Reducing the Fouling of Microfiltration Membranes through
UV-initiated Surface Modifications using Poly (ethylene glycol)
Based Monomers

A Senior Project

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By

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Abstract
Meissner Filtration Product Inc. designs and manufactures membranes for food and beverage, microelectronics, ultrapure chemicals, and pharmaceutical industries. Membrane filters utilized in these industries have lifetimes greatly limited due to membrane fouling. Increasing membrane lifetime will reduce downtime, waste, and cost of operation. The proposed project is aimed to develop a methodology for synthesizing a fouling-resistant polymer coating layer on hydrophobic membrane surfaces for reducing membrane fouling and increasing membrane filterability (membrane lifetime) that are critical for effective membrane operations in the pharmaceutical industry. In this study, commercial membranes were coated with a solution containing a monomer, crosslinker, and photoinitiator dissolved in isopropyl alcohol. The coating was then synthesized by UV-initiated graft polymerization using SpeedCure 2022 photoinitiator and a 365nm UV lamp. Both the control and modified membranes were characterized using Attenuated Total Reflectance - Fourier Transform Infrared Spectroscopy (ATR-FTIR), Scanning Electron Microscopy (SEM), goniometry, water flux tests, and Ovaltine fouling tests in order to confirm the presence and evaluate the effectiveness of the fouling resistant layer on the membrane surface. Characterization of modified membranes reveals that the coating is sufficiently bonded and decreases the porosity of the membrane. The data collected from the above tests suggests that increasing monomer vol% increases anti-fouling properties whereas crosslinker vol% is best maintained at 1-2%. Performing successful fouling tests on modified membranes after 3 water flux tests suggests the fouling resistant coating is robust. Surface modified membranes showed increased operating lifetime by up to 80% relative to base membrane. As the coating both stays on the membrane and increases time to foul, the efficacy of PEG grafting onto membranes is proven.

Keywords: Membrane, Poly (ethylene glycol), PEG, Anti-fouling, UV, monomer, FTIR, SEM, Goniometry, Contact angle, Flux, Fouling, Crosslink
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1. Introduction

1.1 Meissner Filtration Product Inc.

Meissner Filtration Product Inc. Products Inc. is a biotech company that develops, manufactures, and supplies advanced microfiltration products and single-use systems worldwide. Their filtration products are critical components in the Pharmaceutical, bioprocessing, microelectronics, food and beverage, industrial, and chemical industries.

1.2 Problem Statement

With the growing rise of the pharmaceutical industry, alternative sterilization processes are an urgent necessity. Current projections estimate that the worldwide sales of prescription drugs are expected to reach 1.8 trillion US by the year 2024, a compound annual growth rate of 6.9 percent [1]. As a result of this, the demand for efficient and effective solution sterilization methods have grown in conjunction. Sterilization is the process of removing organic and inorganic particles from a surface or solution to be suitable for different applications [2]. Popular sterilization methods used are heat, irradiation, chemical, and filtration. One that stands out is membrane filtration. Research and development into this technique has shown it to be a promising candidate that can fulfill the needs of the pharmaceutical industry. Membrane filtration has shown to be an energy efficient process that produces a sizable increase in yield of high-quality products compared to conventional techniques, resulting in a process that is more economical, environmentally friendly, and safer. One unique characteristic of membrane filtration is that the process does not deteriorate desired biological materials unlike other methods that use heat which denatures them.

2. Background

2.1 Membrane Science

Microfiltration is a pressure driven process with pores in the range of 0.1 to 10 μm, allowing it to filter out different particulates of varying sizes. Due to a combination of their economic value and widespread availability, membranes are regarded as critical components for chemical and biochemical processing. Recent advancements in membrane technology have enhanced chemical, thermal, and mechanical performance enabling it to be suitable for industrial applications. Membranes possess the ability to operate in various conditions like high
temperature, high pressure, and corrosive environments, making it an enticing option for many applications [3]. Below in Figure 1 is a graphic depicting the versatility of membranes through differing pore sizes.

![Figure 1. There are four common filtration types: microfiltration, ultrafiltration, nanofiltration, and reverse osmosis. Each have vastly different pore sizes to filter out different particles. [4]

Despite this, membranes suffer from the buildup of pollutants and microorganisms on the surface, causing pores to plug up and decrease the overall efficiency and productivity of the membrane. This phenomenon is known as membrane fouling.

2.2 Fouling

Fouling is a process where particles are deposited on a membrane surface or within pores through applications like reverse osmosis, ultrafiltration, nanofiltration, and microfiltration which can reduce the performance of the membrane by blocking pores and causing a sharp decline in permeate flux levels. Fouls can come from many diverse sources and impair the membrane through various mechanisms [5].

2.2.1 Types of Fouls

Fouls can be categorized into four main categories: organic, inorganic, colloidal, and biological. Organic fouls are comprised of dissolved components, usually proteins or
hydrophilic and hydrophobic materials that attached to the membrane by adsorption. Inorganic materials like iron, manganese, and silica are particulates that precipitate onto the membrane surface due to a pH change or oxidation. Colloidal foulants are inorganic or organic particles that can physically coat the membrane surface and block the pores, hindering the transport of the feed solution. Lastly, biological foulants are vegetative matters like algae or other microorganisms which can attach to the membrane and cause biofouling [5]. When membranes are used for pharmaceutical purposes, the main solution being sterilized is water-based, which contains organic and biological foulants that must be removed.

### 2.2.2 Fouling Mechanisms

There are several ways that foulants can reduce the permeate flux and stability of membranes. The most widely accepted models for fouling today are the Hermia mathematical models which categorize fouling into four main mechanisms, which are: Complete blocking, Standard blocking, Intermediate blocking, and Cake filtration [6]. Figure 2 gives a visual representation of the different mechanisms.

![Figure 2. The four main ways foulants can accumulate on the surface of a membrane are (a) Complete pore blocking, (b) intermediate pore blocking, (c) cake filtration, and (d) Standard pore blocking [6].](image)

Complete pore blocking is where the foulant particles are deposited onto the unobstructed surface area of the membrane, but do not deposit on top of one another. This results in a complete obstruction of the pores. Standard blocking refers to blocking that develops only within the pores, reducing the overall pore diameter. Intermediate blocking is like Complete blocking but the foulants can build up on top of previously deposited particles. Lastly, cake filtration
occurs when foulant particles accumulate on top of already deposited particles, completely covering the membrane surface in several cake layers. This implies that the foulants do not obstruct the physical membrane pore area, but rather build on top of it. The overall filter mass transfer resistance increases in proportion to the cake layer thickness [6]. A combination of all fouling mechanisms can occur making membrane fouling a complex issue to prevent.

### 2.2.3 Economic Cost of Fouling

Multiple studies have confirmed that membrane fouling plays a significant role in adding to the operational costs of industrial filtration plants. Membrane costs are not just limited to the maintenance of them (physical or chemical cleanings), but also the cost to replace membranes and the productivity loss from fouling and down time. An economic investigation into these costs found that fouling contributed to 20-30% of total operational costs, with the biggest contributor to this being early membrane replacement, followed by additional energy consumption costs [7]. Figure 3 is a flowchart denoting all the operational costs associated with membrane filters.

![Figure 3](image.png)

Figure 3. All the associated operational costs of membrane filtration. The grey box below are all the costs due to fouling [7].
2.3 Ways to Treat Fouling

Membrane fouling can be classified into two different subgroups: reversible fouling and irreversible fouling. The type that occurs is dependent on the context in which the membranes are operated in and cleaned. Reversible fouling can be removed through physical means, while irreversible fouling can only be removed chemically.

2.3.1 Physical Removal

Reversible fouling is caused due to a buildup of foulants on a membrane surface. When this occurs, there are multiple physical methods that can be utilized to remove them. Physical cleaning methods rely on using mechanical force to dislodge and remove foulants from the surface. One method commonly used is backflushing. During backflushing, the flow of the membrane is reversed from the permeate side to the feed side. This is effective in removing cake layer foulants. Another popular cleaning method is membrane relaxation. Relaxation is known as a temporary pause of the feed solution, allowing foulants to move away from the membrane surface via concentration gradient. Intermittent relaxation is also utilized to allow for stable operation for a longer period of time until it can be thoroughly cleaned. [8].

2.3.2 Chemical Removal

Irreversible fouling is fouling that occurs within the membrane pores. Some examples of chemical agents used are hydrochloric acid, chlorine bleach, and hydrogen peroxides. The membrane is soaked in these solutions for several minutes and then flushed out, causing the foulants to be rinsed out with it [9].

2.3.3 Membrane Replacement

Once all the previous options have been attempted and the membrane is still not functioning within acceptable standards, the filter must be replaced. This solution as said before is one of the highest costs of a membrane filtration system as the cost to replace the membrane is coupled with the cost of production downtime.

2.3.4 Surface Modification

In order to reduce capital and operational costs, researchers have been investigating the viability of using surface chemistry to deposit a coating onto filtration membranes. This coating gives membranes inherent anti-fouling properties which would increase membrane lifetime and
productivity. To achieve this, many scientists have attempted to graft hydrophilic monomers onto membranes. A prime candidate among these monomers is poly (ethylene glycol).

### 2.4 Poly (ethylene glycol)

The monomers that will be used are a combination of monofunctional/difunctional methacrylate or acrylate terminated poly (ethylene glycol) (PEG) chains. PEG was chosen for two main reasons. First, PEG is well researched, and FDA approved for a wide variety of over-the-counter medicines and medical applications. Second, and more importantly, PEG forms a “hydration shell” [10]. The oxygens present along the PEG chain can hydrogen bond to water molecules and form a layer of water that can repel proteins. Figure 4 displays this phenomenon well. Mono or di-functional refers to the number of functional groups present at the ends of a chemical. For example, a difunctional methacrylate PEG chain will have methacrylate groups on both ends. The difference between the monomers chosen can be seen in Figure 5. The acrylate and methacrylate functional groups are specifically chosen because they react with UV radiation to form free radicals. With free radicals on the surface of the membrane and the PEG chain, free radical polymerization grafting will occur.

![Figure 4. A graphical interpretation of how PEG acts as an anti-foulant [10].](image)

![Figure 5. (a) Monofunctional PEG (b) Difunctional PEG [11][12].](image)

### 2.5 Grafting

Grafting is the process of adding polymer chains to either a polymer backbone or themselves to impart specific properties or structure. There are three main types of grafting: graft through,
graft to, and graft from. Grafting through is when individual monomers are grafted together with the help of an initiator. The initiator modifies the terminating functional group and allows them to bond in sequence producing a chain. Grafting to is when preformed polymer chains are “grafted to” a base polymer chain through a chemical reaction. The end of the polymer chain covalently bonds to the surface of the membrane. Grafting from is when a surface tethered polymer chain grows from the addition of monomers to the initiation site. [13]. A graphic showing the graft types is seen in Figure 6. The process of grafting, regardless of the method used, improves the anti-fouling properties of membranes as the foulant will interact with the modified surface rather than the membrane itself.

![Grafting Types](image)

Figure 6. The three main methods of grafting polymers. The purple chains are the grafted portions, and the red circles are initiation/reaction sites. [13].

2.5.1 Free Radical Polymerization

In free radical polymerization, a polymer is formed through the addition of free radical building blocks. The creation of these building blocks is facilitated through different mechanisms with their respective initiators. Some examples of free radical initiation are thermal decomposition, redox reactions, ionizing radiation, electrolysis, plasma, and UV irradiation [14]. In many of these cases, the initiator can degrade the membrane or is too costly for industrial applications. For example, thermal decomposition involves heat which can alter the membrane and ionizing radiation or plasma have prohibitive energy costs. As it is relatively cheap, easily scalable to industry production, and does not damage the membrane, UV initiated free radical polymerization is the most suitable choice for membrane surface modifications.
2.5.2 UV-Initiated Graft Polymerization

UV radiation produces free radicals by energizing molecules until bonds break. Photoinitiators are chemicals which absorb UV light and produce free radicals. The process of UV polymerization is detailed in Figure 7.

![Step 1: Initiation](image)

**Step 1: Initiation**

**Step 2: Polymerization with different monomers**

![Figure 7](image)

Figure 7. The process of UV polymerization with HEMA instead of PEG. The methacrylate group is what allows the polymerization to occur [15].

2.5.3 UV Initiator Chemistry

Membrane A will be modified through UV exposure which creates free radicals on the surface of the membrane. This process will be accelerated with initiators that will absorb the UV radiation and chemically alter the surface of the membrane to begin photopolymerization propagation. These photoinitiators fall under two categories, Norrish type I and type II. Type I photoinitiators generally contain benzoyl groups which are attached to a carbonyl group. The carbonyl group absorbs the UV radiation and undergoes homolytic cleavage to produce a free radical. Type II photoinitiators simply absorb the UV radiation to form an excited molecule that then abstracts an electron or hydrogen from a donor molecule [16]. In this application, the abstraction produces a free radical on the membrane’s surface. The free radicals will then initiate polymer chain growth. For this experiment, a blend of type I photoinitiators was used.

2.6 Crosslinking

To stabilize the anti-fouling coating, a crosslinker must be used. During preliminary testing, it was seen that coatings formed without a crosslinker failed to remain hydrophobic for
longer than a few days. Once a crosslinker was added, the coating remained usable for months. For this experiment, both trimethylolpropane triacrylate (TMPTA) and difunctional PEG were used. The dedicated crosslinker chosen was as it has three acrylate groups that will react with UV light to crosslink the PEG monomers. In figure 8, the chemical structure TMPTA shows the three functional groups which enable crosslinking.

![Figure 8. TMPTA with three acrylate groups for crosslinking PEG chains [17].](image)

In difunctional PEG chains, both ends will have free radicals which allows for one or both ends to bond to the membrane or to another free radical site.

### 2.7 Research Question

Meissner Filtration Product Inc. Products Inc. is investigating the viability of using monomer solutions consisting of poly (ethylene glycol) to modify hydrophobic membrane surfaces through UV-initiated grafting in order to reduce membrane fouling and increase membrane lifetime.

### 3. Experimental Procedure

#### 3.1 Design of Experiment

A pilot test was conducted before samples that satisfied statistical significance were produced. The pilot test involved three different compositions with three different concentrations of monomer and solvent. If the samples passed a preliminary wetting test that proved they were hydrophilic, contact angle measurements would be conducted. Once pilot testing concluded, the compositions that passed the wetting test moved on to primary testing. Each composition had multiple concentration levels that were tested. Monomer concentrations chosen were 1, 5, and 10 vol%. Crosslinker concentrations were 1 and 2 vol%. Initiator concentration was held constant at
1 vol%. Each sample was UV polymerized with the same lamp parameters. The wavelength was 365nm, the intensity was 8.4 W/cm², and the time was 30 seconds. For each concentration, 3 replicates were made for characterization, testing, and verification of results.

### 3.2 Terminology

The naming convention used throughout the experiment will be explained in Table I.

<table>
<thead>
<tr>
<th>Term</th>
<th>Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Membrane A/B</td>
<td>Meissner membranes</td>
</tr>
<tr>
<td>CL</td>
<td>Crosslinker</td>
</tr>
<tr>
<td>MFCL</td>
<td>Monofunctional PEG + CL</td>
</tr>
<tr>
<td>DFCL</td>
<td>Difunctional PEG + CL</td>
</tr>
<tr>
<td>Combo</td>
<td>Monofunctional PEG + Difunctional PEG</td>
</tr>
<tr>
<td>#:#</td>
<td>Vol%:Vol% of chemical</td>
</tr>
</tbody>
</table>

An example using the terminology would be MFCL 10:1 which denotes monofunctional PEG 10 vol% with crosslinker 1 vol%.

### 4. Methodology

#### 4.1 Membrane Preparation

Preparation of membrane samples consists of three steps: solution preparation, membrane coating, and UV irradiation. A preliminary step of rinsing base membrane with IPA is not necessary but enhances graft polymerization success. The IPA will remove surface contaminants and evaporate most of the water, if any, trapped in the pores.

#### 4.1.1 Materials

Filtration membranes were supplied by Meissner Filtration Product Inc. The monomers used included Poly (ethylene glycol) dimethacrylate (Sigma-Aldrich), Poly (ethylene glycol) methyl ether methacrylate (Sigma-Aldrich). The photoinitiator used was Speedcure 2022 (Lambson). Trimethylolpropane triacrylate (Sigma-Aldrich) was the only crosslinker used.
4.1.2 Solution Synthesis

In a 50 mL beaker, pipette the proper amount of the monomer used in the composition being tested following the concentrations outlined in Table II. Then using a micropipette, add SpeedCure 2022 to the beaker following the outlined concentration amounts. Measure out the required amount of solvent (IPA) using a graduated cylinder and add it to the solution. This solution was stirred for 3 minutes using a stir bar until the monomer and initiator were dissolved and a homogeneous solution was produced.

<table>
<thead>
<tr>
<th>Composition</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>MFCL</td>
<td>10:2</td>
</tr>
<tr>
<td></td>
<td>10:1</td>
</tr>
<tr>
<td></td>
<td>1:1</td>
</tr>
<tr>
<td>DFCL</td>
<td>10:1</td>
</tr>
<tr>
<td></td>
<td>5:1</td>
</tr>
<tr>
<td></td>
<td>1:1</td>
</tr>
<tr>
<td>Combo (MF + DF)</td>
<td>5:5</td>
</tr>
<tr>
<td></td>
<td>2.5:2.5</td>
</tr>
<tr>
<td></td>
<td>1:1</td>
</tr>
</tbody>
</table>

4.1.3 Membrane Coating

After the solution was produced, a transfer pipette was used to coat the membrane with 2 ml of solution until fully wetted out. Then the membrane was transferred to the UV lamp.

4.1.4 Irradiation

Each membrane sample was irradiated using the UV lamp with a wavelength of 365 nm, working distance of 10mm, and an intensity of 8.4 W/cm². The illumination area is marked with a non-conductive marker and the sample is placed within the area. The irradiation time used for all samples was 30 seconds. A simple graphic showing the general irradiation procedure is shown in Figure 9. After irradiation, the samples were rinsed with IPA and then left to dry. Once dry, a 47mm hole punch was used to punch out multiple samples for testing.
4.1.5 Safety

Two major sources of danger in this experiment are chemical handling and UV light. To minimize risk involved with chemical handling, standard personal protective equipment (goggles, long pants, closed toed shoes, nitrile gloves) were always worn. Additionally, all chemical mixing and preparation were performed inside the fume hood. UV light exposure was limited by sealing the UV lamp where any UV light could possibly leak. To protect the most sensitive parts of the body from UV light, ANSI Z87.1 certified UV glasses and face shields were worn whenever the lamp was powered.

4.2 Characterization

To confirm the presence of the PEG coating on the membrane surface, characterization of the membranes using goniometry, FTIR, and SEM imaging were carried out. Each involved comparisons between modified and unmodified membranes.

4.2.1 Goniometry

The hydrophilicity of the membrane top surface was characterized based on water contact angle measurements (VCA-2500XE) in Figure 10 equipped with video capture at room temperature. The membrane was rinsed with isopropyl alcohol and dried completely in ambient air. A total of 2.25 μL of de-ionized water was dropped onto the membrane surface with a micro-syringe in an atmosphere of saturated water vapor and the size of the drip was captured. 5 contact angles were averaged to get a reliable value. By analyzing the spread of the water drop on the surface of the sample, the angle between the surface and droplet formed will allow surface
energy to be calculated, verifying whether the sample is hydrophobic or hydrophilic. Hydrophobic samples have water contact angles greater than or equal to 90 degrees and hydrophilic samples are less than 90 degrees.

![Goniometer used for contact angle measurements](image)

**Figure 10.** Goniometer used for contact angle measurements [18].

### 4.2.2 Attenuated Total Reflectance Fourier-transform Infrared Spectroscopy

Attenuated Total Reflectance Fourier Transform InfraRed Spectroscopy (ATR-FTIR) was used to assess the chemical compositions of the surface-modified membranes after UV grafting, qualitatively confirming the presence of new functional groups from the monofunctional and difunctional PEG monomers. ATR-FTIR spectroscopy was carried out on a Nicolet iS10 spectrometer shown in Figure 11. ATR spectra were collected using a frequency range of 4000–400 cm\(^{-1}\) at a 4 cm\(^{-1}\) resolution. To obtain a high signal-to-noise ratio, 32 scans were performed for each sample.
4.2.3 Scanning Electron Microscopy

SEM images were taken using a FEI Quanta 200. Before imaging, the samples were sputter-coated with Au-Pd on a Cressington Sputter Coater 108auto seen in Figure 12 to produce a conductive surface that would allow for better imaging. This was conducted to determine if the coating was successfully applied and allowed for the observation of foulants on the membrane. In addition, SEM allowed for morphological observation of the membrane surface and the ability to view the surface porosity. Samples were sputter-coated with gold to form a conductive surface. Images were taken at 4000x with an accelerating voltage of 5kV, a spot size of 4.0, and a working distance of 10.0 mm.
4.3 Performance Testing

4.3.1 Flux Test

To measure anti-fouling properties, flux tests were conducted to quantitatively determine the effectiveness of the coating in allowing water to pass through while repelling various foulants. Flux testing was performed using ASME-Code Pressurized Liquid Dispensing Tank purchased from McMaster Carr (Part #41705K39) and a 47mm polycarbonate membrane holder from Pall. An image of the testing apparatus is depicted in Figure 13. The discs were first tested using 1L of deionized water with a pressure of 10 psi. The water was captured in a graduated cylinder and the mass was recorded with a scale every 30 seconds for 3 minutes.

4.3.2 Fouling Test

To perform fouling tests, 0.1g of Ovaltine in 1L of deionized water was prepared. The membrane fouling test was performed at 10 psi and the mass was recorded with a scale every 30 seconds for 5 minutes. The test was stopped once a flow rate below 10 g/min was recorded 3 consecutive times. This was repeated for the other 3 replicates of that concentration. Due to time constraints, batch testing was not feasible. Batch testing would isolate the errors to each batch and provide more conclusive data over the course of testing all the samples. The data for both flux and fouling tests were averaged and plotted to observe trends.
5. Results

5.1 Characterization

5.1.1 Goniometry

For every membrane that was produced 5 contact angle measurements were taken in random areas of the membrane and then averaged. In all successfully modified membranes, a hydrophilic contact angle was observed. For example, MFCL 10:1 showed an average contact angle of 32 degrees. Values ranging from 18 to 64 degrees were seen in the modified membranes. In comparison, the base membrane produced contact angle values around 120 degrees. As seen in Figure 14 the difference between unmodified and modified is evident.

![Image showing the drastic difference in hydrophilicity of modified vs. unmodified](image)

Figure 14. Images showing the drastic difference in hydrophilicity of modified vs. unmodified.

5.1.2 FTIR

The base membrane and one of each modified membrane’s spectra were plotted on the same graph as shown in Figure 15. From this, 3 major peak changes to the base membrane can be seen. The first two changes are a result of the PEG chain. At ~2840 cm\(^{-1}\) -C-H stretching peaks are seen and in the fingerprint region broad peak and intensity changes are visible. The final major peak change is at ~1720 cm\(^{-1}\) resulting from the carbonyl group.
Figure 15. FTIR spectra of all variations of membrane showing changes in peaks resulting from PEG grafting.

5.1.3 SEM

Images of unmodified, modified, and fouled samples reveal morphology changes at every step of the experiment. All modified membranes show decreased porosity in comparison to unmodified membranes. The extent of porosity change is dependent on concentration and monomer selection. Figure 16 displays images taken of membranes at separate phases.

Figure 16. (a) Unmodified membrane. (b) DFCL 10/1. (c) Combo 1/1. (d) Fouled DFCL 10/1.

5.2 Membrane Performance

5.2.1 Flux Test
The flowchart below in Figure 17 shows the average flow rate (g/min) of all the membranes. Averages were taken from 3 separate measurements of each membrane. Membrane A showed the highest average flowrate at 196.3 ml/min, followed by Membrane B at 56.2 g/min. The best performing surface modified membrane was Combo 1:1, with an average
flow rate of 47.1 g/min. For our Difunctional composition, the best performing one was DFCL 10:1 while for Monofunctional the best composition was MFCL 10:1.

![Bar chart of the average Flowrate (ml/min) of all membranes tested.](image)

Figure 17. Bar chart of the average Flowrate (ml/min) of all membranes tested.
5.2.2 Fouling Test

From the fouling tests performed, it was found that Membrane B was the best performing membrane, with an average time till fouled out at 19 minutes and an average volume filtered at that time being 414 grams of solution. This is followed by DFCL 10:1 being the highest performing modified membrane, lasting on average 18.5 minutes and filtering out 363 grams. Figure 18 is the total volume vs time of the membranes tested.

![Figure 18](image)

Figure 18. Scatterplot of averaged Total volume vs Time of fouling tests.

6. Discussion

6.1 Characterization Analysis

All the characterization methods used confirm that modifications of the base membrane have an effect. Contact angle measurements show that modifying the membrane will make it hydrophilic. This supports the phenomenon of PEG hydration shells and their use in anti-fouling. FTIR spectra and SEM images of the modified membranes reveal that UV grafting PEG is viable for permanent surface modification to impart anti-fouling properties. These results are supported by previous research and characterization methods. For example, the FTIR spectra of
monofunctional PEG produced by Sigma-Aldrich in Figure 19 further confirms that the PEG chains are successfully grafted onto the membrane surface.

Figure 19. Sigma-Aldrich provided FTIR spectrum of the PEG monomer used. Major peaks align with earlier FTIR results [11].

6.2 Performance Analysis

When comparing the flux data with the fouling data there are some general trends that are supported. The first trend seen in the data is that with increasing monomer percentage, average flow rate and average fouling performance increased as well. For the MFCL composition, increasing monomer percentage from 1% to 10% increased the permeate flowrate by 6% and increased the time to foul by 27%. Similarly, for DFCL, a positive linear trend was seen where performance increased from 1:1 to 5:1 and then 10:1 being the best performer for that composition. Combo, we see the opposite trend where increasing monomer percentage decreased overall membrane performance. This could be due to the concentration of crosslinker increasing along with monomer concentration since the difunctional monomer was used as an alternative crosslinker. Table III below shows the quantitative change in performance with monomer change.
Table III. Effects of Monomer Concentration Changes on Average Membrane Performance

<table>
<thead>
<tr>
<th>Concentration Change</th>
<th>Average Flow rate change (%)</th>
<th>Average Time to foul change (%)</th>
<th>Average Volume filtered change (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MFCL +9%</td>
<td>+6%</td>
<td>+27%</td>
<td>+36.6%</td>
</tr>
<tr>
<td>DFCL +9%</td>
<td>+7%</td>
<td>+68%</td>
<td>+91%</td>
</tr>
<tr>
<td>Combo +4%</td>
<td>-6%</td>
<td>-22%</td>
<td>-35%</td>
</tr>
</tbody>
</table>

While there were performance increases throughout all our membranes, the only membrane that came close to commercial standards was the DFCL 10:1. Membrane A while having the highest flow rate, fouled out the fastest out of all the samples. Membrane B on the other hand was the best performing membrane tested. DFCL 10:1 comes close to Membrane B, filtering out 88% of solution that Membrane B did and having a time to foul of 97% compared to Membrane B. This shows with coating solution optimization, PEG coatings have the potential to compete with industry level standards.

6.3 Concerns in Data

6.3.1 Reproducibility

In the membranes produced, variations were present in some data but not in others. Contact angle, for instance, had slight variation even throughout all samples of the same composition. On the other hand, some outliers were seen in fouling tests but did not affect the overall trend. More time and testing are necessary to address the concerns of reproducibility.

6.3.2 Process Control

Although measures were taken to control the process as much as possible, the equipment used is limited in accuracy and consistency. Some cases where this is evident are the micropipettes and UV lamp used. The micropipettes purchased were affordable and naturally their performance is more variable than state of the art micropipettes. Some volume concentrations may have been affected as the volume being pipetted is quite small. The UV lamp provided by Meissner was limited in parameter controls such as working distance and intensity. The intensity of the lamp was kept at maximum on the potentiometer to produce a somewhat
reliable intensity between all membranes. However, without more accurate power control there is no way to account for variations in lamp output.

6.3.3 Batch Test Variability

Outside of processing variability in the batches, testing between batches also carries inherent inconsistencies. For example, the time between membrane modification and testing may affect the results. Similarly, the testing solution of Ovaltine and distilled water may vary from day to day as the amount used and quality of the water have inherent variability.

7. Conclusion

1. UV-initiated PEG grafting can produce a permanent fouling-resistant coating on membrane filters.
2. PEG can turn a hydrophobic membrane into a hydrophilic membrane.
3. Increasing monomer concentration generally improves anti-fouling performance.

8. Future Works

A major point of interest which went uninvestigated due to time constraints is the effect of pore density on anti-fouling performance. This would involve pore density calculations on SEM images of modified membranes which is a time-consuming task. To further optimize the results and more clearly draw correlations between pore density and anti-fouling performance, increased chemical concentrations and individual testing should be carried out. From the limited number of concentrations tested so far, the general correlation between concentration and anti-fouling performance was established. However, pore density is an undocumented factor which could affect the trend. Similarly, batch testing the membranes limits the strength of the correlations which were drawn. If each membrane’s variables were logged and compared to their respective flux or fouling results, more conclusive relationships could be made. This was not performed during the experiment as not every membrane was tested, and the effort is time-consuming without the proper resources. In conclusion, further and more directed testing must be performed before conclusive claims are made.
9. Reference


