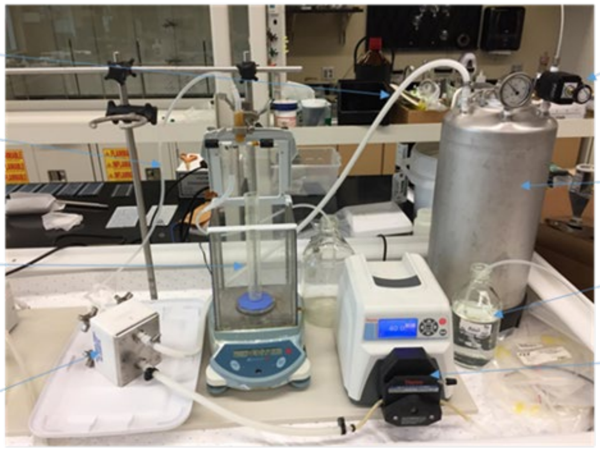


Figure 3. Laboratory Setup for Measurement of Permeate Flux At 5 psi of Supply Pressure for 1 Minute
Flux measurements were collected both pre- and post-exposure to bacteria feed culture.

At the outset of this phase of the project, membrane and feed spacer combinations were interrogated for imperviousness to fresh water microbial fouling when exposed to a simulant surface water feed source; specifically, an ALW formulation enumerated in Table 3. Three replicated bacterial fouling experiments with *P. fluorescens* were conducted for the UF PPMK087-08 membrane to quantify the intra-experiment and inter-experiment methodological variation under ALW filtration conditions. In this regard, the bacterial fouling variation among the four parallel flow cells (i.e., intra-experiment variance) ranged from 11%-39%, while the variation between the three replicated trials (i.e., inter-experiment variance) ranged from 17%-20%; as determined by deriving the mean resazurin fluorescence value from the four replicate flow cells for each experimental run (Figure 4). The ALW simulant repeatability results accorded well with methodological variation data observed previously for the artificial sea water-based method with *Halomonas marina* detailed in the Phase I final report, where the intra- and inter-experiment variance ranged from 7%-35% and 25%-46%, respectively. Consequently, the modified method employing ALW in tandem with the freshwater fouling bacterium *P. fluorescens* was deemed sufficiently rigorous and robust to evaluate membrane/feed spacer microbial fouling resistance properties for the Phase II effort.

Table 3. Artificial Lake Water Composition per 1 liter of Deionized Water

Constituent	Concentration (mg/L)
$CaCl_2$ (anhydrous)	15
$MgSO_4$ X $7H_2O$	15
$NaHCO_3$	20
Peptone	500
Yeast Extract	100

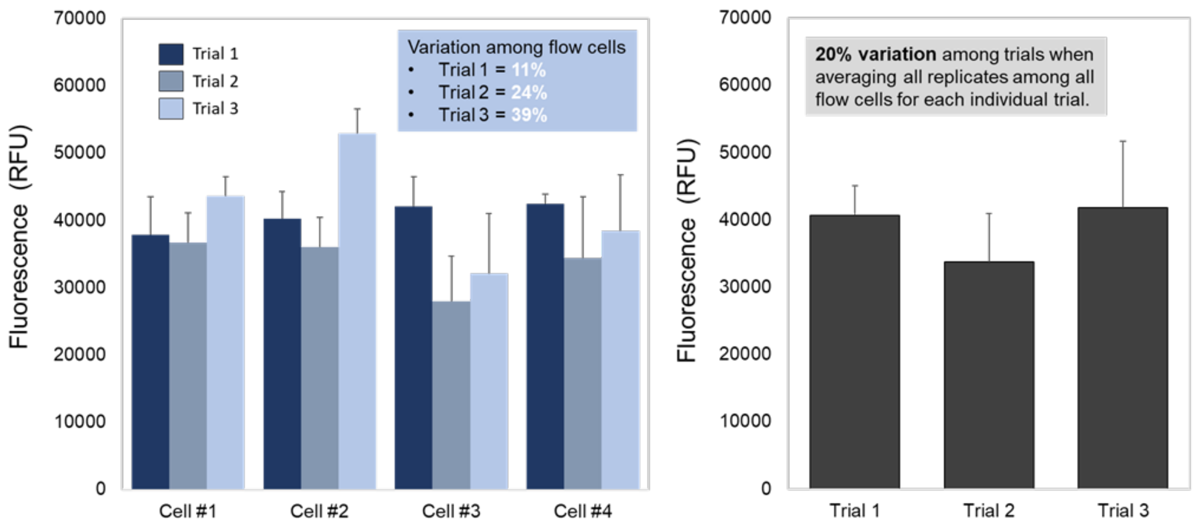


Figure 4. Crossflow Cell Variance of P. Fluorescens Colonization on UF PPMK087-08
Intra-experiment (left) and inter-experiment (right) crossflow cell variance of P. fluorescens colonization on UF PPMK087-08 membrane subjected to continuous flow of ALW simulant feed source at 10 mL/min for 24 hr. Each data point reports the mean relative fluorescence units (RFU) of resazurin (Ex/Em 550/583). Error bars represent one standard deviation of the mean.

A modification to the originally developed flow cell configuration illustrated in Figure 2a was required to promote or achieve comparable bacterial fouling of feed spacer materials among the four replicate flow cells. This system reconfiguration was prompted by the discovery of a consistent trend in substantively reduced *P. fluorescens* colonization of the surface of feed spacer materials mounted in Flow Cells 3 & 4 relative to the Flow Cells 1 & 2 (Figure 5b). Interestingly, this flow cell dependent trend was not observed for the UF PPMK087-08 membrane run in conjunction with the feed spacer over repeated, replicated trials (Figure 5a). The flow cell sequence was reversed to investigate potential flow cell positioning effects that may explain the discrepant results, as illustrated in Figure 5. The reverse ordering of the flow cells resulted in an inversion in the previously observed trend, where substantively more *P. fluorescens* colonized the feed spacer mounted in Flow Cells 3 & 4 relative to Flow Cells 1 & 2 (Figure 5b). The reversed order results insinuated an issue was occurring prior to the flow cells; specifically, the non-equivalent length of the feed culture tubing utilized to connect the peristaltic pump tubing to the inlet ports, where the length of feed culture tubing decreased considerably from Flow Cell 1 to Flow Cell 4. Consequently, the flow cell configuration was modified by incorporating equivalent length feed culture tubing sections between the inlet ports and peristaltic pump lines, which effectively eliminated bacterial fouling discrepancies for the feed spacer material among the four replicate flow cells as illustrated by comparing *P. fluorescens* colonization in Flow Cells 2 and 4 among the original (i.e., non-equivalent length feed culture tubing), reversed order, and modified configurations (i.e., equivalent length feed culture tubing) (Figure 5).

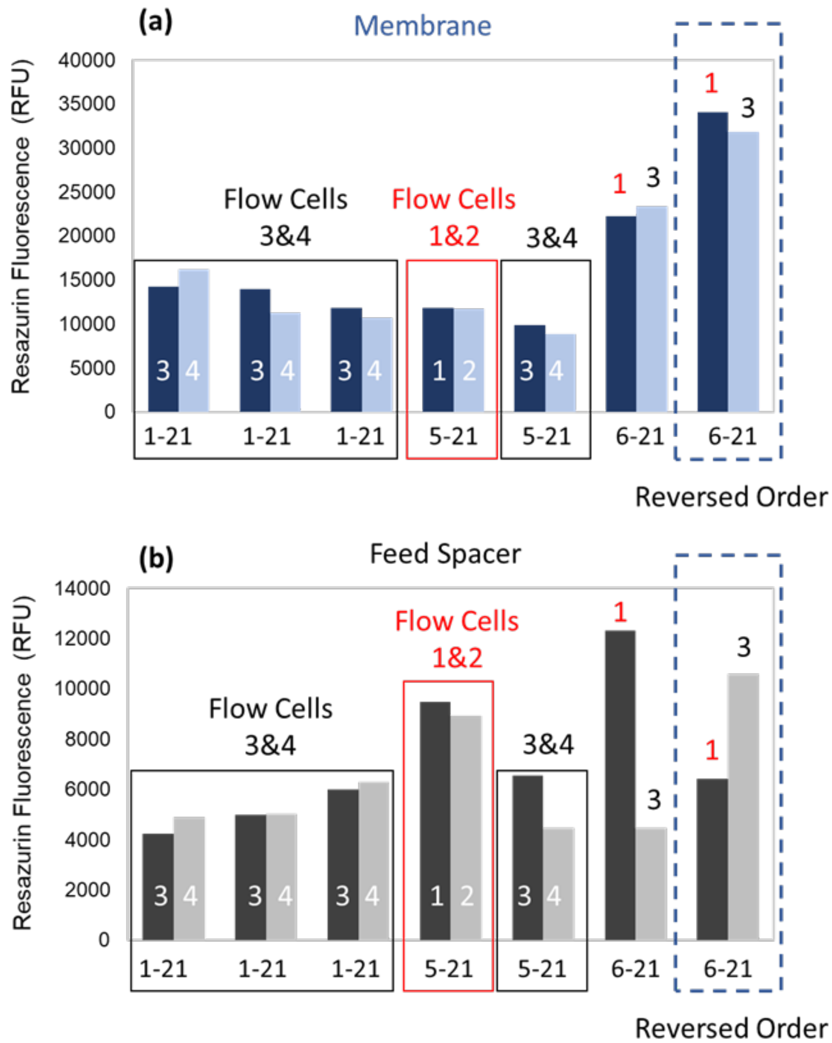


Figure 5. Investigation of the Effect of Flow Cell Position on (a) Membrane and (b) Feed Spacer Bacteria Fouling
Reversed Order data points indicate bacterial fouling on membrane and feed spacer mounted in the Reversed Flow Cell Configuration illustrated in Figure 6. X-axis labeling below each data point designates the month and year of biofouling assessment (e.g., 1-21 = January 2021).

The first biofouling experiment for this phase of the project constituted a comparative assessment of bacteria accumulation on UF membrane PPMK087-08 and MF membrane 769-6412 analyzed in conjunction with a 43-mil diamond feed spacer. Figure 6 displays the results of *P. fluorescens* fouling of the membrane surfaces for four replicated trials. A consistent, repeated trend of decreased *P. fluorescens* surface colonization of the UF membrane relative to the MF membrane was observed for each replicated experiment, ranging from 8%-25% with a mean reduction of 19% across the four independent assessments. This comparative bacteria surface colonization trend was likewise manifest and consistent for the 43-mil diamond feed spacer material (Figure 8). In fact, the reductions in *P. fluorescens* fouling of the feed spacer run in tandem with the UF membrane were considerably more pronounced than observed for the membrane itself, ranging from 30%-46% with a mean reduction of 38% across the four replicated experiments. As a consequence of its enhanced biofouling mitigation properties, the UF PPMK087 was selected as the standard membrane for employment in all subsequent cross-flow cell experiments for this phase of the project aimed at the exploration of a variety of different feed spacer materials for potential use as an integral component of spiral wound filter cartridges.

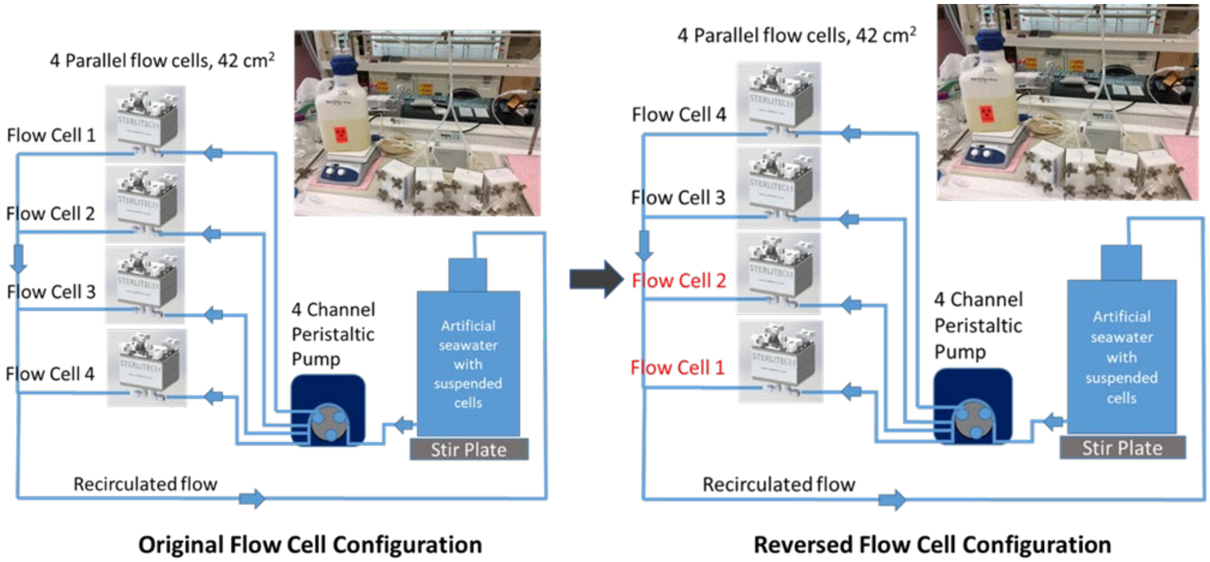


Figure 6. Illustration of Laboratory Scale Crossflow Testing Setup at NDSU to Characterize Bacterial Biofouling on Flat Membrane Sheets and Feed Spacers
Original flow cell configuration constituting decreasing feed culture tubing lengths from Flow Cell 1 to Flow Cell 4 (left). Reversed flow cell configuration constituting increasing feed culture tubing length from Flow Cell 1 to Flow Cell 4 (right).

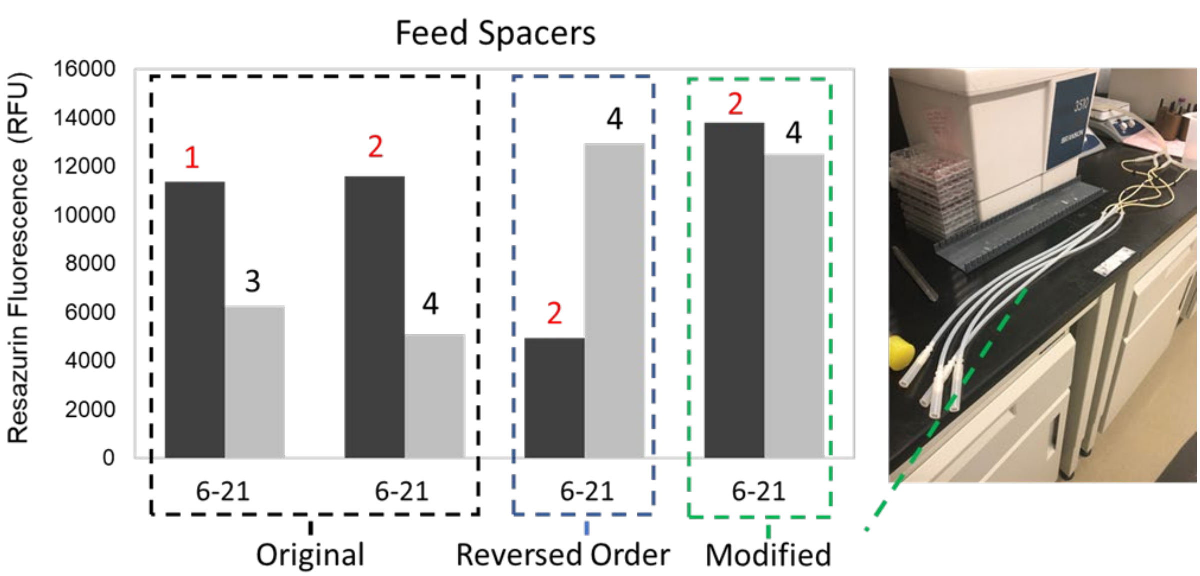


Figure 7 Bacteria Surface Fouling of Feed Spacer Material as a Function of Flow Cell Position/Sequence Using the Original, Reversed Order, and Modified Configurations
Original – non-equivalent length feed culture tubing, left. Modified – equivalent length feed culture tubing, right. X-axis labeling below each data point designates the month and year of biofouling assessment (e.g., 6-21 = June 2021).

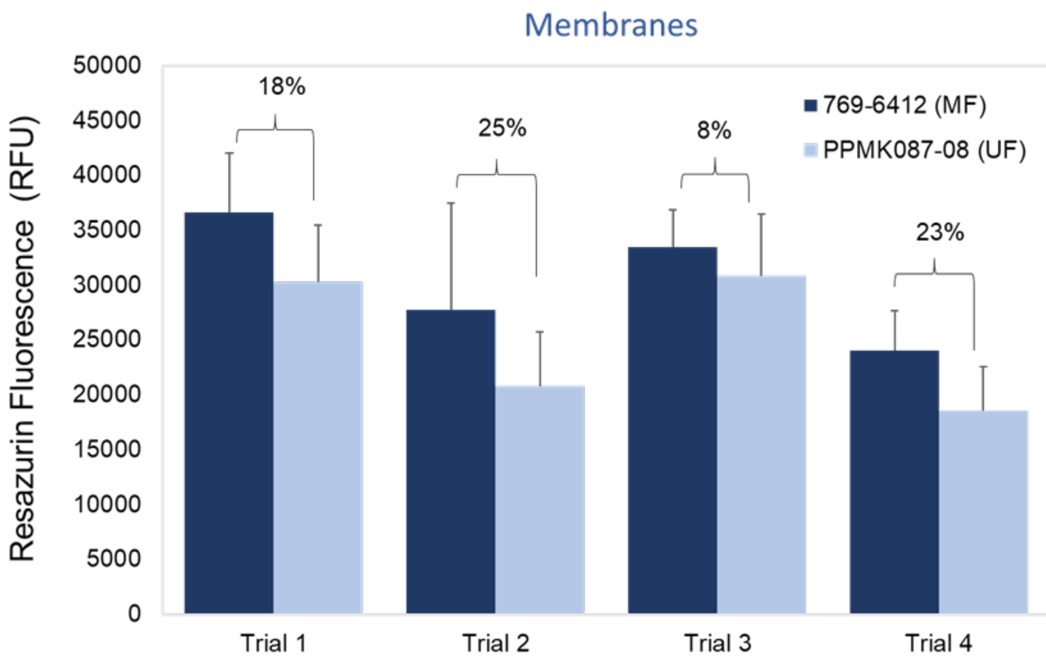


Figure 8. P. Fluorescens Biofouling Accumulation on MF Membrane 769-6412 and UF Membrane PPMK087-08 Analyzed in Conjunction with a 43-mil Diamond Feed Spacer Material, as Quantified by Resazurin Fluorescence *(RFU; Relative Fluorescence Units)*
Each data point is the mean RFU of 12 replicate measurements (4 sections x 3 measurements per section). Error bars represent one standard deviation of the mean RFU.

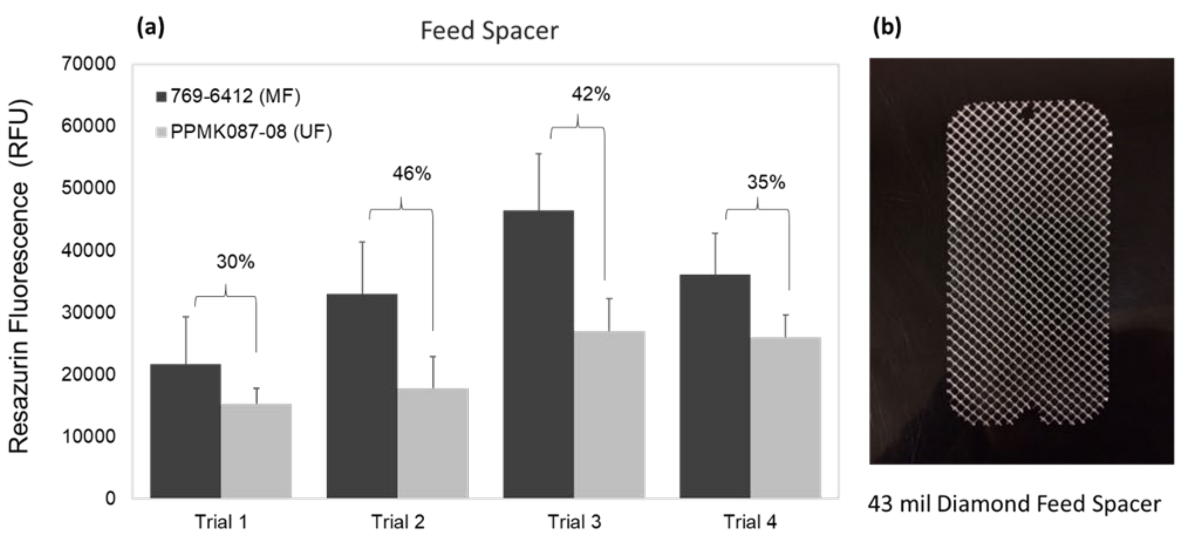


Figure 9. P. Fluorescens Biofouling Accumulation on the 43-mil Diamond Feed Spacer Material Analyzed in Conjunction with MF Membrane 769-6412 and UF Membrane PPMK087-08, as Quantified by Resazurin Fluorescence *(RFU; Relative Fluorescence Units)*
Each data point is the mean RFU of 12 replicate measurements (4 sections x 3 measurements per section). Error bars represent one standard deviation of the mean RFU.

Bacterial biofouling assessments of nine different feed spacer variants submitted to NDSU by PPG were conducted during Phase II, which resulted in the setup and execution of 43 individual flow-cell experiments. Figure 10 and Table 4 summarize *P. fluorescens* colonization of the PPMK087-08 UF membrane analyzed in conjunction with each feed spacer material. The 9x9 43 mil treated, Alg treated 44 mil Int, and non-Alg treated 44 mil Int variants were shown to be the most effective at mitigating microbial biofouling on the UF membrane when exposed to continuous recirculating flow of ALW spiked with bacteria for 24 hours, achieving a 29%-43%, 24%-39%, and 10%-28% reduction, respectively, relative to the six other candidate feed spacer variants. In contrast, the highest degree of *P. fluorescens* membrane colonization was observed for the 48 mil Int variant, which accumulated 38%-76% more bacteria fouling as compared to the three top performing feed spacer materials. Bacteria fouling of the UF membrane run in conjunction with the remaining five feed spacer variants, namely, 7x7, 9x9 46 mil, 9x9 43 mil, 11x11 and 11x11 Industrial Netting (IN), was remarkably consistent, falling within a relatively narrow distribution range of 18,387-21,311 mean RFU, equating to a modest 14% variance.

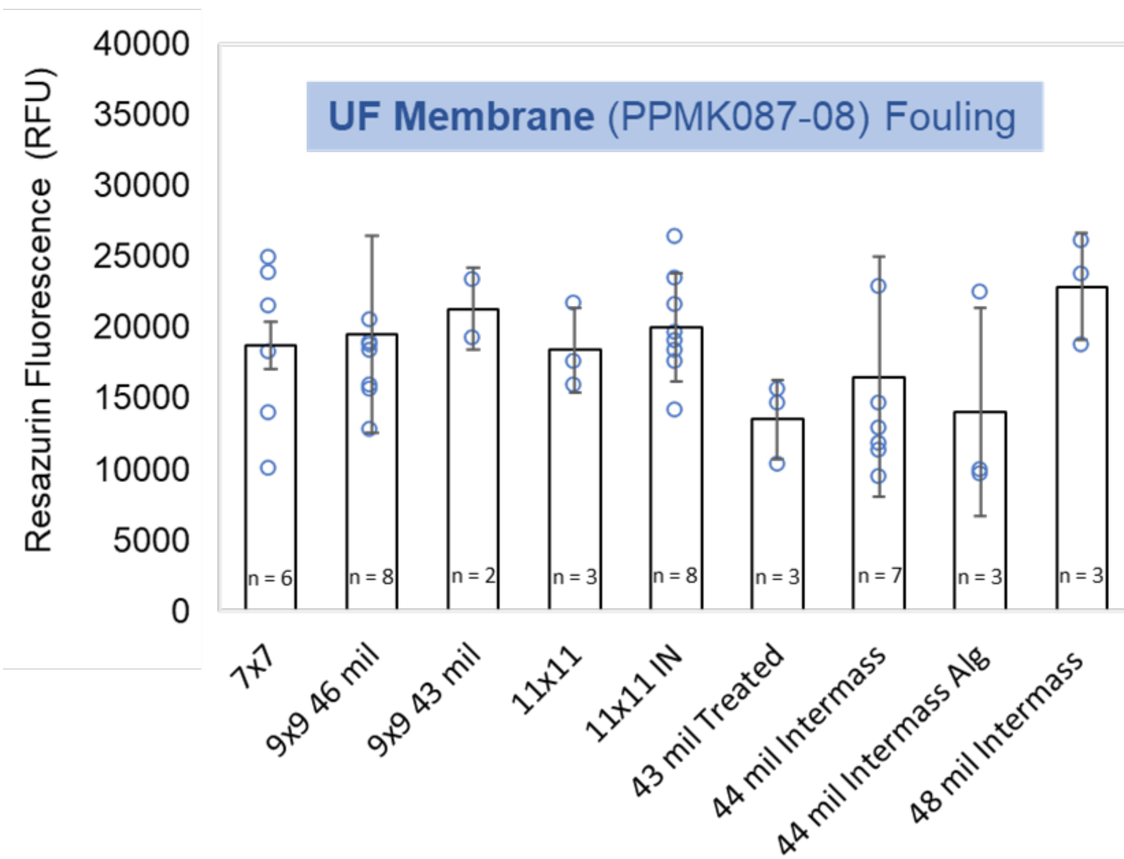


Figure 10. ***P. Fluorescens Biofouling Accumulation on UF Membrane PPMK087-08 Analyzed in Conjunction with Nine Different Feed Spacer Materials, as Quantified by Resazurin Fluorescence*** *(RFU; Relative Fluorescence Units)*
Each data point is the pooled mean RFU for all replicated experiments (n = 2 to 8) conducted for each feed spacer variant. Error bars represent one standard deviation of the pooled means.

Table 4. Mean Resazurin Fluorescence Values (RFU) for P. *Fluorescens* Surface Colonization of UF Membrane PPMK087-08 Analyzed in Conjunction with Nine Feed Spacer Variants

Feed Spacer	Mean RFU (Membrane)
7x7	18,754 ± 1,681 (n = 6)
9x9 46 mil	19,558 ± 6,945 (n = 8)
9x9 43 mil	21,311 ± 2,873 (n = 2)
11x11	18,387 ± 2,971 (n = 3)
11x11 IN	20,007 ± 3,782 (n = 8)
43 mil Treated	13,004 ± 3,608 (n = 3)
44 mil Intermass	16,525 ± 8,449 (n = 7)
44 mil Intermass Algicide	14,011 ± 7,345 (n = 3)
48 mil Intermass	22,886 ± 3,742 (n = 3)

The corresponding *P. fluorescens* feed spacer fouling data to the UF membrane fouling reported in Figure 10 and Table 4 is summarized in Figure 11 and Table 5. An immediate and conspicuous difference in the degree or magnitude of bacterial fouling on the feed spacers relative to the UF membrane is immediately evident upon a cursory examination of the two data sets. In this regard, the mean bacterial colonization for the nine feed spacer materials ranged from 6,718-15,913 RFU as opposed to 13,004-22,886 RFU measured for the accompanying UF membrane. This reduced level of *P. fluorescens* colonization on the surface of the feed spacer variants was most likely ascribed to the positioning of the UF membrane in the cross-flow cells, which is located above the feed spacer and in direct contact with the recirculated ALW. In this configuration, the UF membrane would be expected to be more prone to initial bacteria attachment and subsequent colonization than the underlying feed spacer. Furthermore, the UF membrane is considerably less porous than the suite of feed spacer materials examined, thus providing more surface area for bacteria fouling to occur. In the context of comparative bacteria fouling among the feed spacer materials themselves, the non-Alg treated 44 mil Int variant (i.e., 44 mil Int) evinced itself to be the least prone to *P. fluorescens* colonization over the 24-hour exposure period, accumulating 23%-58% less fouling than its eight counterpart candidates. The two other Int variants (i.e., 44 mil Int Alg and 48 mil Int) also exhibited reduced bacterial colonization compared to the 7x7, 11x11, 11x11 IN and 43 mil treated feed spacers, by approximately 30% on average, and accumulated comparable fouling relative to the 9x9 46 mil and 9x9 43 mil variants.

A modification to the originally developed flow cell configuration was required to achieve comparable bacterial fouling of feed spacer materials among the four replicate flow cells. This feed spacer fouling inconsistency issue was discovered after several of the variants had been evaluated. Consequently, in consultation with PPG, four feed spacers that were characterized using the original configuration (i.e., non-equivalent length feed culture tubing) were selected for repeat assessments utilizing the new/modified configuration (i.e., equivalent length feed culture tubing; Figure 6) to verify the previously observed trends in comparative performance. In this regard, Figure 10 displays the results of the *P. fluorescens* colonization of the four feed spacer variants, namely, 11x11 IN, 9x9 43 mil, 44 mil Int and 44 mil Int Alg, for both the original (i.e., cyan dots) and modified (i.e., dark bars) flow cell configurations. Despite a higher degree of bacterial fouling accumulation overall, the general trend in terms of relative *P. fluorescens* colonization obtained for the modified configuration accorded well with the comparative fouling trend observed for the original configuration (11x11 IN

>9x9 43 mil >44 mil Int Alg >44 mil Int), thereby validating the results of the original series of assessments and affirming the merit of corresponding conclusions made as to the selection of superior/optimal UF membrane-feed spacer combinations to mitigate microbial fouling in spiral wound cartridges.

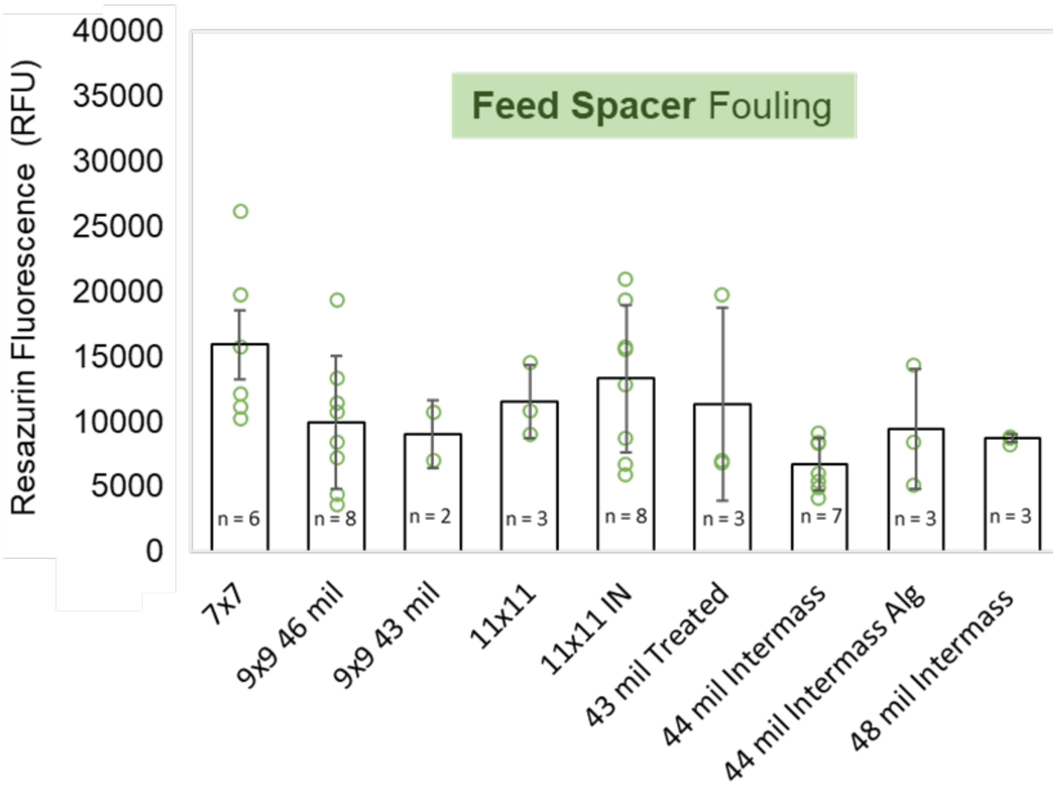


Figure 11. P. Fluorescens Biofouling Accumulation on Nine Feed Spacer Materials Analyzed in Conjunction with UF Membrane PPMK087-08, as Quantified by Resazurin Fluorescence *(RFU; Relative Fluorescence Units)*
Each data point is the pooled mean RFU for all replicated experiments (n = 2 to 8) conducted for each feed spacer variant. Error bars represent one standard deviation of the pooled means.

Table 5. Mean Resazurin Fluorescence Values (RFU) for P. *Fluorescens* Surface Colonization of Nine Feed Spacer Variants Analyzed in Conjunction with UF Membrane PPMK087-08

Feed Spacer	Mean RFU (Feed Spacer)
7x7	15,913 ± 2,648 (n = 6)
9x9 46 mil	9,910 ± 5,083 (n = 8)
9x9 43 mil	9,009 ± 2,590 (n = 2)
11x11	11,519 ± 2,788 (n = 3)
11x11 IN	13,920 ± 5,647 (n = 8)
43 mil Treated	11,531 ± 7,140 (n = 3)
44 mil Intermass	6,718 ± 1,957 (n = 7)
44 mil Intermass Algicide	9,412 ± 4,619 (n = 3)
48 mil Intermass	8,695 ± 318 (n = 3)

Predicated upon the results of the UF PPMK087-08 membrane-feed spacer microbial fouling experiments delineated above Figure 12, the Int 44 mil Alg treated and non-treated variants were selected for subsequent flux testing using the pressurized water flow apparatus illustrated in Figure 2. There was no practical difference in ALW flux measured before and after 24-hour exposure to ALW inoculated with *P. fluorescens*, with pre-bacteria exposure/post-bacteria exposure flux rates of 10.9/10.7 g/min (2% reduction) and 11.2/10.6 (5% reduction) for in 44 mil and Int 44 mil Alg, respectively (Figure 13). The small reductions in flux observed post bacteria exposure for both UF membrane-feed spacer combinations indicate that the degree of bacteria fouling accumulation on their respective surface did not substantively impair membrane permeability.

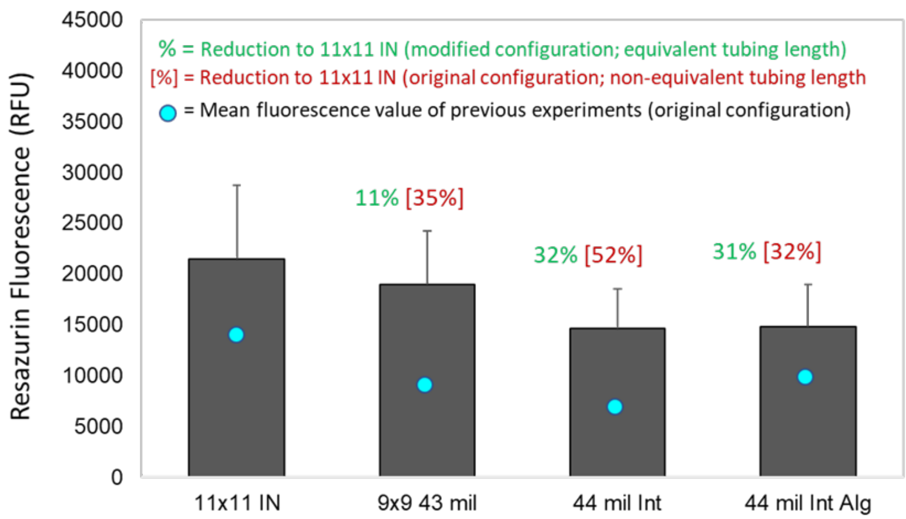


Figure 12. P. Fluorescens Biofouling of 11x11 IN, 9x9 43 mil, 44 mil Int and 44 mil Int Agl Feed Spacers Assessed with the Original Flow-Cell Configuration *(i.e., non-equivalent length feed culture tubing; cyan dots)* ***and Modified Configuration*** *(i.e., equivalent length feed culture tubing; dark bars)*
Red and green percent values indicate bacterial colonization reductions relative to 11x11 IN for the original and modified configurations, respectively.

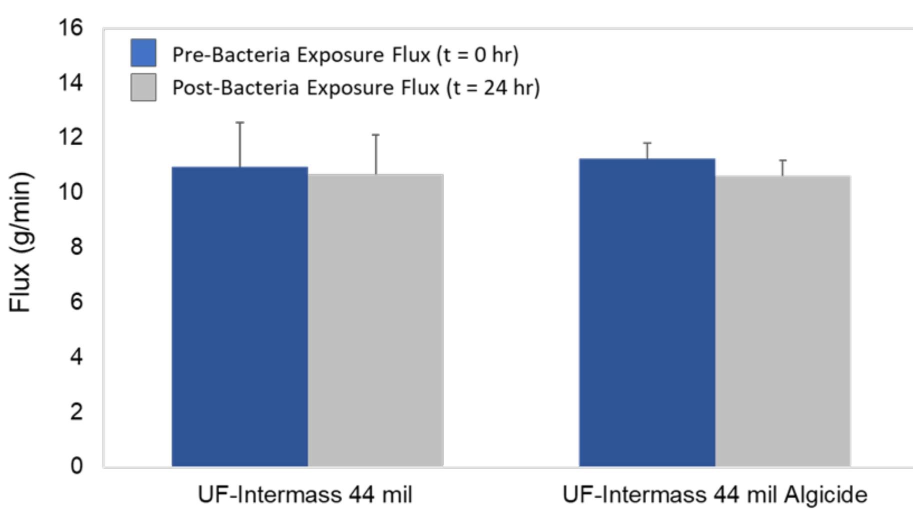


Figure 13. Pre- and Post-Bacteria Exposure Flux Measurements (g/min) for the Non-Alg Treated and Alg Treated 44 mil Int Feed Spacer Materials Analyzed in Tandem with UF Membrane PPMK087-08 *(5 psi of supply pressure for 1 minute)*

Figure 14 summarizes the *P. fluorescens* fouling assessments of an antifouling membrane, 833-1915 (5 mil), in conjunction with the 44 mil Int non-Alg treated feed spacer variant that exhibited the best bacterial colonization mitigation properties in the UF PPMK087-08 membrane biofouling trials. In terms of membrane surface fouling, the antifouling 833-1915 membrane accumulated 23% less *P. fluorescens* than the UF PPMK087-08 membrane after 24 hours of recirculated feed culture exposure. However, an equivalent amount of bacteria fouling still accumulated on the 44 mil Int feed spacer, indicating that the reduction of membrane fouling measured for 833-1915 did not translate into a detectable reduction of bacteria colonization of the underling feed spacer.

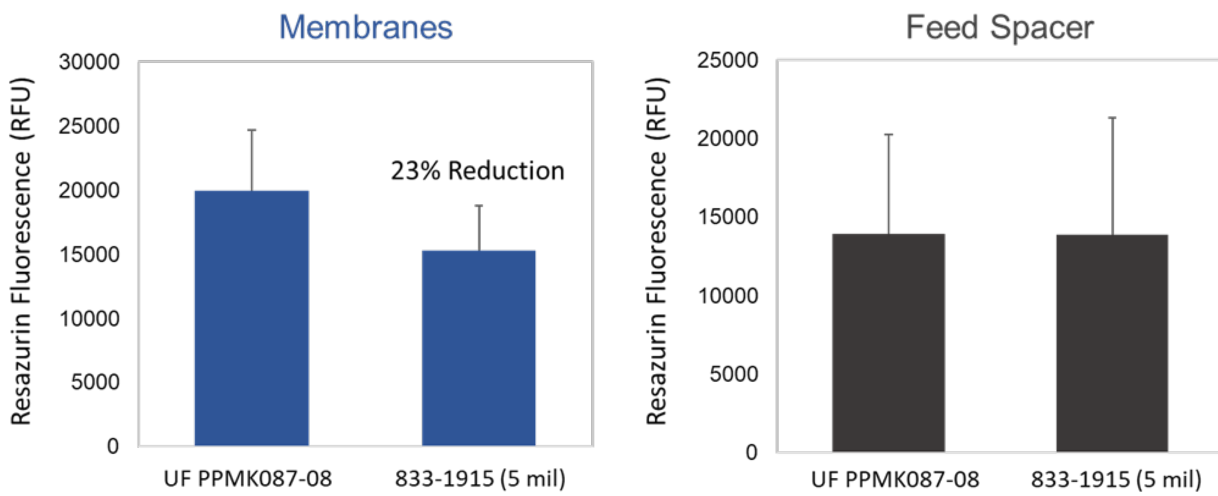
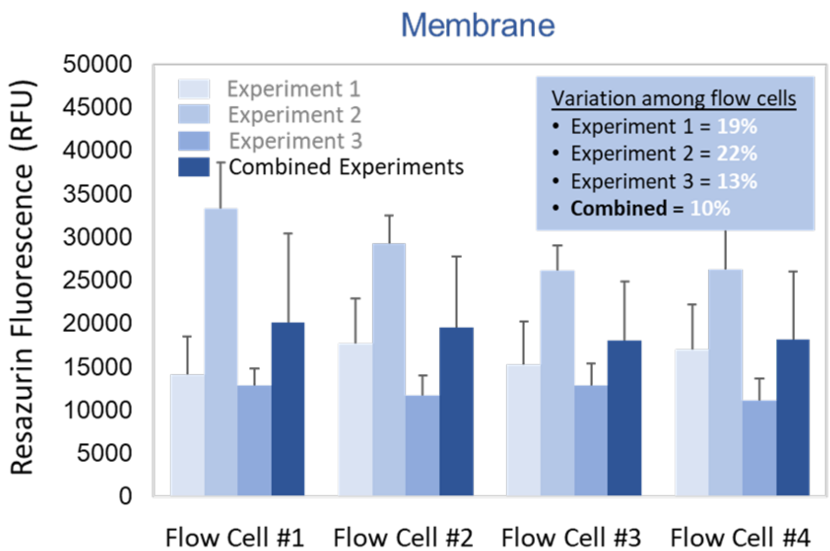


***Figure 14. P. Fluorescens Biofouling Accumulation on UF Membrane PPMK087-08 and Antifouling Membrane 833-1915 (5 mil) Analyzed in Conjunction with the Non-Alg Treated Int 44 mil Feed Spacer, as Quantified by Resazurin Fluorescence** (RFU; Relative Fluoresce Units)*
Each data point is the mean RFU of two replicated trials (8 sections x 3 measurements per section). Error bars represent one standard deviation of the mean RFU.

A secondary task for the crossflow biofouling assessment effort was to characterize the bacterial fouling of the optimized membrane/feed spacer combination identified from the experiments conducted with ALW with synthetic ground water. Table 6 summarizes the constituent content and concentration for a simulant ground water (SGW) formulation recommended by PPG that was employed for skid studies with spiral wound cartridges. Repeatability studies were initially conducted with the UF PPMK087-08 membrane in conjunction with 11x11 diamond feed spacer to determine the intra- and inter-experiment variance of *P. fluorescens* fouling in SGW as a function of three replicated experiments (Figure 13). With respect to bacterial colonization of the UF membrane surface, the intra-experiment variation ranged from 13%-22% while the inter-experiment variation ranged from 9%-61%. The variance for the pooled mean of the three replicated experiments was 10%. For the 11x11 feed spacer variant, the intra-experiment variation ranged from 8%-48% while the inter-experiment variation ranged from 13%-47%, while the variance for the pooled mean of the three replicated experiments was 19%. The results of this study highlight the imperative of executing multiple, repeated experiments for each membrane-feed spacer combination to account for inter-experiment variance attributed to changing/inconsistent ambient laboratory conditions and potential batch-to-batch bacteria culture variations.

Table 6. Synthetic Ground Water Composition Per Liter of Deionized Water

Constituent	Concentration (mg/L)
CaCl2	55.5
MgSO4	42
CaCO3	200
KNO3	5.1
Na2SiO3	37.8
FeSO4	12.8
NaCl	46.4
ISO 121030-1 Test Dust, Fine	30
Humic Acid	0.5 mL/L



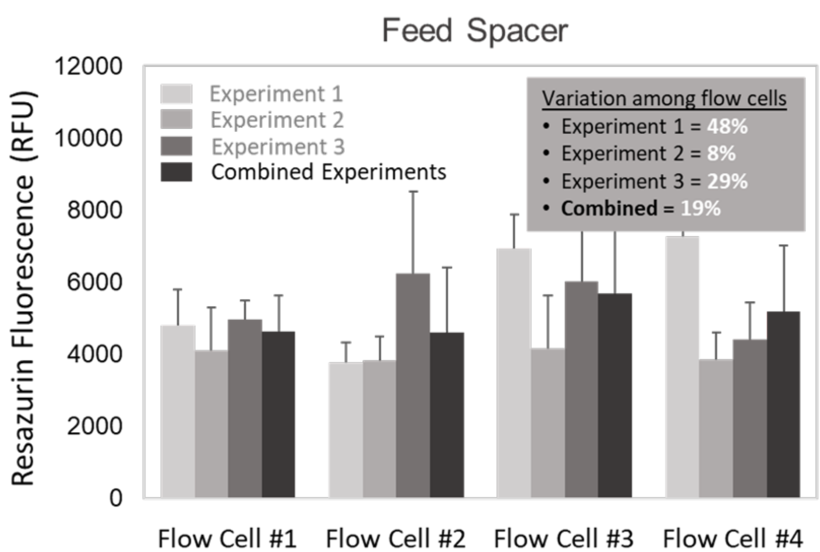


Figure 15. Cross-Flow Cell Repeatability Experiments of P. Fluorescens Colonization on UF PPMK087-08 Membrane and 11x11 Diamond Feed Spacer Subjected to Continuous Flow of ALW Simulant Feed Source for 24 Hours

Each data point reports the mean RFU of resazurin. Error bars represent one standard deviation of the mean.

Due to insufficient funds remaining at the conclusion of the SGW repeatability study, the final set of microbial biofouling experiments for the optimal membrane/feed spacer combination (i.e., UF PPMK087-08 membrane + 44 mil Int non-treated) under SGW filtration conditions will be completed in the initial period of the pending/forthcoming Phase III effort for this project and the results will be included in the corresponding final report.

3.2 Prototype Filter Performance Evaluation

3.2.1 2514 Prototype Filter Element Design Optimization

3.2.1.1 2514 Cartridge Max Permeability by Membrane Leaf Count

When testing elements in parallel, combined concentrate from two filters was controlled to 7 GPM for 24-hour test period Standard UF membrane, 31 mil diamond feed spacer, and epoxy adhesive used for all filters. All clean water performance test data completed with PPG pilot laboratory skid as shown in Table 7 using deionized water with 250 ppm NaCl and 250 ppm PPG MZD7360J preservative. When testing single element, concentrate from one filter was controlled to 3.5 GPM for 24-hour test period.

Table 7. 24-Hour Clean Water Test Data of 2514 Filters

Sample ID	Membrane Leaves	Active Area (ft^2)	Flow (gph)	Flux (gfd)	TMP (PSI)	Specific Permeability (gfd/PSI)
18-WKR-117A	8	6.6.	18.2	66	10.86	6.1
18-WKR-117B	8	6.6	18.7	68	10.87	6.3
18-WKR-116E	6	6.7	31.2	111.4	9.76	11.4
18-WKR-116F	6	6.7	32.8	116.9	9.76	12
18-WKR-116A	6	6.7	23.1	82.2	10.48	7.8
18-WKR-116B	6	6.7	20.9	74.6	10.75	6.9
18-WKR-115D	4	6.8	24	84.3	10.54	8
18-WKR-115F	4	6.8	26.3	92.3	10.59	8.7
18-WKR-115B	4	6.8	20.2	70.8	10.74	6.6
18-WKR-115C	4	6.8	20.9	73.3	10.76	6.8
18-WKR-100 B	2	6.9	13.8	47.6	10.47	4.5
18-WKR-100 C	2	6.9	13.08	45.1	10.435	4.3

The 24-hour test results of filter elements ran in parallel indicated a 71% increase in mean permeability by increasing membrane leaves from two to four in 2514 filters. With respect to the permeability of the four leave filters an increase of 26% in mean permeability by increasing membrane leaves from four to six. Last, with respect to the permeability of the six leave filters a 34% decrease in mean permeability when increasing membrane leaves from six to eight was observed in 2514 filters.

Prior to testing elements in parallel for a 24-hour test with a combined concentrate from two filters was controlled to 7 GPM a step pressure test was conducted increasing inlet pressure from 5.0 psi to 30.0 psi. Upon completion of the step test max permeability of 2514 filters was measured. The result indicates that 71% increase in mean permeability by adding membrane leaves in 2514 filter from two to four, 26% increase in mean permeability from four to six leaves, and no improvement when increasing the number of leaves from six to eight (Figure 16).

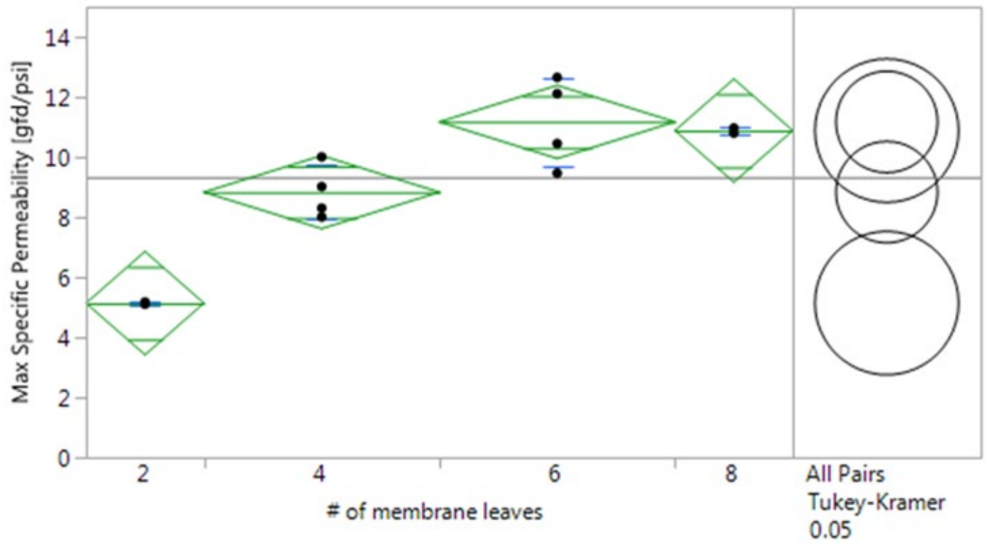


Figure 16. Max Permeability Water Flux vs. Number of Membrane Leaf

3.2.1.2 2514 Cartridge Max Permeability by Permeate Carrier

Test results indicated that holding all other design factors constant, 24% increase in mean permeability with change to more porous 12 mil permeate carrier versus standard 12 mil carrier. Additional 26% increase (56% increase versus control), using 20 mil knit carrier (Figure 17). Repeated trials are needed to validate results. Only one filter of each alternate carrier design passed integrity testing. The 20 mil carrier design was difficult to assemble. Increased thickness of carrier reduces active membrane available within a given nominal size.

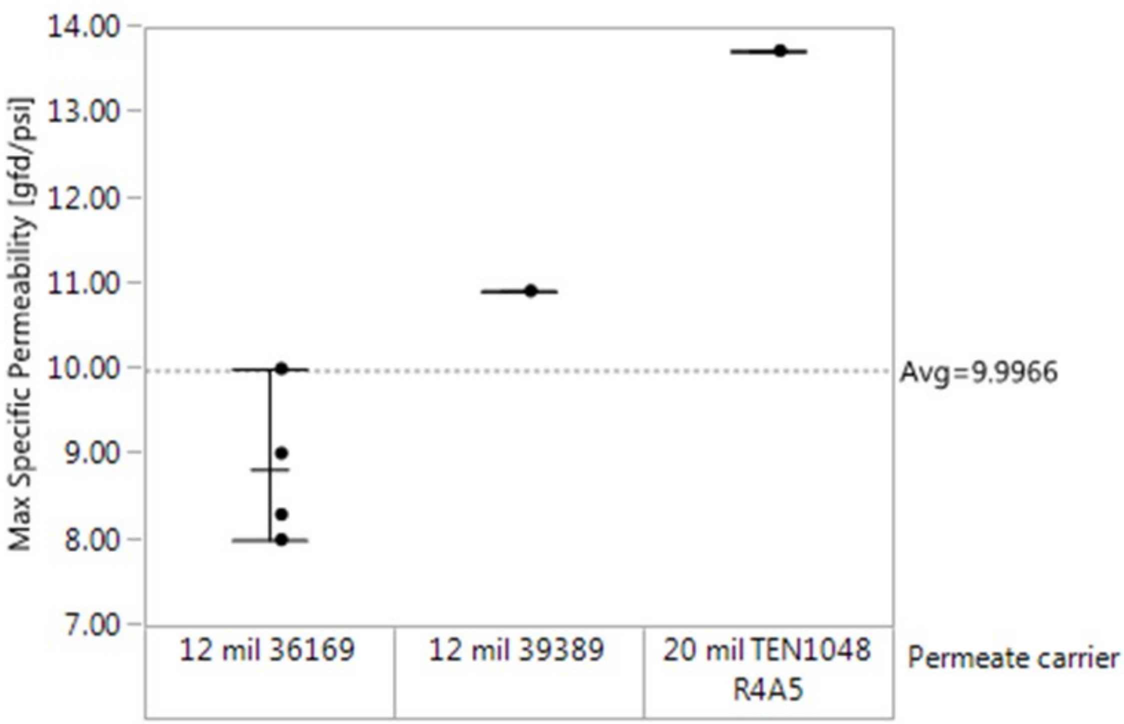


Figure 17. Maximum Specific Permeability of Different Permeate Carriers

3.2.2 2540 Prototype Filter Element Design Optimization

3.2.2.1 2540 Prototype with PPG Standard UF Membrane Test

Standard UF membrane, 43 mil diamond feed spacer, and polyurethane adhesive was used for all 2540 prototype filters. All clean water performance test data completed with PPG pilot laboratory (Figure 18) using deionized water with 250 ppm NaCl and 500 ppm PPG MZD7360J preservative. Elements were tested in parallel pairs, combined concentrate from two filters was controlled to 7 GPM for 24-hour test period. Concentrate crossflow rate for each filter was controlled to 3.5 GPM for 24-hour test period. Elements were tested in parallel pairs. Permeability (specific flux) data was normalized at 20°C using the following correlation of viscosity with temperature:

$$J\_20 = J\_T \cdot e\hat{}([-0.032 \cdot (T - 20)])$$

Figure 18. Lab Test Skid at PPG

3.2.2.2 2540 Cartridges with Standard Membrane Test Result: Oneway ANOVA of 2540 Max Permeability by Design

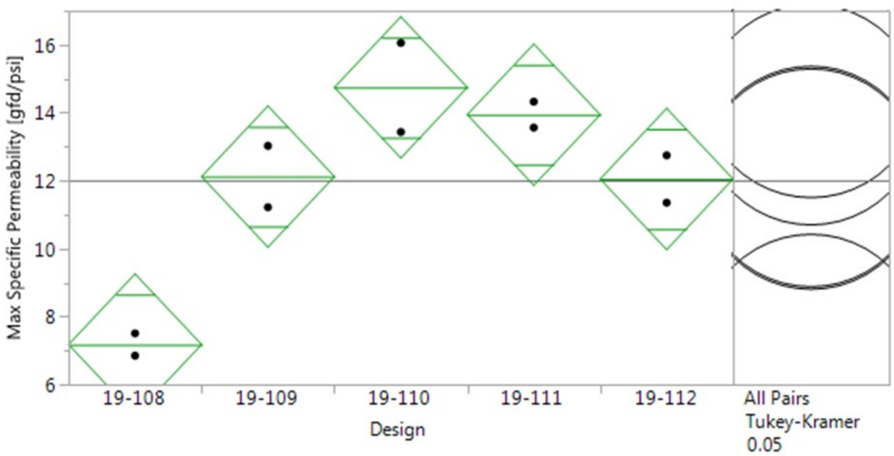
Three different filter designs incorporating varying numbers of leaves to better understand the effects of increasing leaves as well as the active membrane area of filters evidenced in the smaller 2514 filter designs. Maximum specific permeability was analyzed of the three different filter designs, with the only difference being the number of leaves in each filter hence altering the active membrane area as seen in Table 8.

Table 8. Summaries of the 2540 Cartridge 24-hour Test

Sample ID	Membrane leaves	Active Area (ft^2)	Flow (gph)	Flux (gfd)	TMP (PSI)	Specific Permeability (gfd/PSI)
19-WKR-108	2	23.5	60.30	61.6	9.125	6.8
19-WKR-109	4	23.0	81.78	85.3	6.890	12.4
19-WKR-110	6	22.5	85.44	91.1	6.215	14.8
19-WKR-111	4	23.0	84.46	88.1	6.310	14.0
19-WKR-112	6	22.5	79.14	84.4	7.000	12.1

The number of membrane leaves was evaluated in 2540 filter elements to determine how permeability of different leave counts was affected in 2540 as was observed in the 2514 filter elements. Cartridges were tested with clean water, the 24-hour performance test data completed with PPG pilot laboratory skid using deionized water with 250 ppm NaCl and 500 ppm PPG MZD7360J preservative. Standard UF membrane, 43 mil diamond feed spacer, and polyurethane adhesive used for all filters. Elements were tested in parallel pairs, combined concentrate from two filters was controlled to 7 GPM for 24-hour test period. The 24-hour tests results indicated that a 48% increase was observed in mean specific permeability when leaves were increased from two to four leaves in 2540 filter elements. A 1.85% increase was observed when leave count was increased from four to six, which is statistically not significant since an increase in leave count increases chances of error in production.

Prior to the start of the 24-hour test a step pressure test was conducted increasing the inlet pressure from 5.0 psi to 30.0 psi, the max specific permeability was taken immediately after the completion of the step test. The test result indicated that 69% increase in max specific permeability by increasing membrane leaf count in 2540 filter from two to four (Figure 19). This is consistent with previous result of 71% increase in max specific permeability when increasing membrane leaves in 2514 filters from two to four. No statistically significant difference when increasing number of leaves from four to six, or by changing permeate carrier geometry from 12 mil 36169 style to the more porous 12 mil 39389 style.



Connecting Letters Report

Level			Mean
19-110	A		14.745892
19-111	A		13.945503
19-109	A		12.123231
19-112	A		12.051575
19-108		B	7.179839

Levels not connected by same letter are significantly different.

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
19-108	2	7.1798	0.80888	5.101	9.259
19-109	2	12.1232	0.80888	10.044	14.203
19-110	2	14.7459	0.80888	12.667	16.825
19-111	2	13.9455	0.80888	11.866	16.025
19-112	2	12.0516	0.80888	9.972	14.131

Std Error uses a pooled estimate of error variance

Figure 19. Onaway ANOVA of 2540 Max Permeability by Design

3.2.2.3 2540 Prototype with PPG New Development 5 mil Membrane

20-MB-118 and 20-MB-119 prototype designs were constructed using pilot produced enhanced membrane with modified formulation and thickness increased to 5 mil (X-825 and X-826). The X-825 and X-826 membrane samples were produced from the same pilot trial of membrane production, but from different lots to assess membrane manufacturing variability. These membranes were produced via a simplified manufacturing process, which would significantly reduce membrane costs and has the potential for improved manufacturing quality, yield, and contaminant rejection. The properties of both X-825 and X-826 are listed below in Table 9. Flux for the thicker membranes was reduced, as expected, but still allowed water to pass through at a 70-74% reduction in permeability.

Table 9. Properties of 5-mil Membrane Utilized in 2540 Prototype Filters

Membrane ID	Dead End Flux at 50 psi	Gurley (seconds)	Thickness (millimeters)
X-825	66.48	639.5	5.27
X-826	57.25	756.3	5.59

All clean water performance test data completed with PPG pilot laboratory skid using deionized water with 250 ppm NaCl and 500 ppm PPG MZD7360J preservative to prevent bacterial formation. Concentrate crossflow rate for each filter was controlled to 3.5 GPM for 24-hour test period. Elements were tested in parallel pairs. Permeability (specific flux) data was normalized at 20°C using the following correlation of viscosity with temperature:

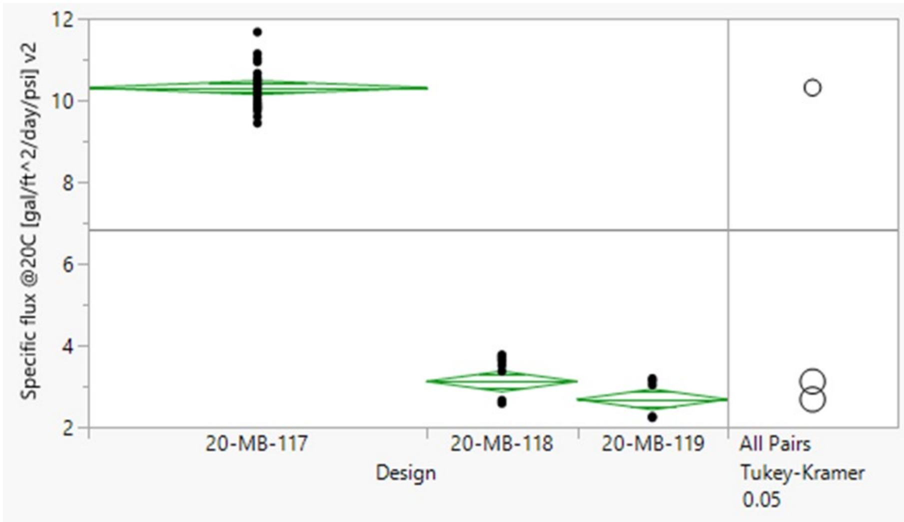
$$J\_20=J\_T \cdot e\hat{}([-0.032 \cdot (T-20)])$$

3.2.2.4 2540 Prototype with PPG New Development 5-mil Membrane Test Result

Three different 2540 filter designs were fabricated to test a modified thicker membrane with all filters having the same design other than the different membranes utilized in construction. Specific flux was compared amongst the three filter designs to analyze the results of the newly developed 5-mil membrane in the design of a spiral wound membrane filter. The 21-MB-117 filter design had higher specific flux than the other two filter designs that incorporated the 5-mil membrane (Figure 20). The higher specific flux is to be expected as the 117 series has a thinner membrane directly increasing the active membrane area compared to 118 and 119, as well as the thinner membrane being more permeable than the thicker experimental membrane.

Table 10 lists the components and design of the three different 2540 filters fabricate. 20-MB-117 design included commercial UF membrane of 4 mil thickness (PPMK087-08). 20-MB-118 and 119 were constructed using pilot produced enhanced membrane with modified formulation and thickness increased to 5 mil (X-825 and X-826). Clean water mean permeability of filters with prototype membranes was reduced by 70% and 74% compared to enhanced filter design from first phase of project, but this is within expectations for a thicker, more selective membrane (Figure 21).

20-MB-118 and 20-MB-119 designs utilized membrane from the same pilot trial of membrane production, but from different lots. >98% of the experimental variation between samples can be attributed to the targeted design factor (membrane).



Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
20-MB-117	36	10.3086	0.08348	10.142	10.475
20-MB-118	16	3.1181	0.12521	2.868	3.368
20-MB-119	16	2.6781	0.12521	2.428	2.928

Std Error uses a pooled estimate of error variance

Figure 20. PPG 5-mil Membrane Onaway ANOVA of 2540 Permeability by Design

Table 10. Summary of the Design of Different 2540 Filter Tested for Clean Water

Sample ID	Membrane	Membrane leaves	Permeate carrier type	Feed Spacer	Glue	PWT diameter
20-MB-117	UF PPMK087-08	4	12 mil 39389	43 mil 11x11	UV 4000	0.84
20-MB-118	X-825	4	12 mil 39389	43 mil 11x11	UV 4000	0.84
20-MB-119	X-826	4	12 mil 39389	43 mil 11x11	UV 4000	0.84

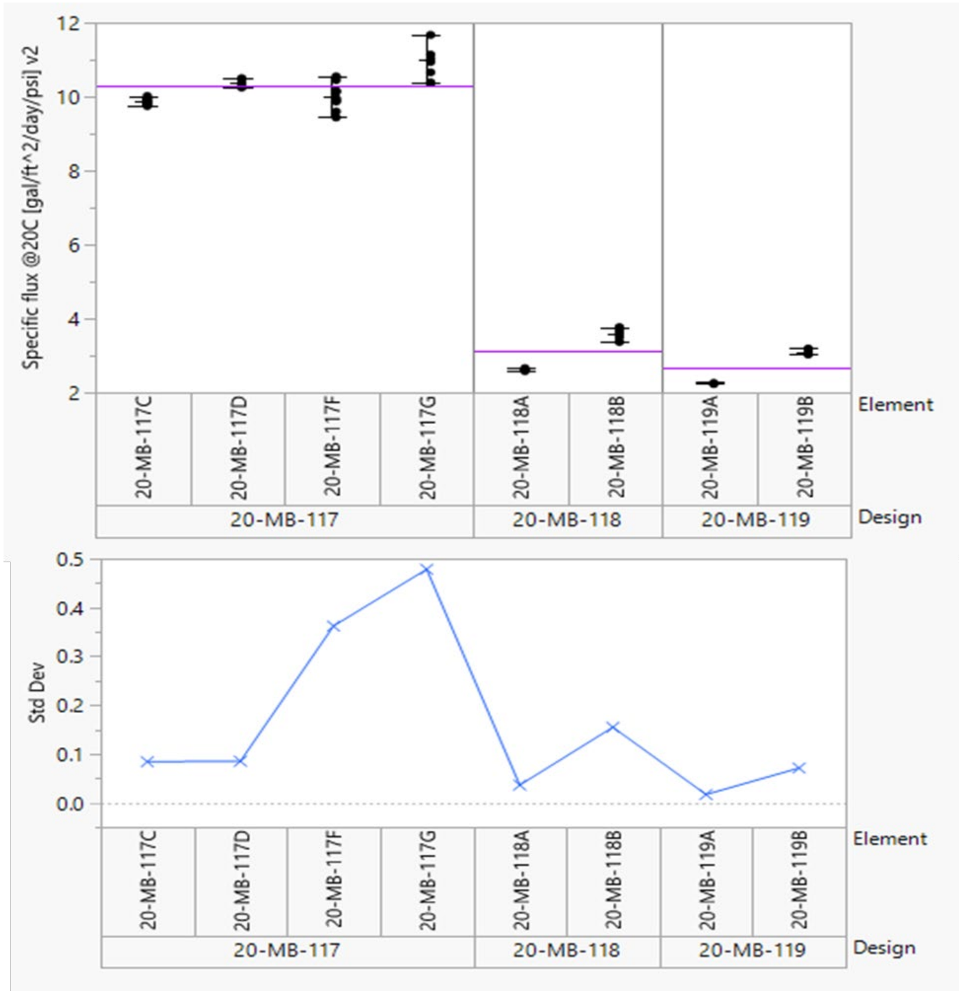


Figure 21. Specific Permeability Variability Chart of all 2540 Filters Tested

3.3 Filter Performance Evaluation

3.3.1 Prototype Filter Leak/Integrity Testing Apparatus

New leak tester has been set up to use for prototype filter integrity testing, replacing the previously used “dunk test.” Apparatus has capability of testing pressure decay, vacuum decay, and air flow. Prior to conducting the integrity testing the filter being tested will be wetted with a wetting solution for 30 minutes with an inlet pressure of 15.0 psi and then connect to the Uson Sprint IQ vacuum pump pictured in Figure 22. To ensure the filter has no defects a pressure decay and vacuum decay test are conducted prior to any baseline testing of designed filters. The pressure decay test fills the housing for two minutes reaching a 5.0031 psi then has a two-minute test cycle followed by a forty-five second vent to finish the pressure decay test. If the pressure decay is lower than 1.00 psi the filter has passed the pressure decay test if not, then the filter has failed. Upon completion of the pressure decay test the vacuum decay test is conducted to ensure there are no defects with the filter being tested. The vacuum decay test fills the housing for five minutes to an initial pressure of -5.112 psi then has a six-minute test cycle followed by a forty-five second vent to finish the vacuum decay test. If the vacuum decay is lesser than -4.112 psi then the filter has passed and if not, then the filter failed the vacuum decay test. The filter must pass both the pressure decay and vacuum decay to pass the integrity test and to warrant any future testing.

Figure 22. Uson Sprint IQ Vacuum Pump Station at PPG

3.4 Spiral Wound Membrane Development

3.4.1 Design

Filter design work builds upon work in the prior phase with a focus on feed channel optimization where the prior phase focused on optimization of permeate path through leaf length and carrier design. Test filters used 6 and 12 leaf designs with UF membrane used in three designs with antifouling UF membrane used in a fourth experimental design listed in Table 11. All filters were created via the same method, the same permeate carrier, and utilized the same adhesive for all four filters created. Feed spacer was selected based on flat-sheet biofouling testing performed by NDSU and the highest performing spacer was then tested in spiral wound membrane elements to determine the impact on pressure drop and flux.

Table 11. Summary of 4040 Filters Design

ID	Membrane	Leaf Count	Area (sq. ft.)	Feed spacer
MB101	PPMK087-08	12	6.25	43 mil 11x11 Diamond SWM
MB102	PPMK087-08	12	6	44 mil Int
MB103	PPMK087-08	6	12.75	43 mil 11x11 Diamond SWM
MB104	Coated UF 09230-01	12	5.5	44 mil Int

3.4.2 Clean Water Performance Testing

Following the assembly of elements, all elements were tested in filtered deionized water with the addition of sodium chloride for conductivity and biocide to prevent bacterial growth in the filters and system to not obscure results though fouling. The system was temperature controlled to approximately 68°F with a step pressure test run from 10 psi to 50 psi followed by 24-hour steady state condition at 20 GPM of concentrate flow on four of each filter design.

Upon completion of the 24-hour steady state test for all four filter elements the normalized flux was plotted against TMP, with 21-MB-102 having the highest normalized flux of all four filter designs (Figure 23). 21-MB-101 had the next highest normalized flux with 21-MB-103 and 21-MB-104 following respectively. The antifouling membrane utilized in 21-MB-104 would decrease the flux as this was observed earlier for a thicker, more selective membrane.

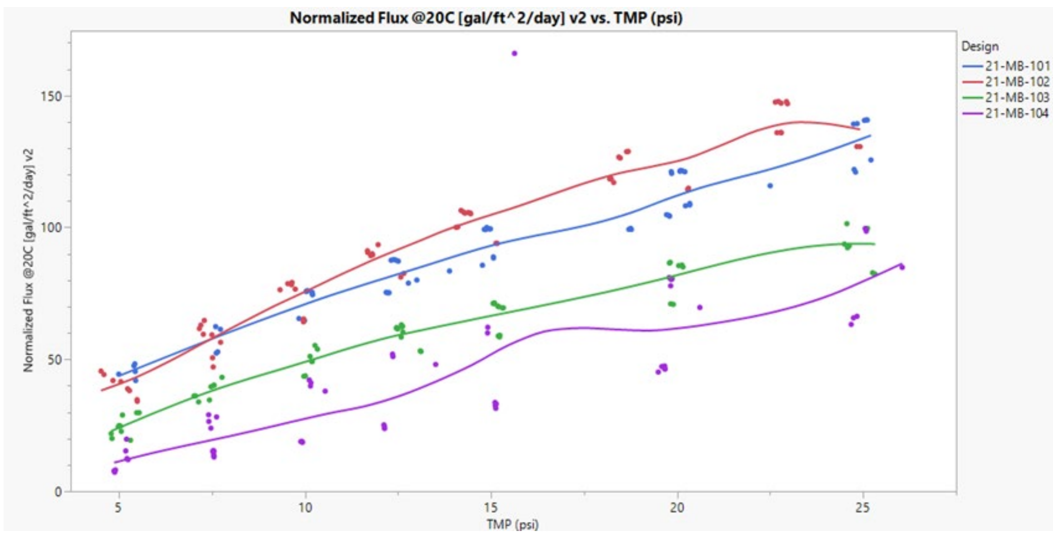
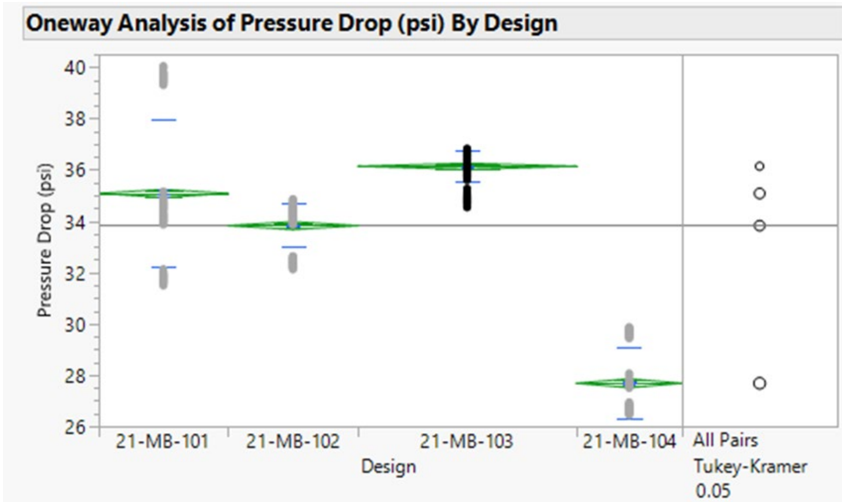


Figure 23. 24-Hour Clean Water Normalized Flux vs. TMP of Four Different Filter Designs

All four filters were analyzed statistically for pressure drop upon conclusion of the 24-hour steady state test (Figure 24). 21-MB-104 had the smallest pressure drop amongst all four filters with 102, 101, and 103 following respectively. The two filters with the 44-mil Int feed spacer produced lower pressure drops than the other two filter designs that utilized the 43-mil 11x11 SWM feed spacer. The 44-mil Int feed spacer improved the pressure drop of filter 21-MB-102 compared to 21-MB-101, which had the same design other than a slight difference in leaf area. The pressure drop observed in filter 104 can be attributed to the thicker membrane as well as the lower porosity which results in a more selective membrane. The highest-pressure drop was observed in 21-MB-103 which was the only filter design with 6 leaves compared to 12 leaves for all other three filter designs.

Upon completion of the 24-hour steady state test all four filters were statistically analyzed for pressure drop and normalized flux of each filter design (Figure 25). 21-MB-102 performed best with the highest normalized flux of 121.330 (gal/ft^2/day) and the second to lowest pressure drop with 33.8262 (PSI). When comparing 21-MB-102 to 21-MB-101 the only differences between them are the feed spacer and the leaf area (ft^2) with 21-MB-102 having 0.25 (ft^2) less leaf area than 21-MB-101. The smaller leaf area in these two filters results in a difference of 3 ft^2 less in 21-MB-102 compared to 21-MB-101. The differences in the two designs led to a 9.2% increase in normalized flux and a 3.7% decrease in pressure drop for 21-MB-102 compared to 21-MB-101. 21-MB-104 filter produced the lowest normalized flux which was observed earlier for a more selective membrane but had a 90.9% decrease in normalized flux compared to 21-MB-101 and a 108% decrease in normalized flux with respect to 21-MB-102.



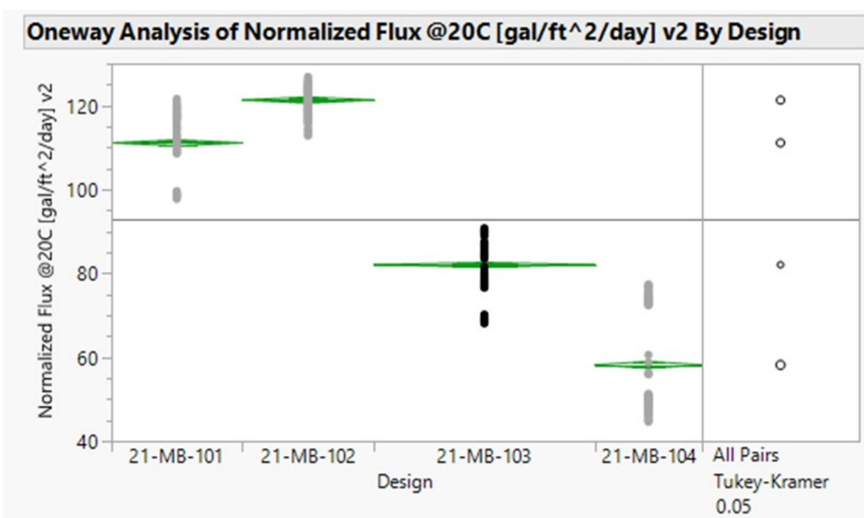
Oneway Anova

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
21-MB-101	383	35.0814	0.08022	34.924	35.239
21-MB-102	386	33.8262	0.07991	33.669	33.983
21-MB-103	648	36.1413	0.06168	36.020	36.262
21-MB-104	314	27.6818	0.08860	27.508	27.856

Std Error uses a pooled estimate of error variance

Figure 24. Onaway ANOVA Analysis of Pressure Drop by Design



Oneway Anova

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
21-MB-101	383	111.120	0.37396	110.39	111.85
21-MB-102	386	121.330	0.37250	120.60	122.06
21-MB-103	648	82.063	0.28750	81.50	82.63
21-MB-104	314	58.186	0.41301	57.38	59.00

Std Error uses a pooled estimate of error variance

Figure 25. Onaway ANOVA Analysis of Normalized Flux of the 4040 Filters Tested

3.4.3 Groundwater Simulant Testing

The elements used in clean water testing were then validated in a fouling environment using a groundwater simulant solution co-developed between PPG and GVSC based a combination of various groundwater sources in which the systems may be deployed with the addition of a test dust to contribute to solids fouling and tannic acid to simulate organic materials present in surface water listed in Table 12. Tests were run for 72 hours with a pair of filters of the same design run in parallel with a combined feed flow of 32 GPM, a slightly lower feed flow was selected compared to clean water test due to the behavior of the electric diaphragm pump at high flow rates. During this test, the pump speed was run at 40 Hz (67%). The permeate control valves were set to discharge to the effluent tank for the first 2,000L of challenge water run through the system and then recirculate to the feed tank for the remaining time on the test. Samples were taken of feed, concentrate and permeate over the course of the test to determine filtration capability, any potential degradation of performance, and rate of fouling in the element.

Table 12. Formulation for Simulated Groundwater Used for Groundwater Testing of 4040 Filters

Salt	g/1000L (ppm)	weight (g)/1500 Gal
CaCl2	55.5	316
MgSO4	42	239
Ca(HCO3)2	324	1842
KNO3	5.05	28.7
Na2SiO3	37.8	215
FeSO4	12.8	73
NaCl	46.4	264
Tannic Acid	15	85.3
ISO specification 12103-A2 fine test dust	N/A*	
* Add until target turbidity of 30-50 NTU		

In Table 13 and Table 14 the overall reduction of the groundwater was analyzed by taking the initial feed samples and the final permeate sample of each filter. The overall series combined each of the four filters were tested with groundwater listed in Table 12. The initial feeds for all groundwater varied in the amount of individual constituents present in the initial feed that was tested analytically, which resulted in varying certain series reductions. Variance in the reduction of series was affected by the initial feed constitution since some factors were extremely high compared to the lower amounts in other feeds resulting in higher percent reductions for series with higher initial concentrations and lower percent reductions for feeds with lower initial concentrations. The amounts varied for all four-filter series feeds as well the two different feeds used between the two filters ran in parallel for each series.

Table 13. Reduction of Groundwater from Initial Feed to Final Permeate for all Four Filters (101 & 102 Series)

	101 Series	% Reduction	102 Series	% Reduction
Laboratory pH	-0.155	-2.003	-0.16	-2.070
Specific Conductance (µS/cm) @25ºC	36.25	8.220	17.75	4.142
Alkalinity (mg/L)	17.375	18.598	39.05	26.906
Acidity (mg/L)	-22.775	26.138	-37.125	21.950
Iron (mg/L)	0.175	91.935	0.0875	64.305
Manganese (mg/L)	0.025	100.000	0.0075	37.500
Aluminum (mg/L)	0.04	35.897	0.065	62.941
Sulfate (mg/L)	0.125	0.616	3.55	14.022
Calcium (mg/L)	42.05	48.376	12.025	21.862
Magnesium (mg/L)	0.175	3.052	0.225	4.245
Potassium (mg/L)	0.15	5.769	0.3	10.023
Sodium (mg/L)	0.325	1.064	0.65	1.983
Chloride (mg/L)	0.325	0.524	0.4	0.666
Turbidity	21.21275	98.928	174.882	99.933

Table 14. Reduction of Groundwater from Initial Feed to Final Permeate for all Four Filters (103 & 104 Series)

	103 Series	% Reduction	104 Series	% Reduction
Laboratory pH	-0.14	-1.801	-0.05	-0.647
Specific Conductance (µS/cm) @25ºC	1.75	0.011	17	3.744
Alkalinity (mg/L)	22.4	21.246	32.525	28.497
Acidity (mg/L)	-31.575	32.162	-28.05	25.947
Iron (mg/L)	0.6325	91.697	0.2525	83.239
Manganese (mg/L)	0.0125	41.667	0.015	50.000
Aluminum (mg/L)	0.0925	89.286	0.0075	-50.000
Sulfate (mg/L)	0.5	2.129	2.375	11.229
Calcium (mg/L)	91.725	47.920	36.85	42.714
Magnesium (mg/L)	-0.225	-11.130	0.525	9.597
Potassium (mg/L)	0.35	13.182	-0.075	-3.107
Sodium (mg/L)	1.45	3.759	0.325	0.482
Chloride (mg/L)	0.325	0.518	-0.65	-1.011
Turbidity	8.152	97.981	19.686	98.935

The four filters in each series were evaluated by InnoH2O for the flux of each filter in the series to capture rapid initial fouling of the filters and is presented for the first five hours of the 72-hour test and listed below in Table 15 with the plots of all 16 filters assessed in Appendix A. The slopes of flux for each filter for hours 6 through 72 are presented in Table 16 with the plots of all 16 filters assessed in Appendix A. The value of slope indicates rate of loss of flux over this period, a lower number indicates the filter is more resistant to fouling in this test while a higher number indicates a filter that is less resistant to the fouling in this test.

Table 15. Summary of Average Slopes of Initial Fouling in Groundwater Testing of all Four Filter Types

Filter Design	Average Slope	Standard Deviation
101 Series	-4.1004	0.4169
102 Series	-3.7850	0.2424
103 Series	-2.6032	0.4877
104 Series	-4.3580	0.4864

Table 16. Summary of Average Slopes of Overall Fouling in Groundwater Testing for all Filter Designs

Filter Design	Average Slope	Standard Deviation
101 Series	-0.3258	.0364
102 Series	-0.3250	0.1428
103 Series	-0.1745	0.0868
104 Series	-0.0918	0.0271

From the initial normalized flux at 20ºC for the first five hours of the 72-hour groundwater test the rapid fouling of the filters can be compared from the slopes of each filter design series of 21-MB-101 to 21-MB-104. In the Table 15 filter series 102 had 21-MB-102B dropped from the average as it was an outlier as well as the 103 series which had 21-MB-103F dropped. The two filters dropped from the average was due to irregular TMP recorded which is used in the normalized flux calculations. They both had permeate flows decline linearly but whenever graphed with normalized flux they do not show linear decline due to the TMP used in the calculations. From Table 15 the filter series rate of fouling can be evaluated with the 103-design having the lowest slope with 102, 101, and 104 designs following respectively. The MB-103 design slope indicates that this series is the least prone to rapid initial fouling but have lower normalized flux than the MB-102 and MB-101 designs indicated by the y-intercept of the equation in each plot. The MB-104 series did show a reduction in decline of flux compared to the MB-102 which is the same design other than the membrane used but had an average flux lower than the MB-102 design. The MB-104 series performed best in fouling over 72 hours with the lowest slope with MB-103, MB-102, and MB-101 following respectively. The MB-104 series had the lowest normalized flux of all filters designed with MB-102 having the highest over 72 hours with respect to the y-intercept in the charts listed in the appendix with MB-101, MB-103 and MB-104 following respectively. In Table 17, the normalized flux of all four filter designs is presented after the initial fouling of 6 hours then every 24-hour increments. MB-102 series produced the highest initial normalized flux after initial fouling with 61.75 (gal/ft^2/day) and a final normalized flux of 48.78 (gal/ft^2/day) after 72 hours. The lowest normalized flux initially was the MB-104 series and resulted in the lowest final normalized flux after 72 hours which was evidenced earlier for a more selective membrane. MB-103 series had the lowest reduction in normalized flux over the 72 hours of groundwater testing with a reduction of 13.16% which was followed by MB-102 with a 21.00% reduction, MB-104 had a 25.67% reduction, and MB-101 had a 35.33% reduction in normalized flux upon completion of the 72-hour groundwater testing. The higher flux and lower slopes indicate that the MB-102 and M-104 series should be further evaluated since they performed better than the other two filter designs.

Table 17. Normalized Flux (gal/ft2/day) of all Four Filter Designs Over the 72-Hour Groundwater Test

Filter Series	MB-101	MB-102	MB-103	MB-104
Normalized Flux @ 6 Hours	61.49	61.75	49.07	27.42
Normalized Flux @ 24 Hours	52.91	58.13	45.87	22.56
Normalized Flux @ 48 Hours	45.38	53.39	44.38	21.24
Normalized Flux @ 72 Hours	39.76	48.78	42.61	20.38

3.5 Skid Design

3.5.1 Design

Skid design was performed concurrently to filter design and construction in order to extract the best performance from high flux spiral wound membrane. InnoH2O performed upgrades to the PPG test skid to reduce downstream pressure created by higher than anticipated permeate flow and improved data logging capabilities in anticipation of automation based on flow and pressure conditions in the deliverable skid. The deliverable skid was also constructed by InnoH2O with a fully automated valve system and a single electric diaphragm pump to be used for both positive pressure and back-pulsing cycles.

3.5.2 PPG Filtration Skid Upgrades

InnoH2O installed upgrades to PPG pilot skid used for prototype filter performance testing to maximize the filtration capability and lifetime of designed filters (Figure 26). To maximize the potential of the filters causes of biofouling was the guiding principle in the upgrades to the system. The skid upgrades completed include relocation of the centrifugal process pump, filter housings, bag filters, the addition of an electric diaphragm pump, and rerouting of permeate, feed, and concentrate lines for reduced backpressure. The human machine interface and programmable logic controller have been upgraded from that provided by the initial AVANTech design, which includes full data acquisition capability for improved experimental data collection. A Cooling coil in feed tank has been installed to maintain a constant temperature when conducting 24-hour tests. Permeate line was rerouted directly to tank to more closely match GVSC skid design. Equipment upgrade performance has been validated using Phase I enhanced design filter to compare to PPG skid prior to upgrades and Phase I skid delivered to GVSC. Clean water performance test data completed with PPG pilot laboratory skid using deionized water with 250 ppm NaCl and 500 ppm PPG MZD7360J preservative to prevent bacterial growth in the system.

Figure 26. Upgraded Skid Design Currently at PPG

3.5.3 PPG Filtration Skid Upgrade Test Result

Performance testing utilized the enhanced design 4040 prototype filter 21-MB-101. This design is equivalent to 10-MB-110 series filters. These filters contain commercial UF membrane (PPMK087-08), 43 mil diamond feed spacers with 11x11 strand/inch mesh, and polyurethane adhesive. 4040 filter elements are tested individually with a step ramp test with 5 psi increments from 10 psi to 50 psi as measured at pressure sensors at the inlet to the membrane vessels. Permeate flow was unrestricted, and testing was performed with both centrifugal and diaphragm pumps. Feed pressure ramp testing was performed to compare filter performance using upgraded PPG skid to Phase I delivered GVSC skid and PPG skid prior to upgrades. Permeate line rerouted in rev1 of upgraded skid, normalized flux at 20C compared to hydraulic horsepower now matches efficiency of GVSC skid, permeate pressure is reduced. Permeate flow sensor was maxed out at high pressure conditions.

The upgraded system provided improvement in pressure drops of the system at higher feed pressures than previous skid designs (Figure 28), which will aid in the reduction of biofouling as seen in Figure 27. The permeate pressure decreased by roughly 80% when feed pressure reached 50 psi in the upgraded skid compared to the previous skid used at PPG. From Figure 26 the upgraded skid design also provided higher normalized flux than previous skid designs with a doubling of normalized flux at 0.7 horsepower (hp) from the upgraded skid to the older skid. The upgraded skid at PPG had higher normalized flux than the GVSC skid, which will aid in improvement of the filters as well as increase the recovery potential of the filters. The upgraded PPG skid behaves more closely to the GVSC skid when comparing normalized flux and permeate pressures.

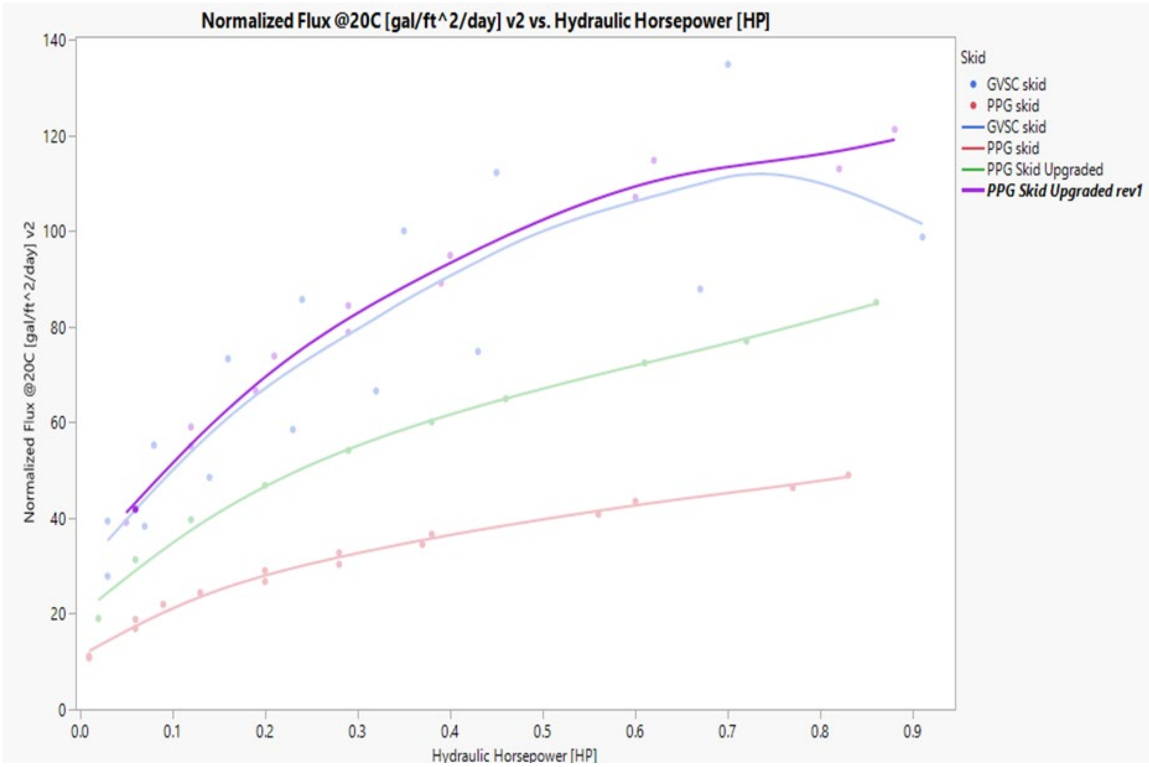


Figure 27. Normalized Flux vs. Hydraulic Horsepower of Different Skid Designs

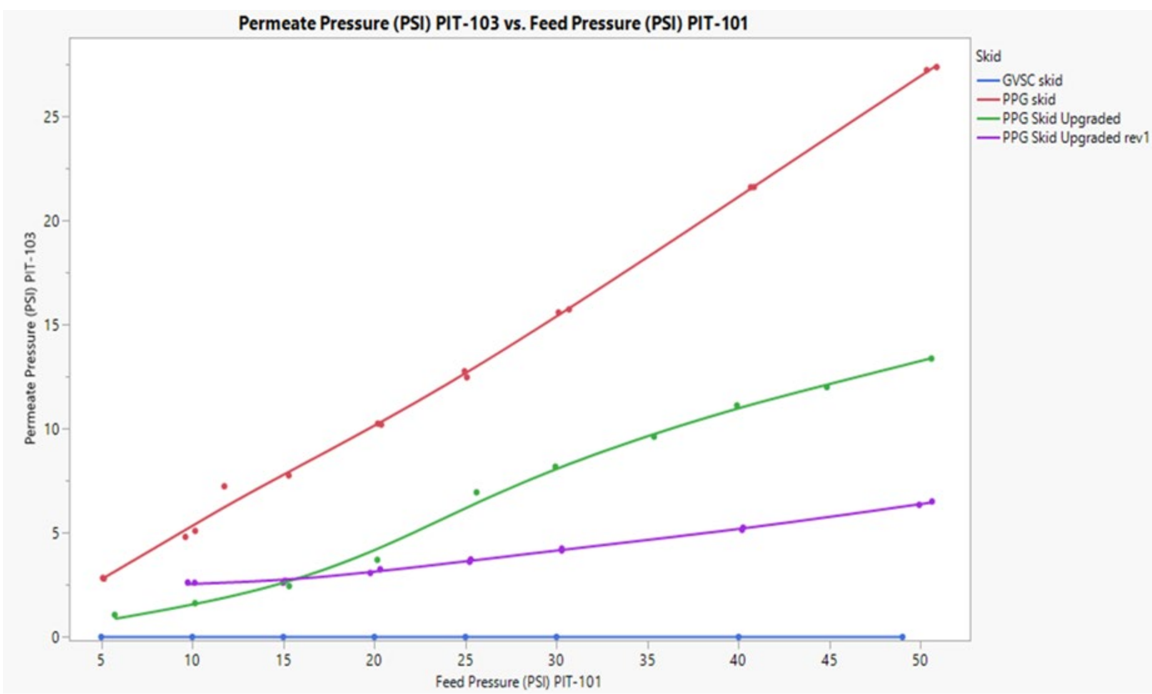


Figure 28. Plot of Permeate Pressure vs. Feed Pressure of Different Skid Designs

3.5.4 Enhanced Filter Design Comparison

Comparative testing was completed using enhanced design 4040 prototype filter 21-MB-101 and 21-MB-102. These filters contain commercial UF membrane (PPMK087-08), same number of leaves, permeate carrier and polyurethane adhesive. The experimental element utilized the same construction, but 44 mil diamond feed spacers from specialty supplier Int was utilized in 21-MB-102 while 21-MB-101 used 43 mil diamond feed spacer with 11x11 strand/inch mesh. 4040 elements were tested individually under the same conditions to compare the design of the two best designs witnessed in previous tests. Feed pressure was ramped in 5 psi increments from 10 psi to 50 psi as measured at pressure sensors at the inlet to the membrane vessels. Permeate flow was unrestricted, and testing was performed using the centrifugal pump while being temperature controlled. Low limits on flow prevented some 5 psi readings on this test, and high-pressure conditions exceeded max flow readings on the permeate sensor.

In Figure 29, pressure drops when plotted against concentrate flow both filter designs behaved similar with 21-MB-102 having slightly higher pressure drops at lower concentrate flows while 21-MB-101 had higher pressure drops with higher concentrate flows respectively. When TMP was plotted against normalized flux the 21-MB-101 series outperformed 21-MB-102 as evidenced in Figure 30 with the 102 series producing higher flux with lower TMP. The trend is the same as seen in smaller filters with higher flux produced by the filter series with the larger active membrane area which 21-MB-101 has when compared to 21-MB-102.

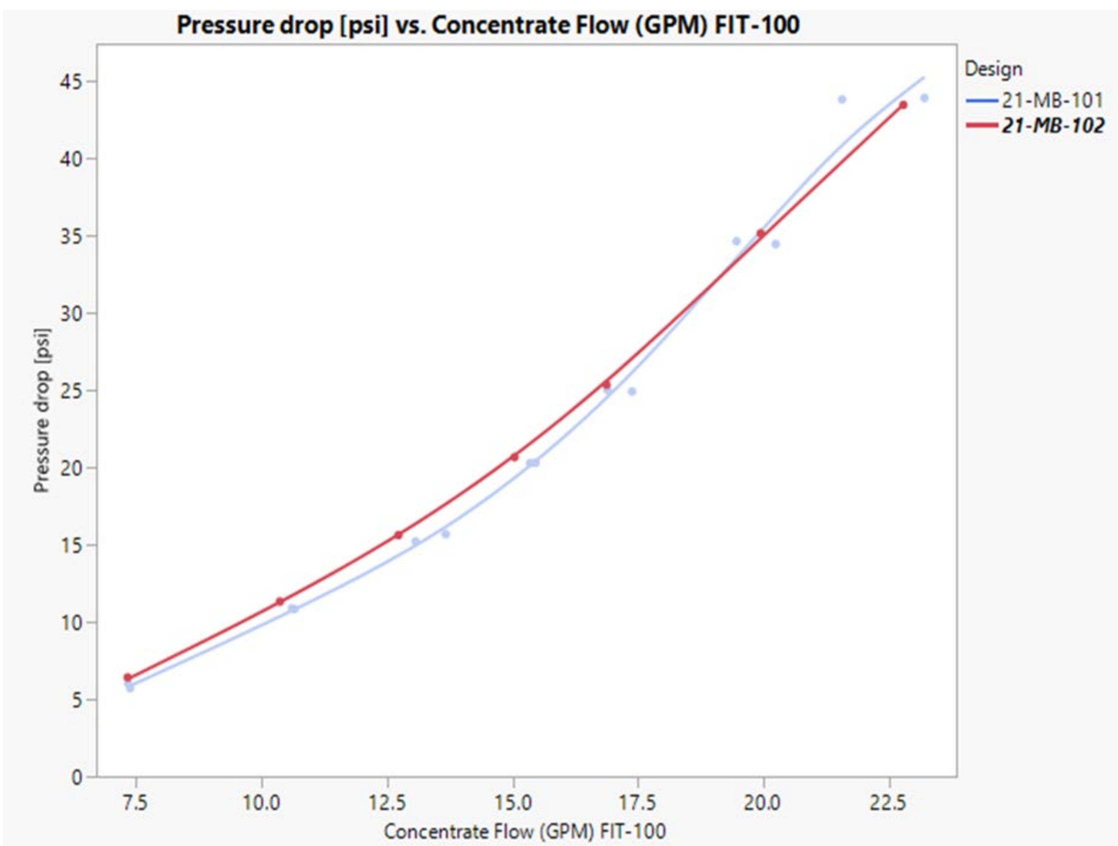


Figure 29. Pressure Drop (psi) vs. Concentrate Flow (GPM) 43 mil 11x11 diamond (21-MB-101) vs. 44 mil Int (21-MB-102)

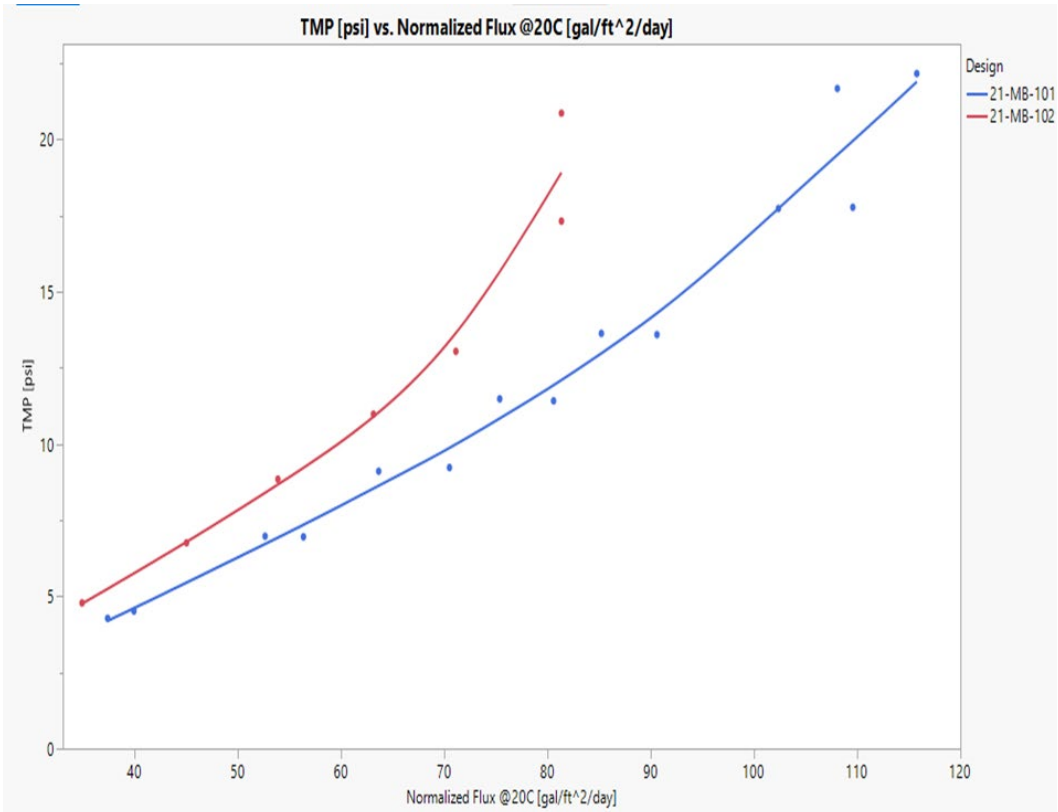


Figure 30. TMP (psi) vs. Normalized Flux at 20°C (Gal/ft2/day) 43 mil 11x11 diamond (21-MB-101) vs. 44 mil Int (21-MB-102)

In Figure 31, the MB-21-101G had a maximum concentrate flow of 23.7 GPM at a 43.2 psi pressure drop. While in Figure 32, the MB-21-102E had a maximum concentrate flow of 23.3 GPM at a 43.0 psi pressure drop. The MB-21-101 series had an average maximum concentrate flow of 22.3 GPM while the MB-21-102 series had an average maximum of 23.1 GPM. The 21-MB-102 series performed better with lower pressure drops and higher concentrate flow compared to the 21-MB-101 series, which has been observed in the clean water and groundwater tests performed thus far.

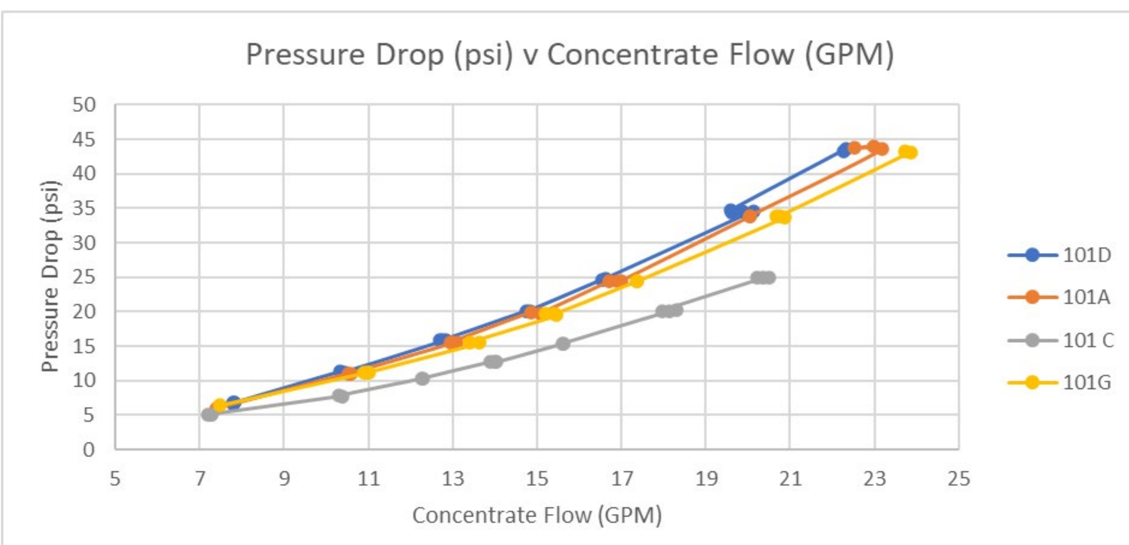


Figure 31. Pressure Drop (psi) vs. Concentrate Flow (GPM) of 21-MB-101 Filters

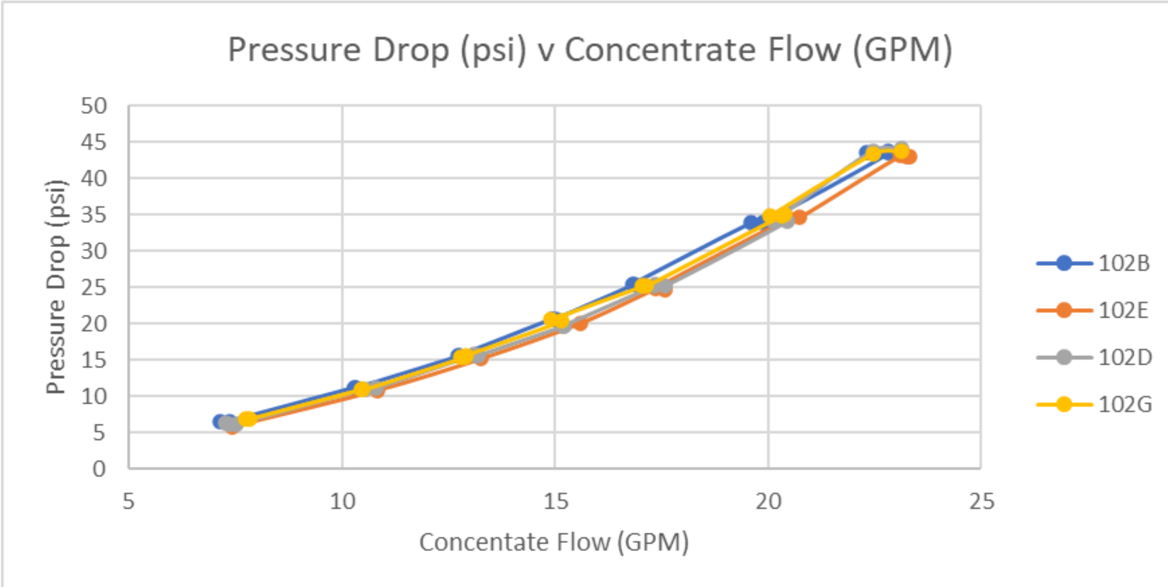


Figure 32. Pressure Drop (psi) vs. Concentrate Flow (GPM) of 21-MB-102 Filters

In Figure 33, the 21-MB-101A filter had a maximum normalized flux of 134.2 GPM with a TMP of 25.1 psi and in Figure 34 the 21-MB-102D had a maximum normalized flux of 140.8 GPM with a TMP of 22.9 psi. The 21-MB-101 filter series had an average maximum normalized flux of 127.5 GPM with an average TMP of 24.3 psi, where the 21-MB-102 filter series had an average maximum normalized flux of 133.5 GPM with an average TMP of 23.4 psi. The 21-MB-102 filter series performed better than 21-MB-101 filter series as evidenced when comparing the concentrate flow to the pressure drop of each of the four filters in each design. The 21-MB-101 series performed similarly to the 21-MB-102 series but did not perform with the maximum normalized flux and indicative of the 44-millimeter Int feed spacer present in the 21-MB-102 series which was superior to the 43-mil 11x11 diamond feed spacer in previous testing.

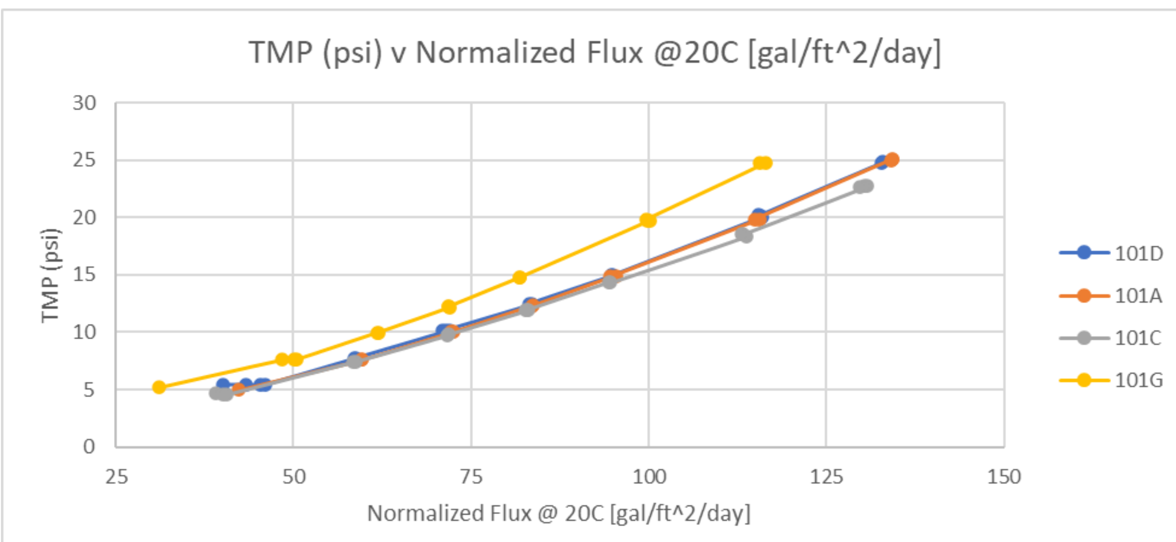


Figure 33. TMP (psi) vs. Normalized Flux at 20°C [gal/ft2/day] for 21-MB-101 Filters

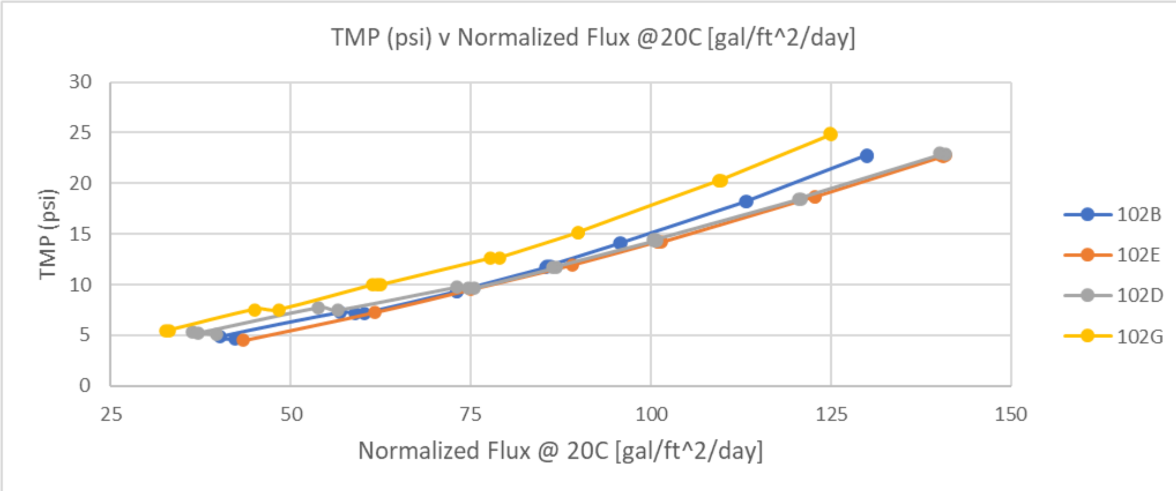


Figure 34. TMP (psi) vs. Normalized Flux at 20°C [gal/ft2/day] for 21-MB-102 Filters

3.5.5 Pilot Scale Skid Redesign and Relocation

The original membrane skid was housed at Ehly 108 at NDSU. The skid was co-located with many construction and structural engineering equipment and dust was a major problem there. Further, the location did not have enough working space. NDSU worked with InnoH20 on the new design (Figure 35 and Table 18). The skid will be upgraded to match the PPG test skid currently located at Monroeville. InnoH2O will perform commissioning of the upgraded system as well as training on how to utilize the skid with the automated data logging to maximize spiral wound membrane filters potential and lifetime.

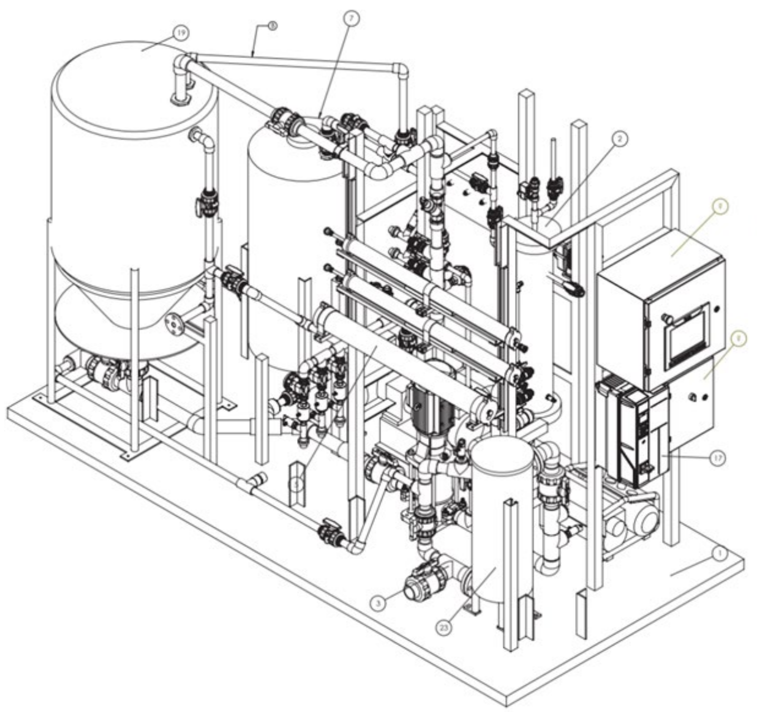


Figure 35. Sketch of the New Skid at NDSU
The numbers represent individual component of the skid.

NDSU decided they were going to upgrade the skid, and this provided an opportunity to move the skid to a better location. InnoH2O offered to make the skid more compact as they upgraded the unit from 10’x20’ to a more compact 5’x8’ skid. The NDSU team dismantled the old unit (April 2022), and the dismantled parts were shipped to InnoH2O (second week of May 2022). InnoH2O reused most of the parts from the old system and shipped the updated (new) unit back to NDSU in the first week of October 2022. Pipes were increased in size to minimize pressure drops across the system to better match the PPG skid at Monroeville. The capability to add a second 4040 housing was added to the system so, that two 4040 filters can run in parallel. Diaphragm control valves added to the permeate side of the membrane housings and updated control system with the ability to collect data were the upgrades from the Phase I skid to the current Phase II skid. The new skid is now waiting commissioning (at its new home at NDSU R1A 1124). InnoH2O will be commissioning the upgraded skid at NDSU. The upgrades to the NDSU skid are listed in Table 18.

Table 18. Components of the New Skid at NDSU

#	Component	Function/Purpose
1	P001, Multi-stage Centrifugal Primary Process Pump	Primary process pump, used for flow water across spiral wound membranes; 5hp
2	P002, Electric Air Diaphragm Pump	Alternate primary process pump and back-pulsing pump; used to flow water across spiral wound membrane and back-pulse same; 2hp
3	TK001, Process Tank	This 110-gal. tank is used for wastewater collection and circulation. It can also be used as a clean-in-place tank for membrane cleaning.
4	BF001, Pre-Filter	This filter is used to remove suspended solids that may otherwise plug or foul the membrane filter. The stainless-steel housing uses a #1 Bag.
5	SWF001, 4040 Membrane Housing	This is a traditional end-ported housing made for 4040 spiral wound membranes. It has 3 ports- feed inlet, concentrate outlet, and filtrate.
6	SWF002 & SWF003, Membrane Housing	This is a traditional end-ported housing made for 2540 spiral wound membranes. It has 3 ports- feed inlet, concentrate outlet, and filtrate.
7	Back-pulse Pressure Vessel	The Back-pulse pressure vessel can be used to back-pulse/backwash the membranes.
8	TK002, Permeate Tank	Used for collecting permeate from the membrane.

4. Conclusions

4.1 Membrane Optimization

For membrane optimization, microbial fouling was completed at NDSU applying the laboratory scale cross flow methodology developed at NDSU. For membrane flux different membranes were extruded at PPG with different amounts and types of silica to optimize flux via flat sheet testing of dead-end flux at PPG. The flux of membranes increased with an increase in the amount of silica loaded into each mixture and was selected for scaling up. The three membranes tested for biofouling varied in lab bench testing with the MF membrane producing the highest flux with the antifouling membrane the lowest, the antifouling had the least porous compared to the MF which was the most porous of all three membranes tested. Three membranes were analyzed for biofouling of the membranes, the antifouling (833-1915) membrane produced a 23% reduction in comparison to the UF membrane (PPMK087-08) which had a reduction in bacterial colonization with a mean reduction of 19% compared to the MF membrane (769-6412). When comparing the bacterial colonization of the three different membranes feed spacers were also tested with a mean reduction of 38% for the UF membrane in comparison to the MF membrane, and the antifouling (833-1915) membrane had an equivalent amount of bacterial colonization with respect to the UF membrane. Eleven different feed spacers were compared with the UF membrane utilized as the control membrane for all feed spacers analyzed for bacterial colonization. The Int feed spacer produced the lowest RFU correlating to bacterial colonization with the 44-mil Int having a mean RFU of 6,718 compared to the 44-mil Alg mean RFU of 9,412 and the 9x9 43 mil treated with a mean RFU of 9,009. The 44-mil Int feed spacer had a 28% reduction in bacterial colonization compared to the treated 44-mil Int feed spacer. The Int feed spacers were tested for flux before and after the 24-hour biofouling test with the 44-mil Int having a 2% reduction in flux, while the 44-mil treated Int had a 5% reduction in flux after biofouling. With the completion of the biofouling testing conducted at NDSU the UF (PPMK087-08) membrane and Int feed spacer will be utilized in
Phase III.

4.2 Prototype Filter Design

For the prototype design 2514 filters were first analyzed then scaled up to 2540 filters and finally 4040 filters were fabricated based on earlier prototype designs at PPG. The 2514 filter series varied the number of leaves and the type of permeate carrier to increase the flux of the filters when given a nominal size. When varying the number of leaves the filters utilized the standard UF membrane, 31 mil diamond feed spacer and epoxy adhesive was used in fabrication of the filters and had a 24-hour clean water test to measure permeability of each filter design. In Table 7 the mean specific permeability from the 24-hour test as well as the elemental design is listed, and the increase in mean specific permeability trend observed saw increases with the increase of number of leaves until eight leaves were used which was statistically not significant. The increase in mean specific permeability for two to four leaves was a 71% increase, a 26.5% increase for four to six leaves, and then a 34.9% decrease was observed for six to eight leaves. The max specific permeability illustrated in Figure 15 had a similar trend observed with a 69.9% increase from two to four leaves, 27.1% increase from four to six leaves, and a 2.02% decrease when increasing from six to eight leaves. The permeate carriers tested were illustrated in Figure 16 which saw the 20-mil permeate carrier having the largest increase in max specific permeability. The 20-mil permeate carrier had a 26% increase to the porous 12-mil permeate carrier and a 56% increase to the standard permeate carrier utilized. The more porous 12-mil permeate carrier had a 24% increase in max specific permeability compared to the standard 12-mil permeate carrier. From the 2514 filters fabricated and tested the 2540 filters fabricated and tested had the number of leaves varied and different experimental membrane to evaluate filter performance. From Table 8 the same trend as the 2514 filters was observed when increasing the number of leaves utilized in the construction of the 2540 filters. The increase in mean and max specific permeability was significant for increases of two to four and not statically significant for increases of four to six leaves. The increase of two to four leaves had a 48% increase in mean specific permeability and a 69% increase max specific permeability, while increase of four to six produced a 1.85% increase in mean and a 3.07% increase in max specific permeability. The two different experimental membranes tested saw a 70% reduction in filter designs with the X-825 membrane and a 74% reduction in the X-826 membrane filters compared to the control which had the standard UF membrane. The two experimental membranes tested were less porous and thicker than the standard membrane thus resulting in the reduction specific flux, which is expected for a more selective membrane. Upon completion of the smaller filters fabrication and testing as well as trends observed a set of four different 4040 filters were fabricated and tested for clean water testing at PPG. The fabrication design of these four-filter series is presented in Table 11 and had 24-hour clean water testing to compare all four series. These filters were later tested for groundwater by InnoH2O and is discussed in the sections following. Upon completion of all testing the 21-MB-102 and 21-MB-104 filters were selected for future evaluation.

4.3 Filtration Performance of Fresh Water

The 21-MB filter designs fabricated had a 24-hour clean water test to determine a design for Phase III efforts. The 21-MB-102 series performed the best with the highest normalized flux of 121.330 (gal/ft^2/day) and had a 22% increase in pressure drop compared to the 21-MB-104 series which was constructed of a more selective membrane. Presented in Figure 24, MB-102 series had an 8.41% increase compared to MB-101 which had a normalized flux of 111.120 (gal/ft^2/day) and 82.063 (gal/ft^2/day) and 58.186 (gal/ft^2/day) for MB-103 & MB-104 respectively. Pressure drops were also analyzed with minimal differences amongst MB-101,102, and 103, but MB-104 had a 21.09% and 23.41% decrease compared to 101 and 103 designs demonstrated in Figure 23. The Int feed spacer was utilized in the 102 and 103 series which performed best with pressure drops thus should result in less fouling when clean water is not utilized. MB-103 was the only filter that was constructed with 6 leaves compared to 12 leaves for the other three filter designs confirming the observed trends of the increase of flux with increases in number of leaves in the smaller prototype designs. The MB-102 series performed superior to the other three filter designs when maximizing the flux of the filters which confirms this design selection for Phase III studies.

4.4 NSF 61 Certification

NSF 61 certification was transferred from Phase II to Phase III. NSF provide the development of public health standards and certification programs for water, consumer products and environment. NSF can provide certification of membrane filter elements to NSF/American National Standards Institute (ANSI) Standard 61/372: Drinking Water System Components health effects. PPG has completed the product information form, certification parts list and client information form to work for an estimation quote. The certification process will need our toll cartridge manufacture to provide component information as next step.

4.5 Fouling Assessment of Groundwater

The four different filter series evaluated for clean water testing were subjected to stimulant groundwater for 72 hours, with initial and final feeds, concentrate, and permeate samples for chemical analysis. The initial feeds varied for all series as well as intra series chemical analysis leading to variance in percent reduction of certain chemicals amongst the filter series in Table 13 & 14. All filter series successfully removed over 97.0% of turbidity from the initial feed amount to the final permeate sample after 72-hours of groundwater stimulation. The MB-102 series removed the most turbidity with a 99.93% reduction, while 98.94%, 98.92%, and 97.98% for MB-104, MB-101, and MB-103 respectively. Upon completion of testing rapid initial fouling of the filter series was analyzed with the slope of the normalized flux for the first five hours of the testing, which resulted in MB-103 producing a 36% and a 31% decrease in slope compared to MB-101 and MB-102. The MB-102 series was the second best in rapid initial fouling, but also produced the highest normalized flux at the beginning (61.75 gal/ft^2/day) and at the end of the 72-hour test with (48.78 gal/ft^2/day). MB-103 had the lowest reduction in normalized flux of 13.16% but was also the second lowest initial normalized flux and MB-102 had a 21.00% reduction in normalized flux. From the groundwater stimulant study 21-MB-102 performed the best with respect to normalized flux for the entirety of the test and 21-MB-104 performed best with overall fouling with a 71.82% decrease in slope compared to 21-MB-101. From the chemical analysis, normalized flux, rapid initial fouling, and rate of fouling from the groundwater stimulant over 72 hours the MB-102 and MB-104 series performed the best compared to the control filter series of MB-101. As in previous sections these two filter designs both utilized the Int feed spacers which prove to be superior to the other feed spacers in terms of fouling and minimization of flux reduction. The MB-102 and MB-104 filter series will be utilized in future studies for Phase III of this project.

4.6 Skid Design

Skid design was conducted concurrently with filter design to maximize spiral wound membrane filters potential, reduce fouling, and maximize data acquisition of filters. To increase the filters potential pressure drops resulting in quicker fouling were reduced via upsizing piping, rerouting lines, and addition of a cooling coil to prevent a favorable environment for bacteria growth. The skid at PPG was upgraded by InnoH2O to reduce pressure drops as well as improving the data acquisition of the skid to resemble the GVSC skid more closely. Upon completion of the upgrades to the skid at PPG performance testing of the system was conducted by utilizing the 21-MB-101 prototype filter, which was a scaled-up version of 10-MB-110 series filters. The performance testing of the upgraded system had permeated pressure decreased by 80% versus the older skid at PPG at 50 psi feed pressure and produced a normalized flux slightly higher than the skid at GVSC. Automated data acquisition of the skid was upgraded from the original AVANtech design to conduct full data acquisition for the entire system to improve data collection. The original skid from phase I at NDSU was also upgraded to perform more closely to the upgraded skid at PPG, most parts from the original skid were utilized in the fabrication of the upgraded skid shipped back to NDSU. The skid had the control system updated and added the capability of data acquisition to allow a more user-friendly system. The commissioning and training for the enhanced skid at NDSU was completed January 2023.

Appendix A – Groundwater Simulant Test Fouling

5-Hour Groundwater Simulant Test Initial Fouling

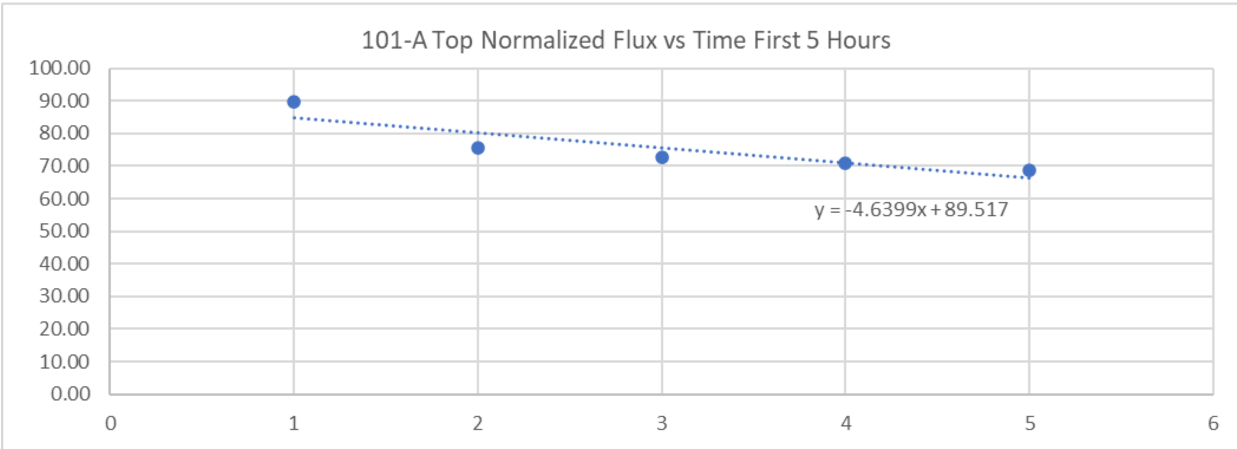


Figure A-1. Normalized Flux (gal/ft²/day) for First 5 Hours of Groundwater Testing for 21-MB-101A

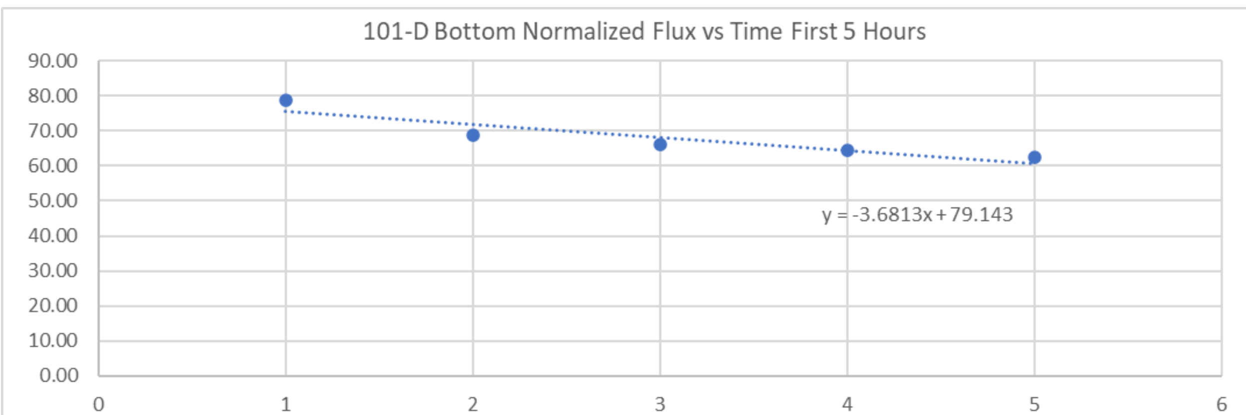


Figure A-2. Normalized Flux (gal/ft²/day) for First 5 Hours of Groundwater Testing for 21-MB-101D

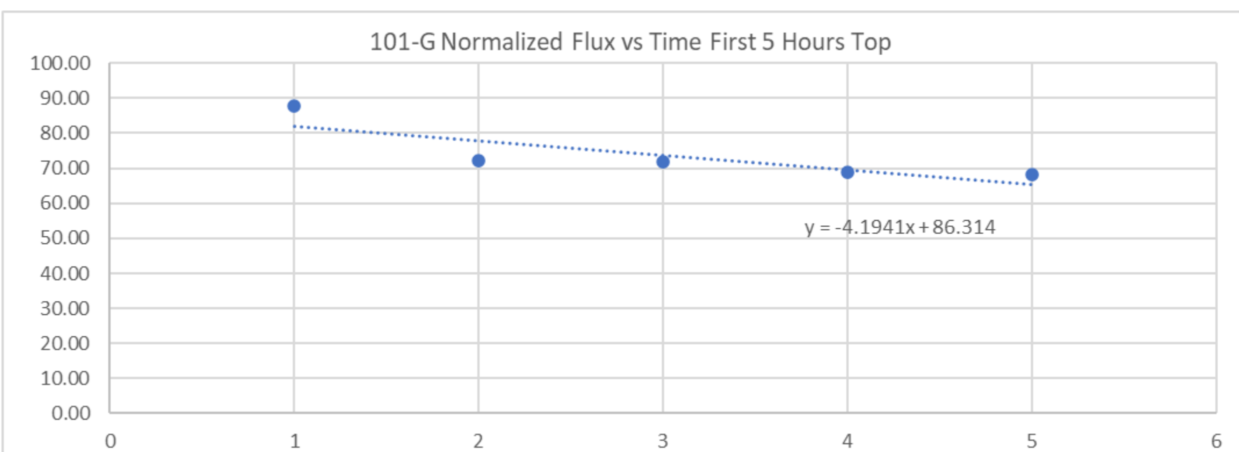


Figure A-3. Normalized Flux (gal/ft²/day) for First 5 Hours of Groundwater Testing for 21-MB-101G

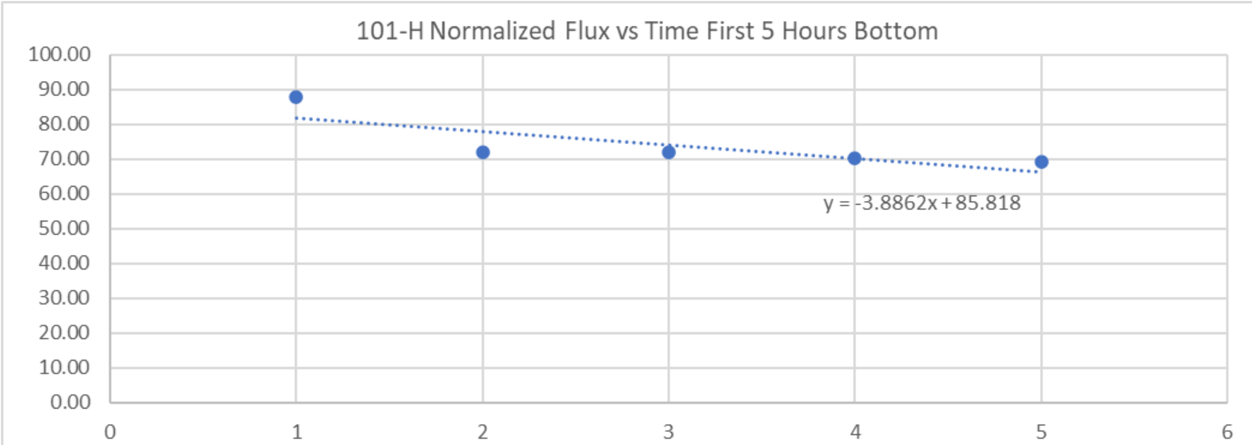


Figure A-4. Normalized Flux (gal/ft²/day) for First 5 Hours of Groundwater Testing for 21-MB-101G

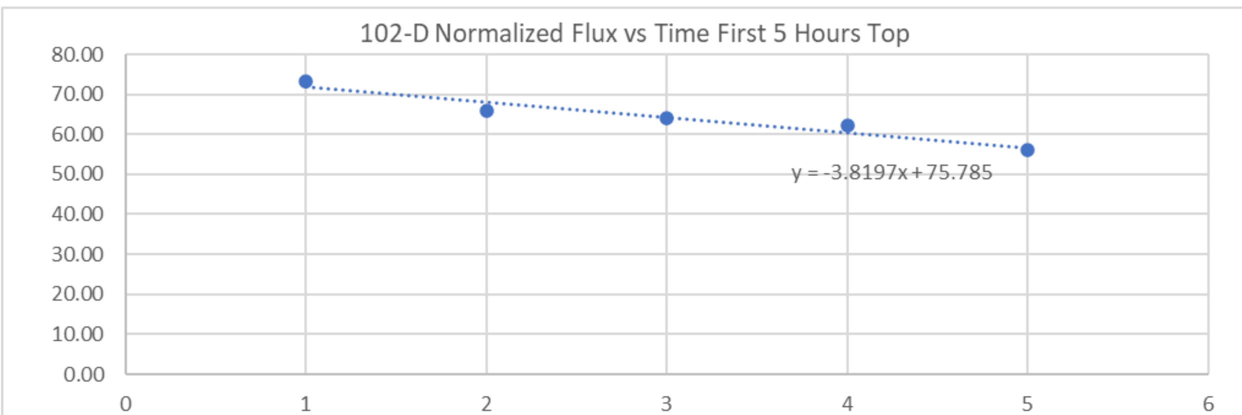


Figure A-5. Normalized Flux (gal/ft²/day) for First 5 Hours of Groundwater Testing for 21-MB-102D

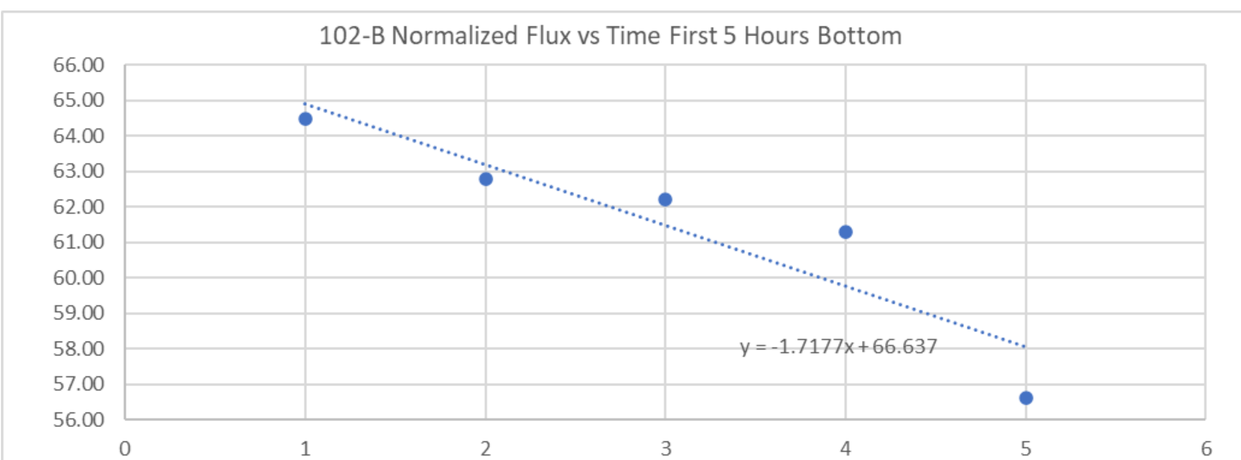


Figure A-6. Normalized Flux (gal/ft²/day) for First 5 Hours of Groundwater Testing for 21-MB-102B

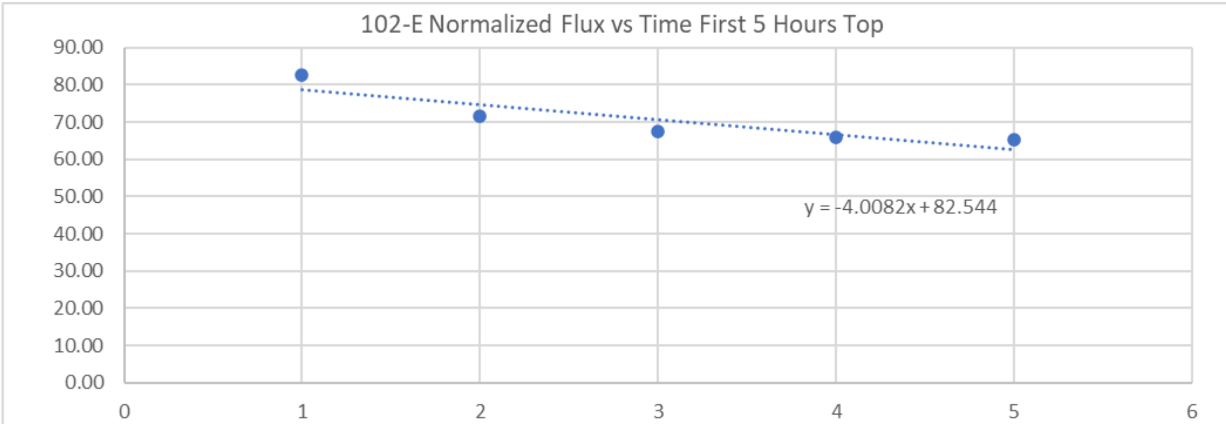


Figure A-7. Normalized Flux (gal/ft²/day) for First 5 Hours of Groundwater Testing for 21-MB-102E

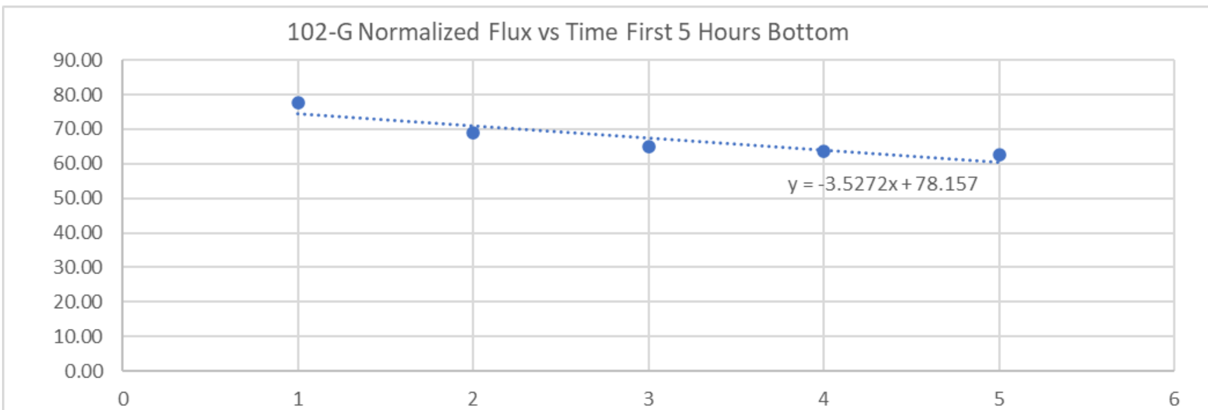


Figure A-8. Normalized Flux (gal/ft²/day) for First 5 Hours of Groundwater Testing for 21-MB-102G

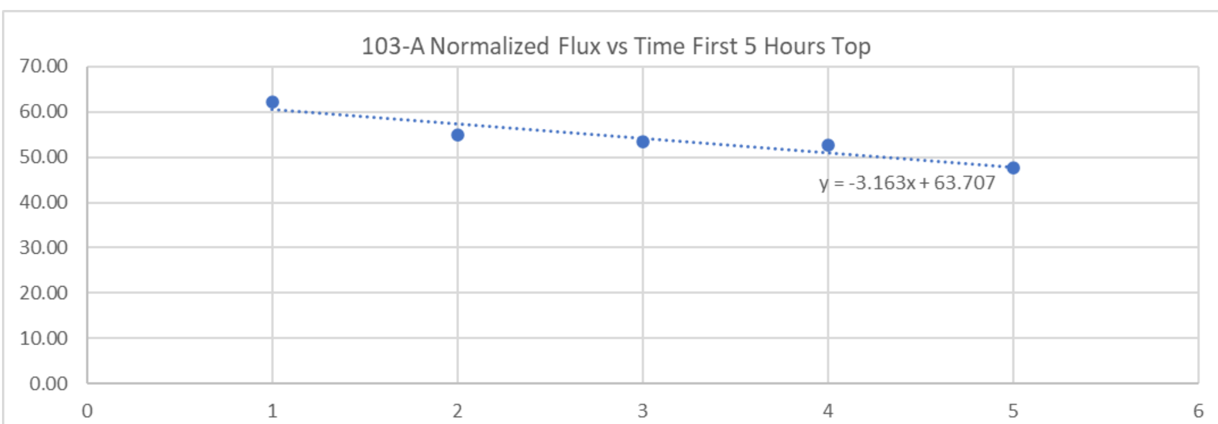


Figure A-9. Normalized Flux (gal/ft²/day) for First 5 Hours of Groundwater Testing for 21-MB-103A

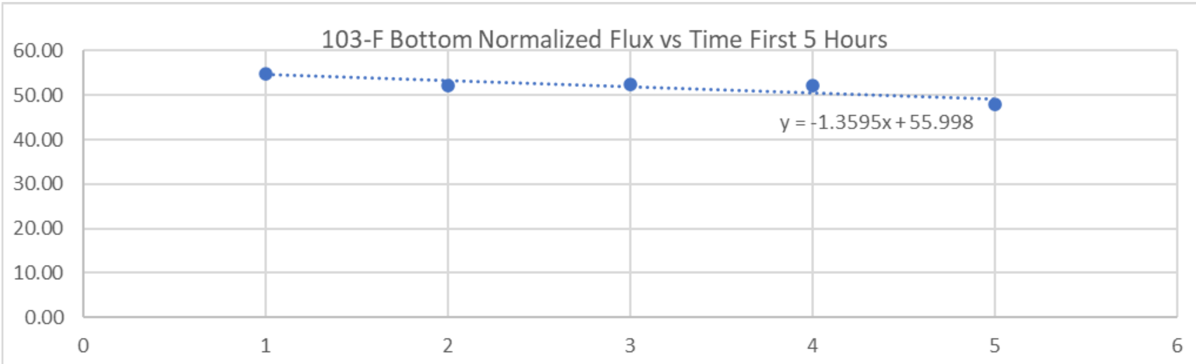


Figure A-10. Normalized Flux (gal/ft²/day) for First 5 Hours of Groundwater Testing for 21-MB-103F

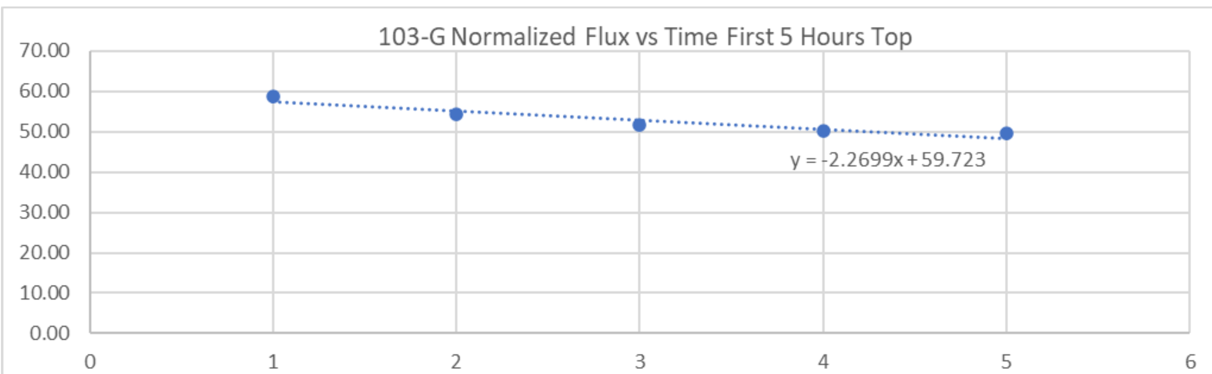


Figure A-11. Normalized Flux (gal/ft²/day) for First 5 Hours of Groundwater Testing for 21-MB-103G

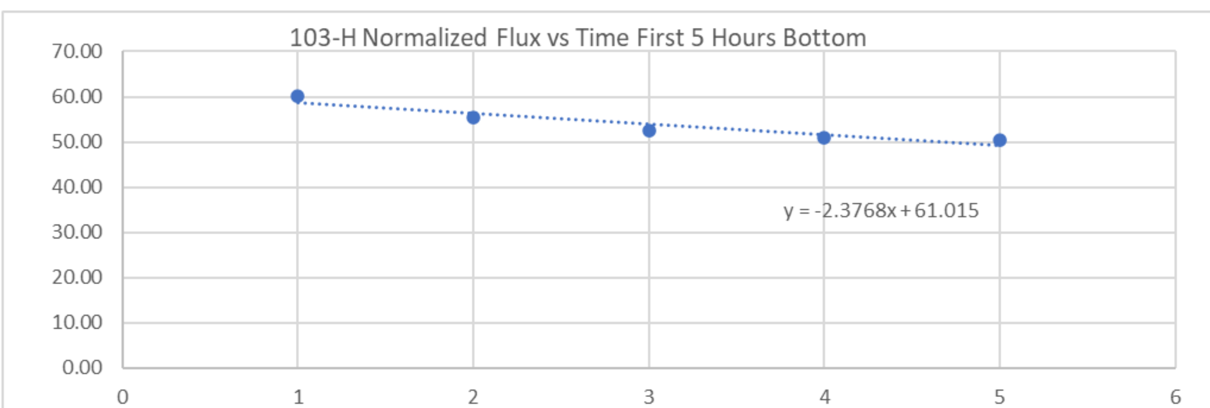


Figure A-12. Normalized Flux (gal/ft²/day) for First 5 Hours of Groundwater Testing for 21-MB-103H

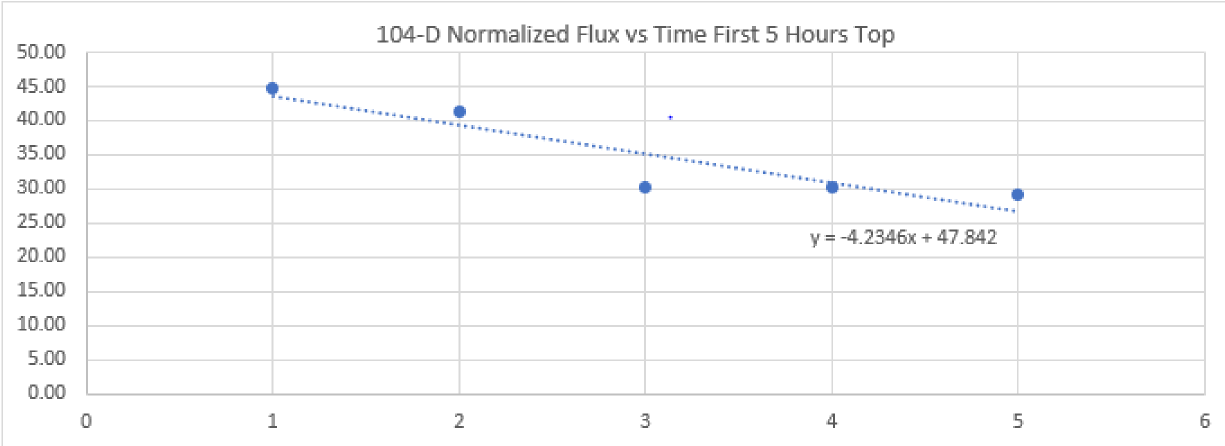


Figure A-13. Normalized Flux (gal/ft²/day) for First 5 Hours of Groundwater Testing for 21-MB-104D

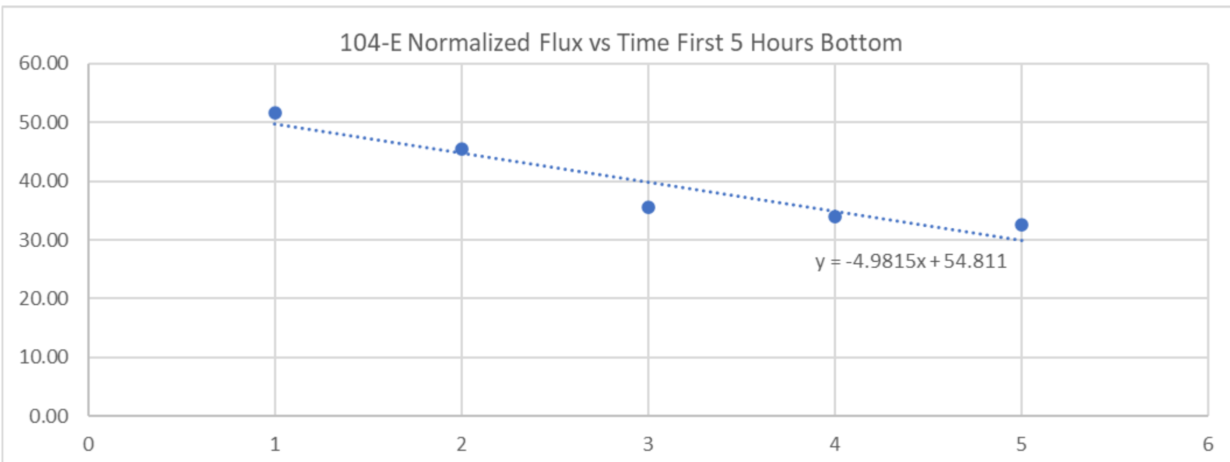


Figure A-14. Normalized Flux (gal/ft²/day) for First 5 Hours of Groundwater Testing for 21-MB-104E

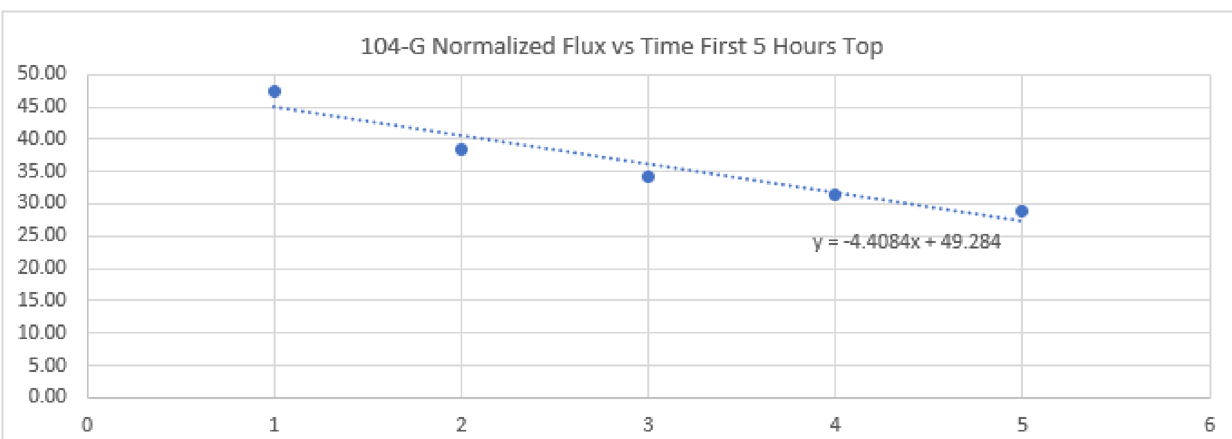


Figure A-15. Normalized Flux (gal/ft²/day) for First 5 Hours of Groundwater Testing for 21-MB-104G

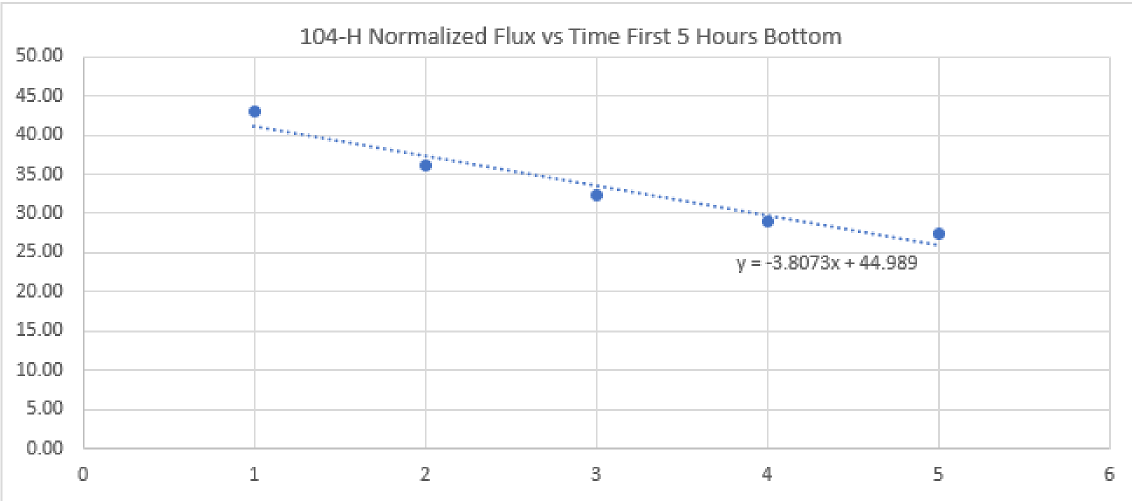


Figure A-16. Normalized Flux (gal/ft²/day) for First 5 Hours of Groundwater Testing for 21-MB-104H

67-Hour Groundwater Simulant Test Fouling

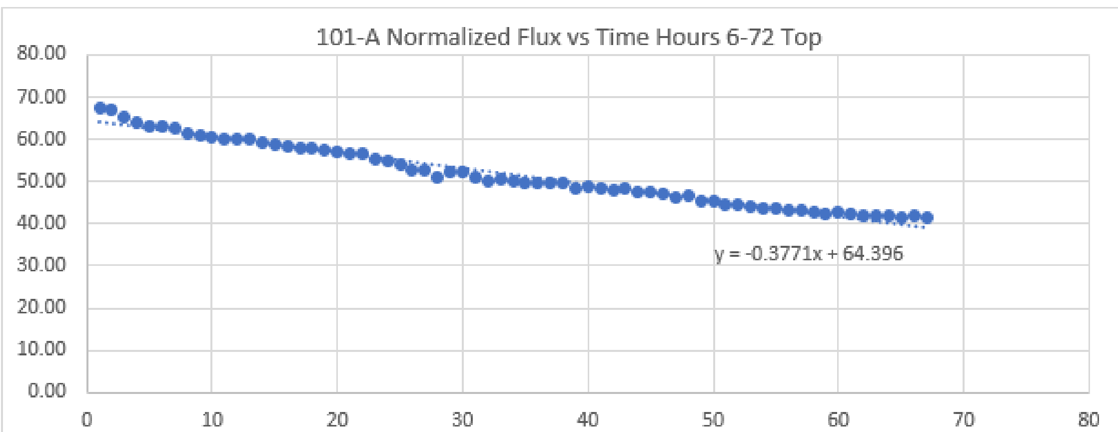


Figure A-17. Normalized Flux (gal/ft²/day) for Last 66 Hours of Groundwater Testing for 21-MB-101A

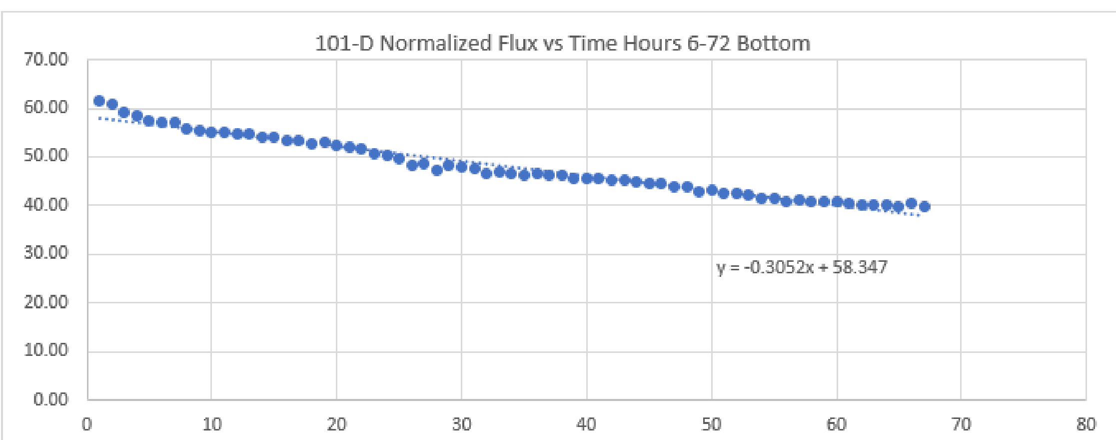


Figure A-18. Normalized Flux (gal/ft²/day) for Last 66 Hours of Groundwater Testing for 21-MB-101D

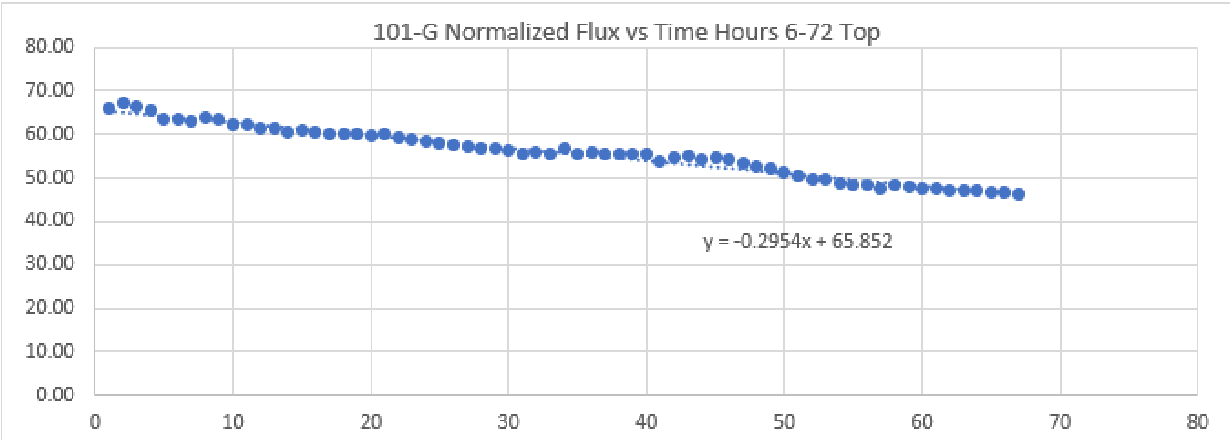


Figure A-19. Normalized Flux (gal/ft2/day) for Last 66 Hours of Groundwater Testing for 21-MB-101G

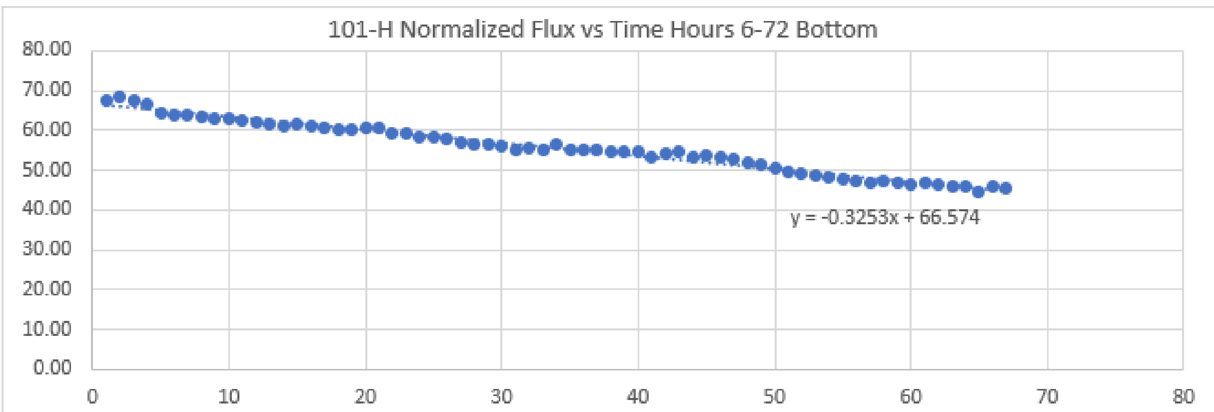


Figure A-20. Normalized Flux (gal/ft2/day) for Last 66 Hours of Groundwater Testing for 21-MB-101H

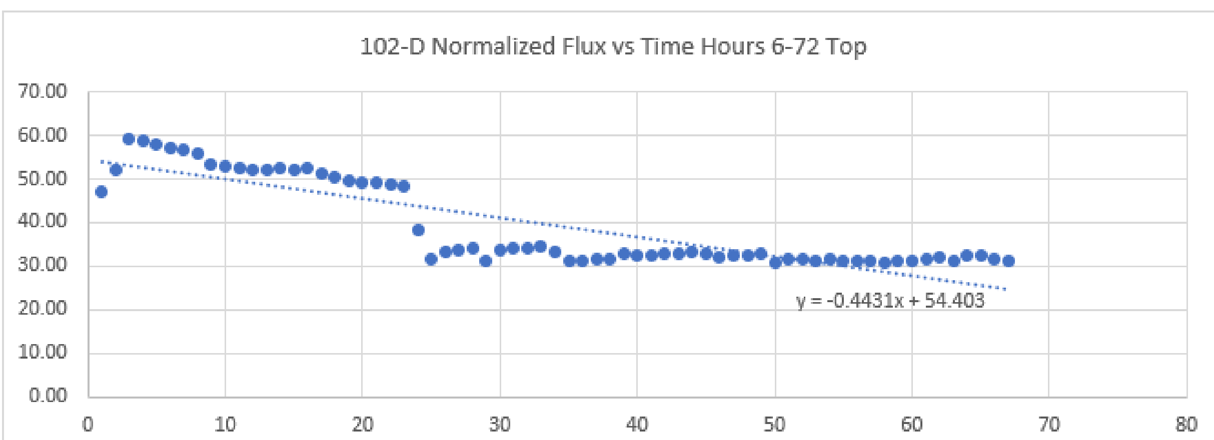


Figure A-21. Normalized Flux (gal/ft2/day) for Last 66 Hours of Groundwater Testing for 21-MB-102D

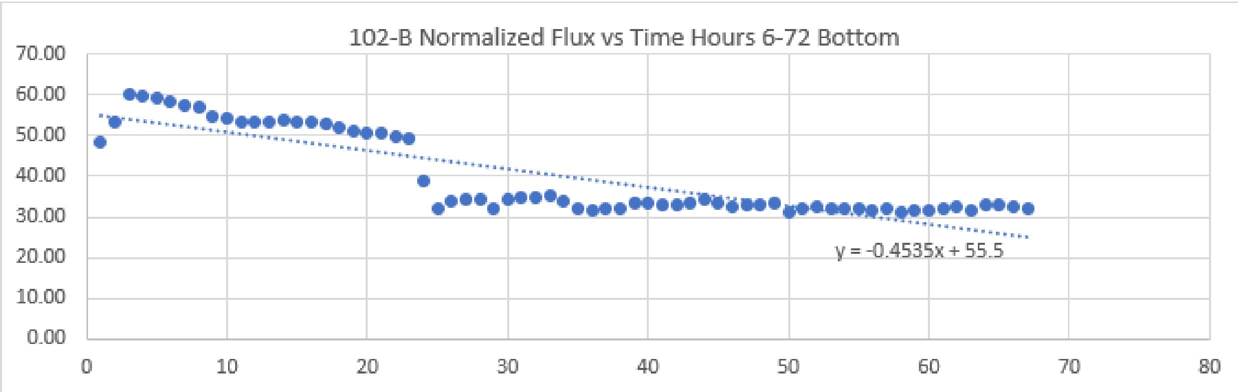


Figure A-22. Normalized Flux (gal/ft2/day) for Last 66 Hours of Groundwater Testing for 21-MB-102B

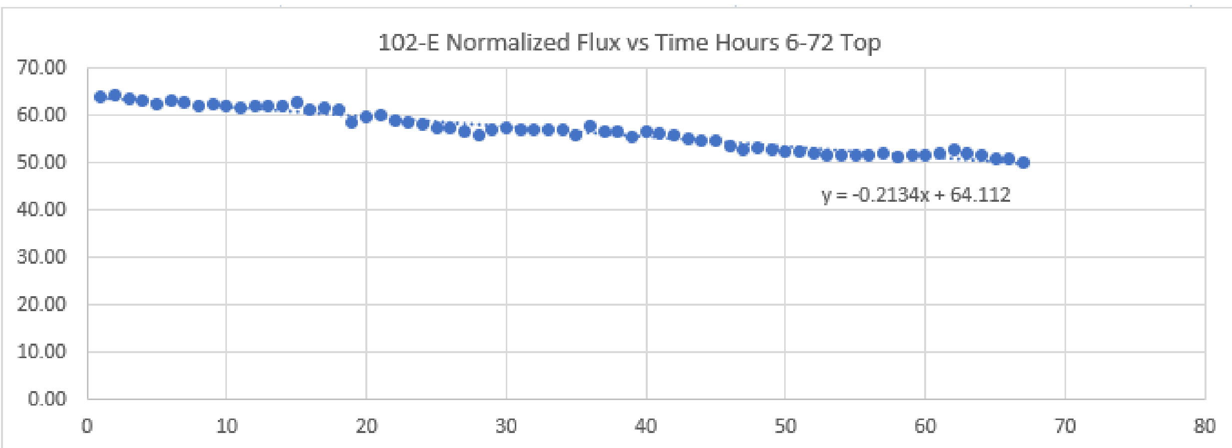


Figure A-23. Normalized Flux (gal/ft2/day) for Last 66 Hours of Groundwater Testing for 21-MB-102E

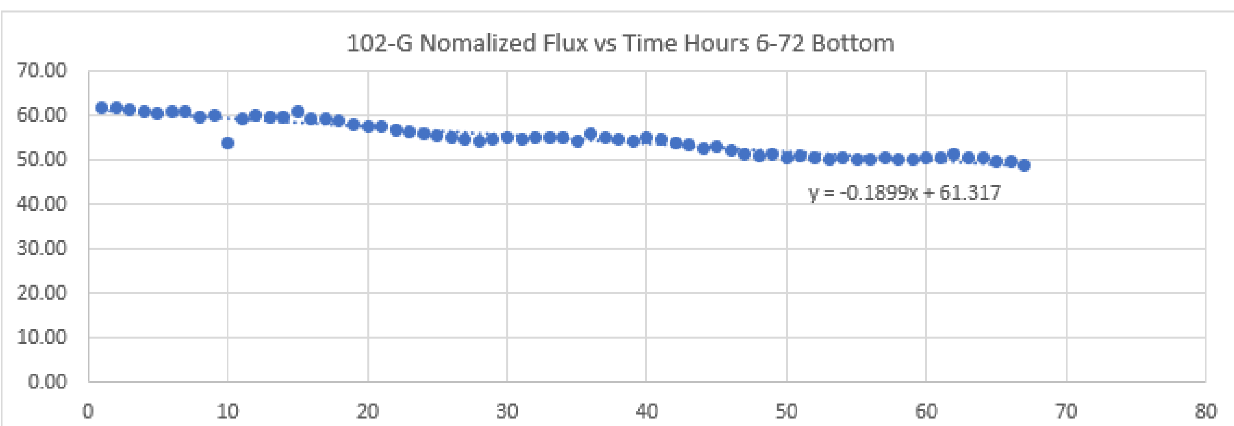


Figure A-24. Normalized Flux (gal/ft2/day) for Last 66 Hours of Groundwater Testing for 21-MB-102G

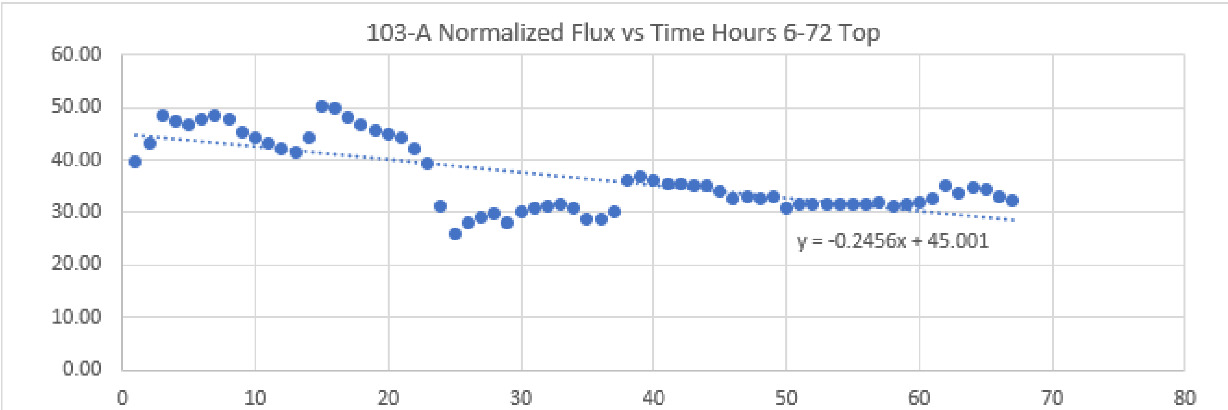


Figure A-25. Normalized Flux (gal/ft²/day) for Last 66 Hours of Groundwater Testing for 21-MB-103A

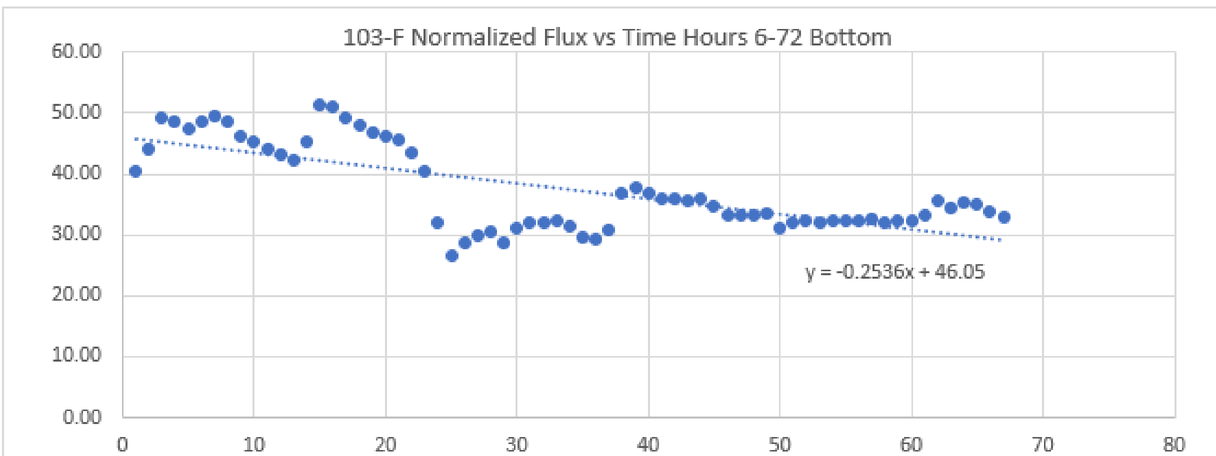


Figure A-26. Normalized Flux (gal/ft2/day) for Last 66 Hours of Groundwater Testing for 21-MB-103F

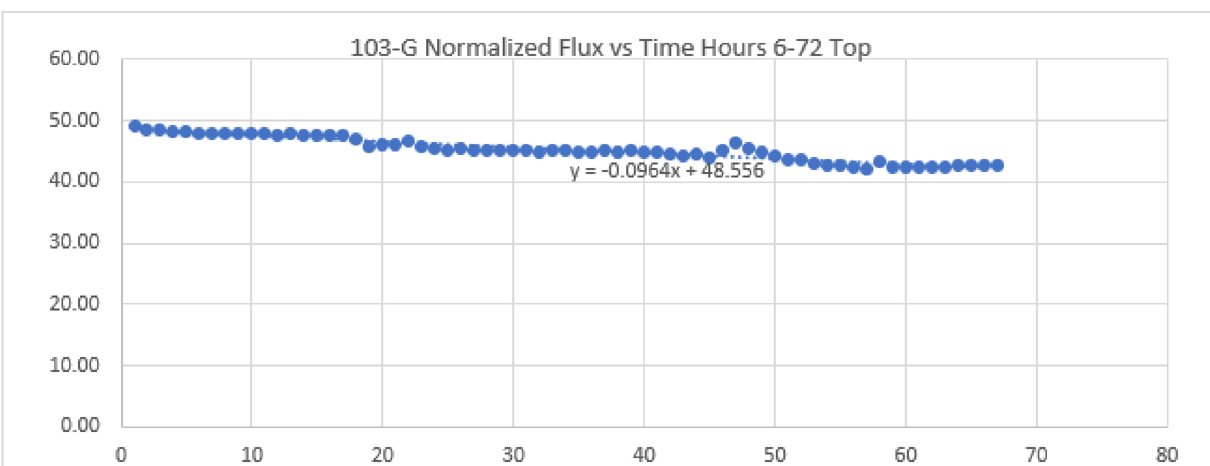


Figure A-27. Normalized Flux (gal/ft2/day) for Last 66 Hours of Groundwater Testing for 21-MB-103G

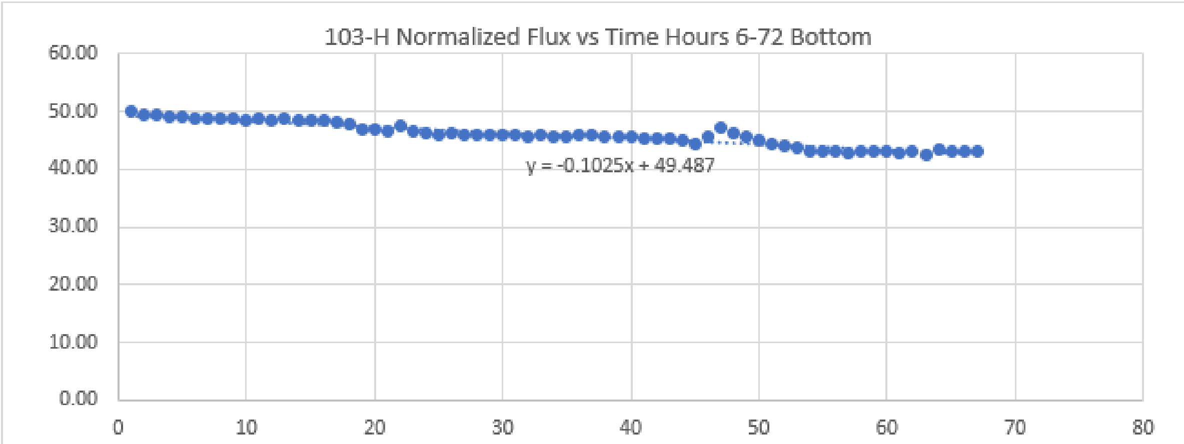


Figure A-28. Normalized Flux (gal/ft2/day) for Last 66 Hours of Groundwater Testing for 21-MB-103H

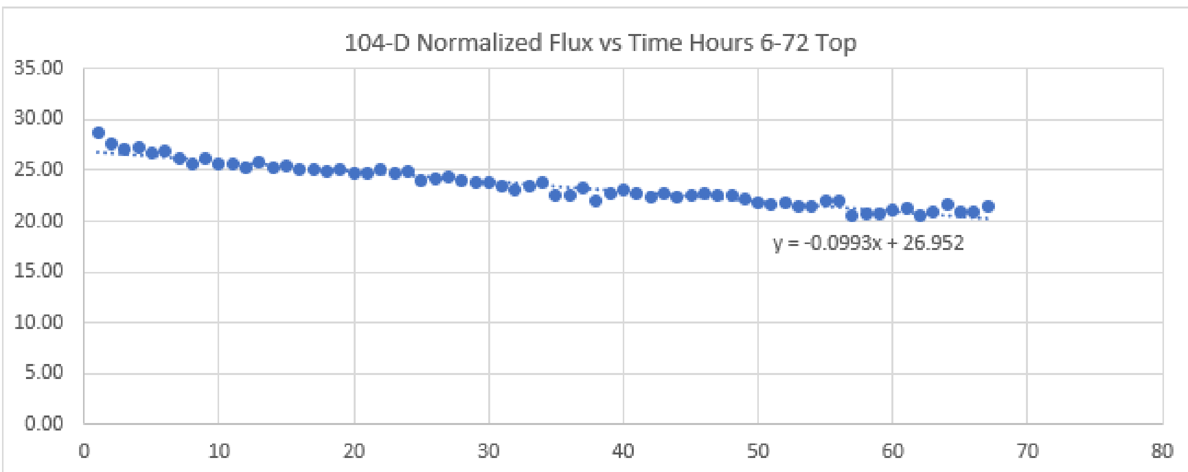


Figure A-29. Normalized Flux (gal/ft2/day) for Last 66 Hours of Groundwater Testing for 21-MB-104D

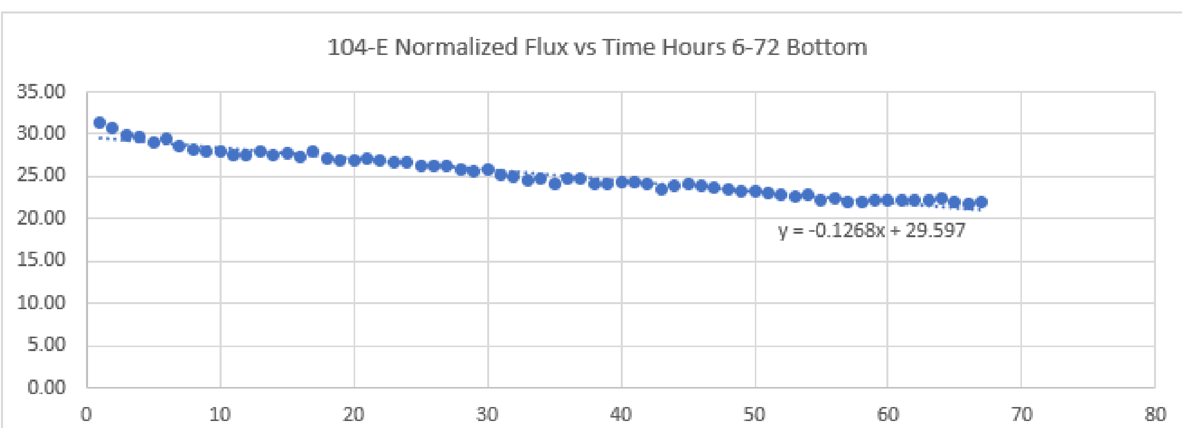


Figure A-30. Normalized flux (gal/ft²/day) for last 66 Hours of Groundwater testing for 21-MB-104E

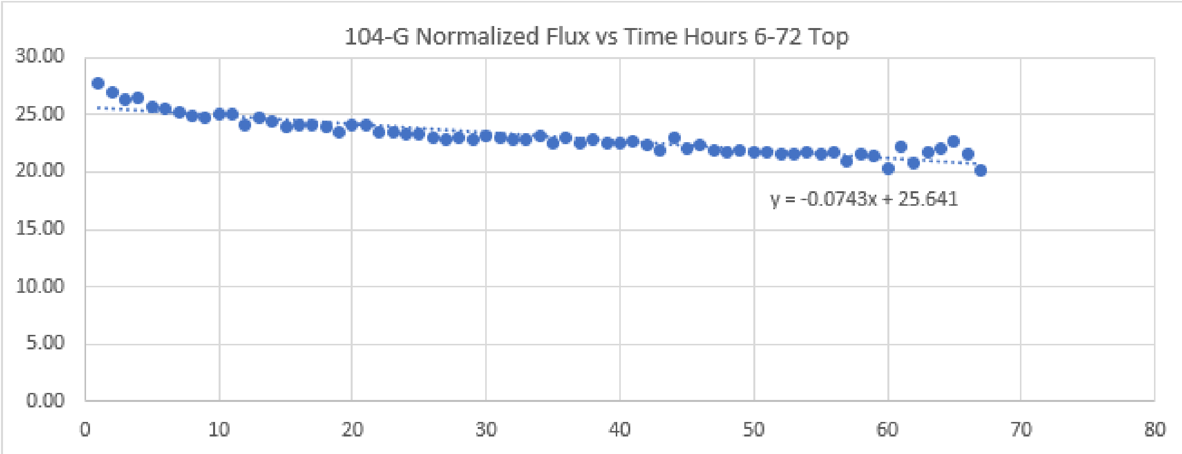


Figure A-31. Normalized Flux (gal/ft2/day) for Last 66 Hours of Groundwater Testing for 21-MB-104G

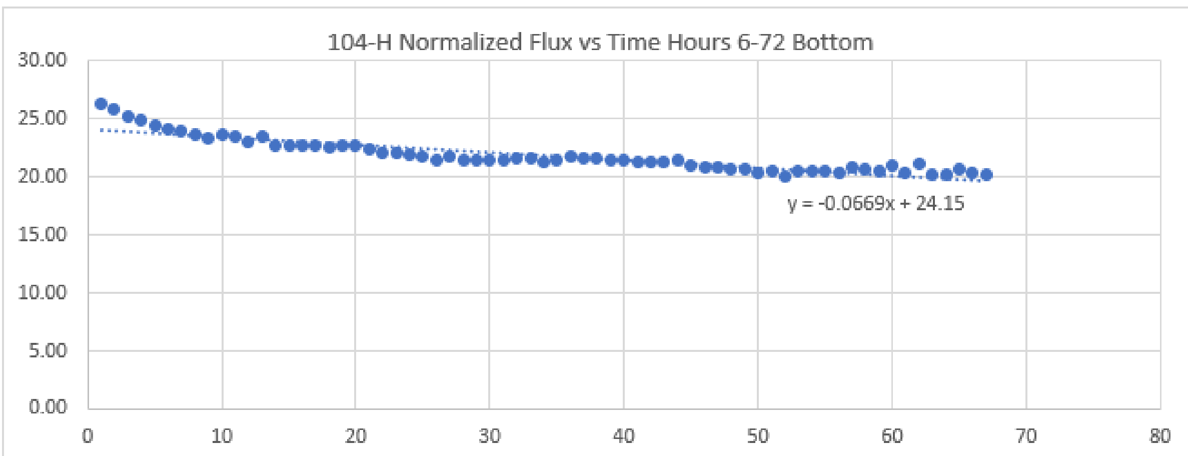


Figure A-32. Normalized Flux (gal/ft2/day) for Last 66 Hours of Groundwater Testing for 21-MB-104H