

QATAR UNIVERSITY

COLLEGE OF ENGINEERING

EXPERIMENTAL ASSESSMENT OF FORWARD OSMOSIS MEMBRANE SYSTEM FOR

SEPARATION OF SUSPENDED COLLOIDS

BY

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A Thesis Submitted to

the College of Engineering

in Partial Fulfillment of the Requirements for the Degree of

Masters of Science in Environmental Engineering

January 2020

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ABSTRACT

AHMED, MOHANNAD, H., Masters : January : 2020,

Masters of Science in Environmental Engineering

Title: Experimental Assessment of Forward Osmosis Membrane System for Separation of Suspended Colloids

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The formation of the stable suspended colloids is well known in many industrial applications, whether you are in the wastewater treatment, dairy, chemical refinery, or food processing and production industry. Always there is one common challenge everyone faces: Total Suspended Solids (TSS). These suspended colloids can be very stable and difficult to separate due to their small size (less than $2\mu\text{m}$). Many methods were tested for treating removing such colloidal particles. However, each method has its limitation in capability, depending on the condition and concentration of such colloidal particles. In this study, Forward Osmosis (FO) is used to remove such stable suspended particles without any flocculation and coagulation pretreatment. Forward Osmosis (FO) is an emerging technology in water treatment, which has several applications in different industries, and it has many advantages over other membrane processes. FO treats complex effluents with high rejection rate, has lower fouling/cake formation affinity, and relies on the difference in osmotic pressure across the membrane without the need of high-pressure aids which means minimum energy requirement. The utilization of FO in separating suspended colloids and its performance under different operating conditions. The results showed that

FO membrane significantly remove the suspended colloids without having permanent fouling. This study concluded that FO membrane is promising technology for removing stable colloidal particles from wastewater effluent.

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Chapter 1: INTRODUCTION

1.1 Water Consumption

1.1.1 Water Worldwide Consumption

Water is the most essential component for the human beings as it represents 70% of the body fluids which transfers nutrients and macromolecules through body. Also, water helps body to flush out toxins and boosts immune system to keep the body healthy and its energy at peak. In addition, water is a mandatory factor for agriculture, and it was clearly proven by improving the irrigation techniques “spray and drip irrigation”. According to United Nations, the most common used water resources in earth are oceans, seas, rivers, groundwater, which located in the sub surface of the earth. The extracted water from all these resources undergo purification processes, so it will be suitable for human use [1]. Fresh water has an important role in human’s life sustainability and it is quite essential for the individual’s health. However, having a fresh water supply all over the world cannot guarantee a quick access of clear water for almost 783 million persons in different continents, Figure 1, as well as it cannot guarantee a good proper health since it increases the percentage of water-related diseases, whether these diseases are caused by deficient water supply, sanitation or hygiene [2].

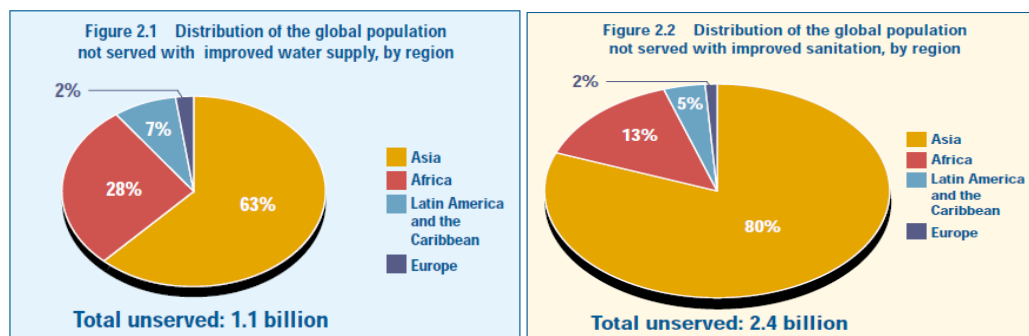


Figure 1. Distribution of population with no access to fresh water [2].

Water is the base of sustainable improvement and is vital for upgrading society and country's economic status, energy, ecological community system integrity, nourishment manufacturing and human survival. Additionally, water is the basis of adjustment to climate change, acting as an interface between society and the environment [3].

Water is also a matter of rights. As the world's population increases, the need for a balance between all the commercial requirements of water resources is increasing, allowing communities to get enough water. In particular, women and girls should have access to private and clean sanitation facilities that ensure safety and dignity when dealing with biological female issues such as maternity.

At the human level, water and sanitation both are vital to reducing the global burden of disease, as well as their role in improving health, education and economic productivity of the population [1]. Due to statistics shown above, the percent of water usage "exhaustion" is dramatically increasing through the years. Therefore, water resource engineers worked on water management systems and designed hydraulic structures, such as breakwaters and dams to maintain water rates within its normal levels [2].

1.1.2 World Population Impact on Water Resources

World population has an impact on water levels. According to WHO status, the global population increased by 15% during the period between 1990-2000 which means that has increased from 5.27 billion to 6.06 billion. Regarding the population growth in 1990 that by 2000 extra 620 million people gained access to water supply and extra 435 million people gained access to sanitation facilities. For instance, the access of improved water by continent were reported as shown in figure 1. The distribution of the global population not served with improved water supply by region and it illustrates that Asia represents 63%, Africa 28%, Latin America and the Caribbean 7% and Europe 2% of the global population. However, the global population not served with improved sanitation distributed as follows: Asia 80%, Africa 13%, Latin America and the Caribbean 5% and Europe 2% [2].

1.1.3 Water and Sustainable Development

According to goal number 6 in the Sustainable Development Goals (SDGs), it is a global target to ensure the sustainable availability and management of water and sanitation for all [3]. Presently, the major challenge is to extend nourishment production using less water. Water shortage, turbid and unclean water and insufficient sanitation influence the food security, academic, sustenance and economic status of destitute family units around the world [4, 5].

On average, 70 percent of drawn water goes for crops and livestock fields which is quite lesser by 25 percent in some developing countries. Theoretically, as the world population increases, the drawn water for agriculture, agricultural crops and livestock

increases and that mainly appears as a dramatic increasing of global food consumption that continuously needs extra water production [3].

Food and Agriculture Organization (FAO) works with countries to ensure the efficiency of water used in irrigation and agriculture and guarantee a beneficial and environmentally friendly water. The strategy applied is based on producing more food with less possible water, adding irrigation methods and water sources with modern technologies for agricultural areas which are able to adapt to floods and droughts. FAO is also supporting other countries to put water stress levels and water resources usage under surveillance. The United Nations has put emphasis on the crisis of excess demand of water supplies all around the world to ultimately achieve economic, humanitarian and agricultural needs. The United Nations Water Conference (1977), the International Drinking Water and Sanitation Decade (1981-1990), the International Conference on Water and the Environment (1992) and the Earth Summit (1992) focused on this vital resource [6].

The International Decade for Action, Water for Life, 2005-2015 helped nearly 1.3 billion people in developing countries to access safe drinking water and accelerated progress in sanitation related to efforts to achieve the Millennium Development Goals [6].

Recent agreements include the 2030 Agenda for Sustainable Development, the Sendai Platform for Disaster Risk Reduction for the period 2015-2030, the Addis Ababa Plan of Action of the Third International Conference on Financing for Development and the Paris Agreement of the United Nations Framework Convention on Climate Change [6].

1.2 Water Issues

1.2.1 Water Major Problems

Nowadays, the world suffers from enormous water-related problems, such as floods which is an overflow of a large amount of water beyond its normal limits, especially over what is normally dry land. Contamination of rivers and large dams, the large dams have a severe impact on the rivers by reducing the flow of the rivers hence dams can affect the ecosystem's fauna and flora. Also, large dams hold sediments which are lead to serious coastal erosion because of the heavy gravels and cobbles behind the dam and make the river deprived of its sediment load as we found in the Aswan High Dam, statistics shows that 98% of sediments are remaining behind the dam which led to decreasing in the depth of the soil and its productivity also the severe change in Egypt's floodplain agriculture [7]. Water scarcity which is the lack of sufficient available water resources to meet the demands of water usage within a region. Now, 20% of the world's population live in physical water scarcity and the other 80% are approaching to the state of scarcity as well. Water scarcity could be because of the dry local conditions or human interference with the water cycle, which mainly appears in the use of factories, which produce greenhouse gases that turn into clouds with sulfuric acid which is the main component of acid rains that harms vegetation and water life. Due to acid rains, the water cannot be drinkable. Thus, water scarcity occurs. Siltation of river systems, silt is granulated material obtained from rock and soil of a grain size between sand and clay. Silt exists as a soil deposited on a river, and that precipitation can be defined as sediment pollution because it most often caused by sediment drops and soil erosion. One of the drawbacks of siltation that silt remains in the river which put marine life at risk as well as it affects the irrigation canals and make it

working improperly. Consequently, insufficient irrigation canals may cause severe health concerns on human beings, alteration in the fish migratory manner, wastage of wetland and change on the coastline [8, 9].

1.2.2 Efforts and Solution Efficiency

Wastewater treatment depends on the area itself, if it rural or urban. However, water pollution could be solved by effluent sewage systems. Effluent sewage is less common sewage disposal method than gravity sewage systems that lay on gravity as pumping where needed to send raw sewage and other wastewater straight from consumers to a sewage treatment plant. Effluent sewage system approximately has the same efficiency of the gravity sewer system, but operate at 25% of the cost. The gravity sewage system is a solution that might help the rural area that cannot afford treatment factories so this solution with water education programs could be applicable. Moreover, environmental institution could enforce regulation to decrease water pollution since in some urban area's countries, the water pollution comes from the industrial sector more than the domestic use. Therefore, by putting regulations, the generated money could be used in the water purification operations and finding many other solutions to reduce water pollution. Also, water conservation could be the fastest, most reliable and cheapest way to expand water supplies and save water resources, hence reduce the water scarcity which is using water efficiently to decrease needless water usage. Water conservation contains all techniques and policies to manage the natural resource of water to meet the future and present human demands with the water amount, so fixing water shortages. Another solution that most commonly be used in the Middle East is desalination, which include removing dissolved molecules like

salt from saline ground water by electro dialysis, boiling and filtration.

This operation is quite expensive, and requires huge energy input to produce water much better and more expensive than the fresh water from traditional sources. In addition, recycle wastewater counts as an effective solution to reduce water scarcity, water reclamation is the process of transferring or converting used water into water that can be reused for other purposes [10].

1.2.3 Other Water Remedying Methods

The most popular water purification process are coagulation and flocculation which play a dominant role in water reclamation as it treats wastewater and reduce turbidity “the number of suspended particles in water” using metal coagulant, such as iron or aluminum salts in order to coagulate contaminates in larger particles, so it easily settles down due to gravity and flocculants are also added to facilitate the agglomeration process. As a result, it will remove the unwanted taste of water and odor compounds and lower the rates of infection. The next step is sedimentation, the water tanks are left to give the chance for the heavy aggregated particles to settle in the bottom by gravity. Then, filtration is well-known method to separate colloidal particles from water as water passes through several filters that trap and remove particles that remain stuck in water after the sedimentation process. Finally, disinfection is the most essential part of this process, after completing the previous four steps, the water is free of particles and microorganisms. However, substances such as chlorine are added to make sure that any pathogens that may be present in water are eliminated (this is also done in swimming pools). After completion of the previous step,

the resulting water will be safe and ready for packing, distribution, home supply or for commercial purposes, Figure 2, [11].



Figure 2. Water remedying methods.

Previous research efforts have investigated the feasibility of membrane systems in separating colloidal particles off varying industrial effluents, but up to the author's knowledge the reported studies have lacked the emphasis on using different foulant models (Silica was used all the time), studying the combined effect of feed and draw solutions concentration on filtration rate, and investigating the effect of pretreatment approaches on the overall process efficiency.

In this work, the performance of Forward Osmosis membrane system in separating highly stable colloidal particles is been investigated. Three main aims were set out of this work:

1. The coupled effect of variable Bentonite (as a highly stable colloidal model) and salt concentrations on the efficiency of the Forward Osmosis process.
2. The effect of varying pH values of the Feed Solution on the separation of the stable colloids.
3. The behavior of the treatment process after introducing a coagulant to the feed solution.

Chapter 2: LITERATURE REVIEW

The integration of membrane processes has been implemented by different industries in wastewater treatment. The driving mechanisms of these processes are different, some of them operates under the electrical force, and some utilizing the difference in osmotic pressure to treat the effluent. The membrane processes include: Electrodialysis, Ultrafiltration, Nanofiltration, Reverse Osmosis, and Forward Osmosis (FO). Electrodialysis: In this process, the electrochemical fundamentals are combined with different unit operations in water using an ion-exchange membrane. It consists of five different compartments: 1) DC source which is used to support the ion-exchange process; 2) electrodes where the electrochemical reactions (i.e. oxidation and redaction) take place to reinforce the exchange process; 3) ion-exchange membranes which is simply a carriageway of the ions throughout the compartments; 4) solvents which is used as a space filler to adequately transfer the ions between the electrodes and the ion-exchange membranes; and 5) electrolytes as a current medium between anode and cathode electrodes [12]. A schematic diagram of the Electrodialysis process is shown in Figure 3.

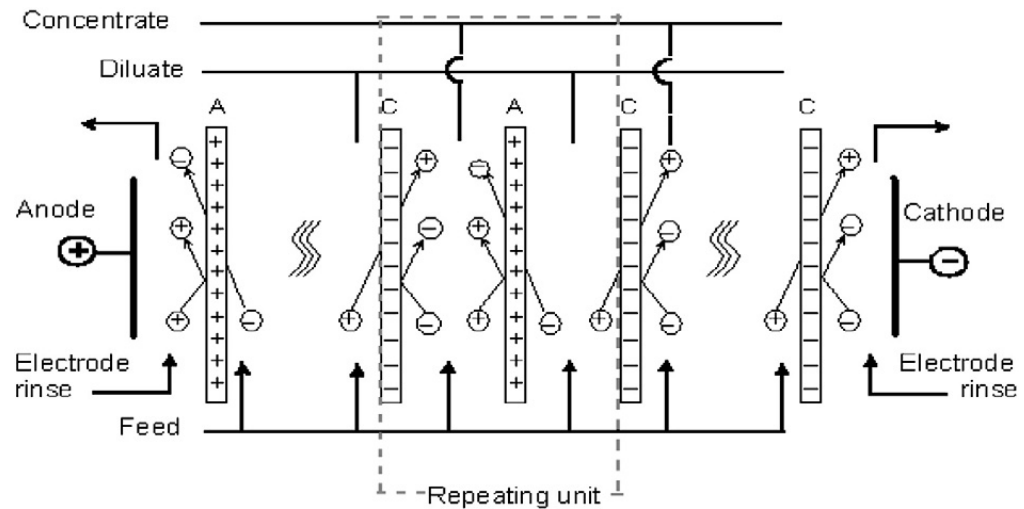


Figure 3. Electrodedialysis schematic diagram [13].

Ultrafiltration/Nanofiltration: Principally, both Ultrafiltration and Nanofiltration processes separate large molecular weight species out of the stream by letting through solvent and smaller species across the membrane, utilizing the power of pressurized flow as a driving force [14]. Also, they are sharing the same setup which consists of two main components a pressure pump and a dead-end filtration cell which mounts the semipermeable membrane, as illustrated in Figure 4. The only difference is the pore sizes of both membranes which reflects on how effective they can separate different type of species. For instance, UF can separate suspended particles and macromolecules, on the other hand, since the NF pore sizes are usually smaller, it can effectively separates what UF is capable of separating in addition to sugars, divalent salts, and dissociated acids [15].

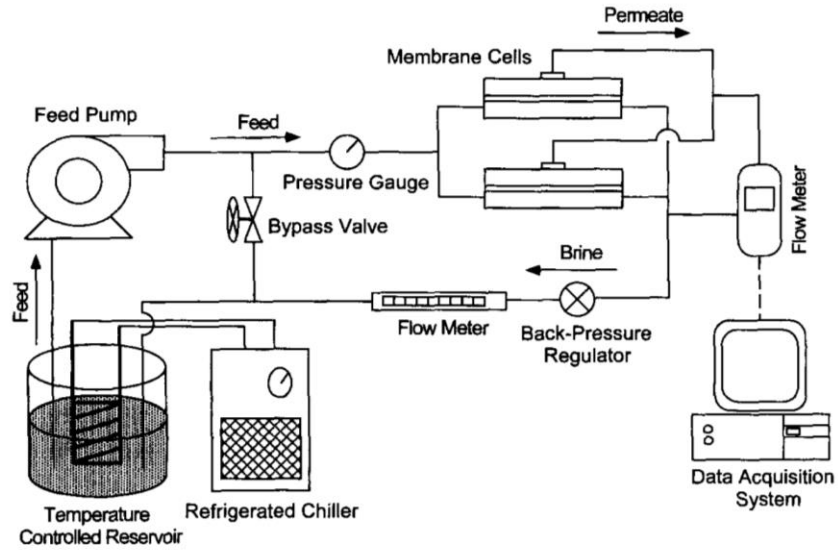


Figure 4. Ultrafiltration setup [16].

Reverse Osmosis This technology is one of the prominent membrane processes in water treatment. It has several applications in production of potable water, treatment of wastewater streams, and desalination of high salinity water sources like seawater. It forces high pressurized solvent through a semipermeable membrane against their osmotic pressure direction. By this process, the permeate is separating from the concentrate producing filtered water as illustrated in Figure 5 [17].

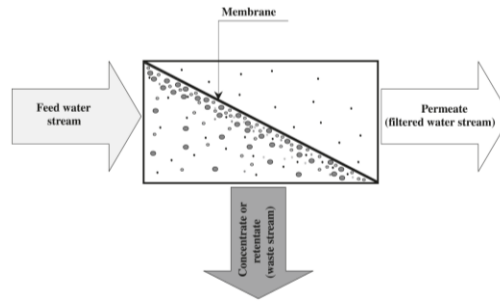


Figure 5. RO process [17].

Table 1 summarizes the different processes according to their driving forces.

Table 1. The Driving Forces of Different Membrane Processes

Membrane Process	Driving Force
Electrodialysis	Electrical potential between the membrane compartments [13]
Ultrafiltration	Hydraulic pressure [15]
Nanofiltration	Hydraulic pressure [15]
Reverse Osmosis	Hydraulic pressure greater than osmotic pressure of the feed solution [18]
Forward Osmosis	Osmotic pressure difference across the FO membrane coupon between both the feed and the draw solutions [19]

2.1 Forward Osmosis

According to 2010 report of world energy outlook, the energy consumption is forecasted to increase by 49% during the interval starting from 2007 to 2035 [20]. Further, due to 2008 financial crisis, many countries started to look into alternative solution to meet the water's market demand with the lowest cost possible, especially in water treatment industry [21]. Forward Osmosis technology was proven to have a relatively low energy consumption compared to different water treatment technologies (up to 85% energy saving) [22]. FO is a physical water treatment process by which the water transport across a semi permeable membrane due to the difference in osmotic pressure between two different solutions. The two solutions are referred to them as Feed Solution (FS) and Draw Solution (DS), where the first is the actual feed which needs to be purified, and the latter is used to induce difference in osmotic pressure between the two sides [23]. A schematic diagram of the FO process is shown in Figure 1. The attention to this promising technology has increased due to the fact that it doesn't require any hydraulic pressure in the separation process [24]. It has been applied in the treatment of different streams and wastewater effluents [25].

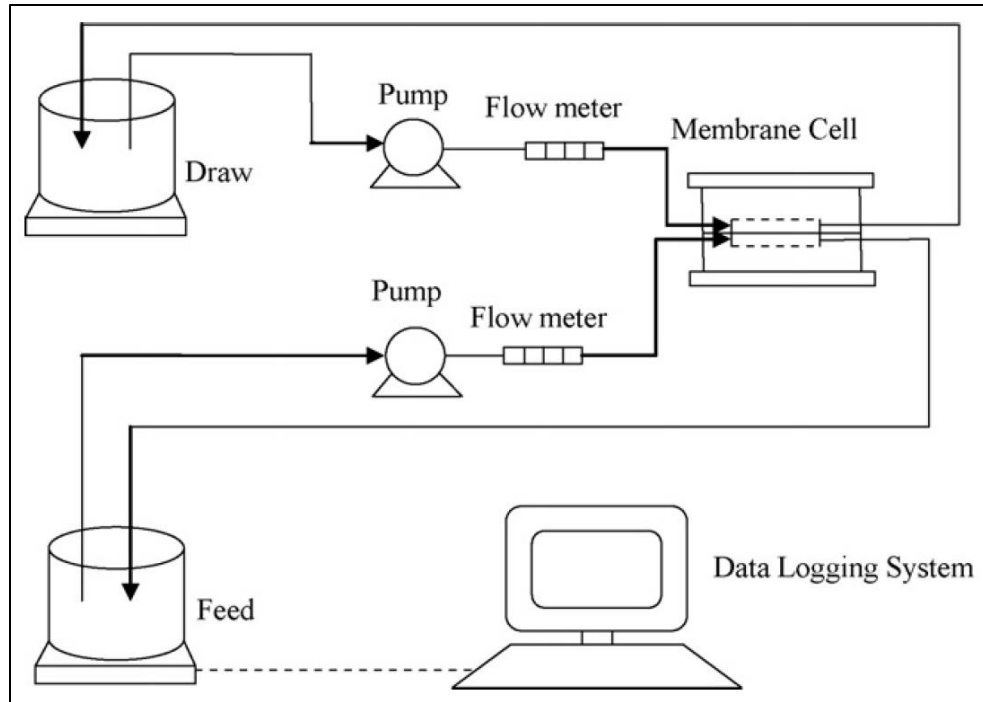


Figure 6. FO schematic diagram [26].

2.1.1 FO Materials

FO membranes are made up by different type of materials, they vary from a single component to a composite structure. Usually, the membrane material for either the commercialized or those fabricated for research purposes are categorized as shown in Figure 7.

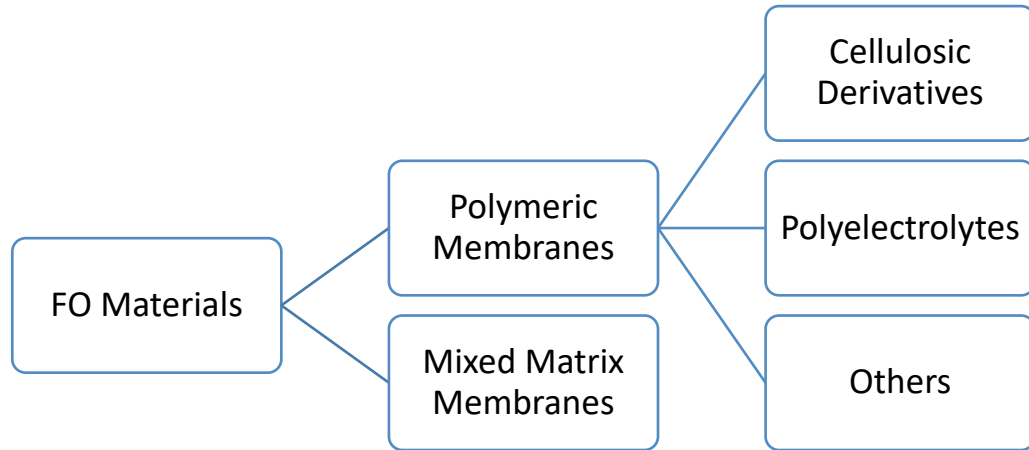


Figure 7. FO materials matrix.

2.1.1.1 Polymeric Membranes:

The structure of a typical FO membrane is consisting of 2 layers, active layer and support layer. The main characteristics of the active layer are the high value of water flux/permeability and low value of reverse diffusion of the draw solution. The support layer, on the other hand, tolerates the transfer of high amount of feed solution and decreases the effect of concentration polarization. Also, it enhances the overall features of the membrane's module in terms of resistance of the feed chemistry and structural stability [27, 28]. The main research focus are on Polyelectrolytes, Polyamide/Polymers, and Cellulosic [29].

Cellulosic derivatives:

The most well-known cellulosic derivatives' membrane is supplied commercially by Hydration Technology Innovations (HTI) [30]. It has a good rejection rate of salt (>95%) compared to the commercial RO membrane [31]. However, the water flux of the

FO process was found to be lower than the theoretical value [32]. The difference between the values is attributed to the formation of external and internal concentration polarization across the membrane surface [27, 33]. The external part can be reduced by optimizing the hydraulic characteristics of the fluid (e.g. turbulence), and the internal part is mainly a function of membrane's structure [34]. The correlation between the membrane performance and its structure is still being studied, but a concept, which has the capability of lowering the internal concentration polarization, was demonstrated. Its thickness is 71nm, with a high water flux rate (~22 LMH) [35]. Nonetheless, these membrane modules are not practical due to their poor mechanical strength [36].

Polyelectrolytes:

Membranes made of polyelectrolytes have several advantages; they are thermally stable, have a good solvent resistance, good water flux, and high rejection rate of the feed's divalent ions [37]. It was found that using the polyelectrolytes in manufacturing the modules lead to an enhancement on the fouling resistance over the conventional cellulosic membranes, by minimizing foulants' clogging within the porous support which cause a leap on mass transfer of the feed across the FO membrane [38]. The first experimental test of a membrane made of polyelectrolyte was carried over in 2011. A high-water flux rate was achieved (28.7 LMH), for DI water feed solution against 1.0 MgCl₂ draw solution. Unfortunately, high reverse diffusion of the draw solution was observed [37]. It was found that accumulating larger bilayers of polyelectrolytes inhibit the diffusion of the draw solution, but a reduction in the water permeability was observed [39].

Others:

The use of polybenzimidazole (PBI) in membrane manufacturing was first introduced in 1983. It is well known for its thermal, chemical, and mechanical characteristics [40]. The first experimental work with a PBI hollow fiber membrane yielded a high rejection rate to divalent ions ($\text{MgSO}_4 > 99\%$) and monovalent salt ($\text{NaCl} \sim 97\%$) for a single salt draw solution against DI water. Nevertheless, the maximum water flux achieved was around 9 LMH [41].

2.1.1.2 Mixed Matrix Membranes

It is a new fabrication technique at its early stages. Mainly, it is the integration of different filler types into a polymeric matrix in FO membrane manufacturing [42]. An experimental work showed a promising improvement of the FO overall performance, a high water flux rate was achieved (95.7 LMH) and a limited draw solution diffusion was observed (4.8 gMH) and both values were 160% higher and 30% lower than the values achieved by the conventional TFC FO module [42]. The similar concept was further studied in a different configuration to achieve higher water flux, but lower internal concentration polarization. Also, a good fouling resistance to humic acid was noted [43].

2.1.2 Advantages and Potential Uses of FO

FO technology has several advantages over the other membrane processes. It removes different types of pollutants such as total dissolved solids with high rejection rate [27]. It requires lower energy input than the other membrane processes due to its nature which utilizes the difference in osmotic pressure without the aid of high hydraulic pressure, and that also reduces the strength required of the membrane module [44]. In a simultaneous process, FO is a highly efficient in “thickening, digestion, and direct dewatering of raw waste activated sludge”. It has a great effect on the reduction of both mixed liquor

suspended solids and mixed liquor volatile suspended solids amount of the activated sludge [45]. Also, it has a lower fouling rate than other processes with a more fouling reversibility of the membrane [46, 47].

FO process has the efficient to treat different type of streams. It treats landfill leachate, desalinates seawater/brackish water, and treats different wastewater effluent with high rejection rate [27, 33]. Aside from water treatment, FO technology also has different applications as industrial practices. The technology is used as an osmotic pump in order to release medical drug in a controlled manner. It improves the delivery of insoluble drugs to patients and protects them from different safety concerns including dose dumping [48]. In food industry, it has been utilized in concentrating freshly squeezed fruit and vegetable juices, and its efficiency in dealing with pulpy and cloudy sources has been proved [49]. Different applications of the process based on the classification are illustrated in Figure 8.

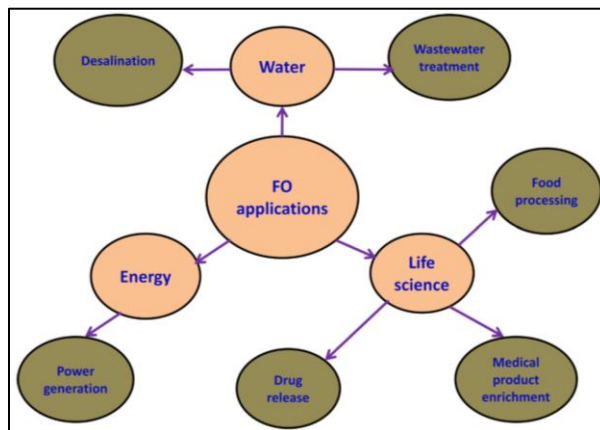


Figure 8. FO Applications [33].

2.2 Challenges in FO

2.2.1 Cake Formation/Fouling in FO

Although FO was proven to have less fouling effect across the membrane [33], the formation of cake layer has an adverse effect over the overall efficiency of the FO process [50]. Different mitigation techniques were carried over in several FO experimental studies, namely, physical cleaning, utilizing a feed spacer, varying the operating conditions like temperature and pH values, and using different cleaning agents as shown in Figure 9. Physical cleaning, for instance, is utilizing the hydraulic pressure to remove the foulant accumulations which devolved across the membrane surface over time [51]. The use of feed spacer was proved to inhibit the formation of biofilm over the membrane surface, and its application become useful in wastewater with a rich organic content [52]. Also, the changing the operating conditions of the process was found to affect the fouling on the membrane module [53]. Finally, chemical cleaning is simply the use of different cleaning agents to recover the declined flux rate of a membrane. Different cleaning agents were evaluated by Porcelli et al. [54]. Table 2 highlights the mitigation techniques used in different types of feed solutions and the main outcomes resulting from applying these mitigation methods.

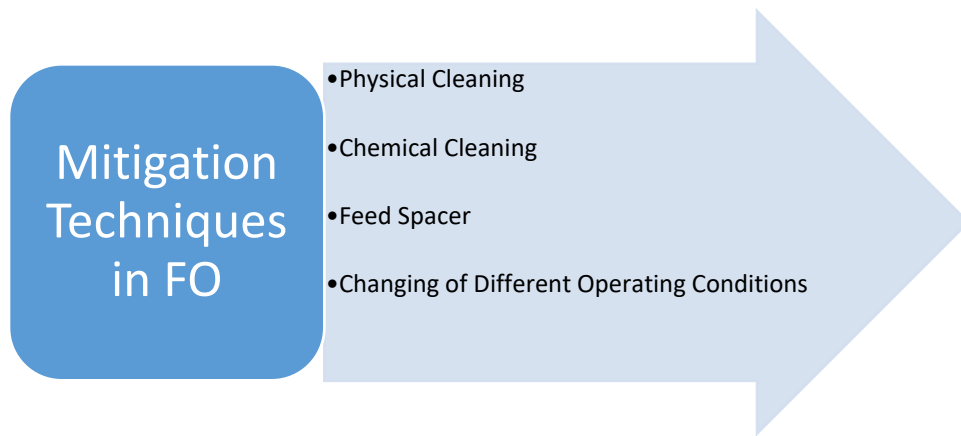


Figure 9. FO's mitigation techniques.

Table 2. Flux Recovery Techniques in FO

Membrane Type/Manufacturer	Feed Solution Composition(s)	Mitigation Measure	Main Findings	References
Hydration Technology Innovations	Silica solution	Physical cleaning	<ul style="list-style-type: none"> Physical cleaning was able to mitigate the colloidal fouling layer. The thickness of the cake layer formation is inversely proportional to the water flux across the membrane. 	[55]
Hydration Technology Innovations	3-fold concentration of synthetic wastewater mixed with some organic constituent	Hydrodynamic mixing	<ul style="list-style-type: none"> The accumulation of organic/inorganic constituents over the membrane's surface is increased at higher feed water flux. Hydrodynamic mixing was found to be highly efficient in fouling control since the nature of FO doesn't utilize any hydraulic pressure. 	[56]
Hydration Technology Innovations	Sodium alginate	Physical Cleaning	<ul style="list-style-type: none"> The fouling of sodium alginate is fully reversible. After physically cleaning the membrane module, 98% of the water flux was recovered. 	[57]
Fabricated Polymer Polyethersulfone (PES) hollow fibers	Diluted Dextran	Hydraulic cleaning (2 different modes: back wash and crossflow flushing)	<ul style="list-style-type: none"> Back washing mode was found to enhance the flux up to 85% and the initial flux was fully recovered with further flushing 	[58]
Hydration Technology Innovations	Natural seawater	Hydraulic cleaning (crossflow flushing)	<ul style="list-style-type: none"> The mitigation technique was found to reach a stable salt rejection efficiency of 98%. Silica formations were found difficult to be mitigated through hydraulic cleaning. 	[59]

Membrane Type/Manufacturer	Feed Solution Composition(s)	Mitigation Measure	Main Findings	References
Hydration Technology Innovations	A combination between organic (humic acid, alginate, and bovine serum albumin) and colloidal foulants (silica colloids).	Hydraulic cleaning	<ul style="list-style-type: none"> Reverse salt diffusion (i.e. from the draw solution to the feed solution) was found to increase the resistance of the fouling layer. Selecting a proper draw solution for the treatment process improve the rejection of reverse salt diffusion by FO membrane. 	[60]
Hydration Technology Innovations	Synthetic brackish groundwater	Physical cleaning	<ul style="list-style-type: none"> Diammonium phosphate as a draw solution had a severe fouling effect over the membrane surface due to its reverse diffusion. Physical cleaning was found to fully recover the water flux. 	[61]
Hydration Technology Innovations	Sodium alginate (Organic foulant) and gypsum (Inorganic foulant)	Physical cleaning with contiusly aerating the rinsing water.	<ul style="list-style-type: none"> The synergistic effect of the foulants caused faster reduction in water flux. Initial water flux recovery requires chemical cleaning. 	[62]
Hydration Technology Innovations	Synthetic brackish water	Physical cleaning	<ul style="list-style-type: none"> The higher the TDS (>20,000 mg/L) of the feed solution, the lower the water flux. 	[63]
Hydration Technology Innovations	Two feed solutions have been used over the filtration course. Secondary wastewater (Municipal effluent) followed by synthetic municipal wastewater (No presences of bacteria) for 5 cycles of FO filtration.	Feed spacer	<ul style="list-style-type: none"> The water flux reduction due to biomass accumulation can be mitigated using a thicker feed spacer. The biofilm spatial distribution varies according to the spacer's thickness. 	[64]

Membrane Type/Manufacturer	Feed Solution Composition(s)	Mitigation Measure	Main Findings	References
Hydration Technology Innovations	Brackish water	Physical cleaning along with variation in working temperature	<ul style="list-style-type: none"> Working temperature has a great impact on different FO parameters. It was found that varying the working temperature effects both the initial feed flux across the membrane and recovery efficiency. However, it has adverse effects on the module's fouling and the cleaning process. 	[33]
Hydration Technology Innovations with Ag/TiO ₂ nanoparticles coating	Non-chlorinated groundwater with the aid of sodium acetate, sodium nitrate, and sodium dihydrogen phosphate	Ag/TiO ₂ coating and physical cleaning	<ul style="list-style-type: none"> It was found that the Ag/TiO₂ nanoparticles coating has less bacterial growth by 11 times than the untreated (virgin) membrane. 	[65]
Hydration Technology Innovations (Flat Sheet Membrane) and Singapore Membrane Technology Centre (Hollow Fiber Membrane)	Produced water	<ul style="list-style-type: none"> Powdered activated carbon pretreatment Chemical cleaning Physical cleaning 	<ul style="list-style-type: none"> It was found that the water flux and rejection efficiency of HF is higher than FS. Organic rejection of HF is >99% Physical cleaning technique is unsuccessful in removing Carbon foulants. Sodium dodecyl sulfate was found to enhance the process of water flux recovery. 	[66]
Hydration Technology Innovations	Alginate (Organic foulant) and silica (Inorganic colloidal)	Physical cleaning at different pH values	<ul style="list-style-type: none"> The cake layer formed due to the synergistic between organic and inorganic foulants is irreversible if a hydraulic pressure is applied. The variation in pH values couldn't mitigate the combined effect of the foulants. 	[67]
Hydration Technology Innovations	Sodium Alginate and calcium chloride	Physical cleaning	<ul style="list-style-type: none"> The introduction of high hydraulic pressure in physical cleaning leads to irreversible fouling in FO membrane. 	[68]

Membrane Type/Manufacturer	Feed Solution Composition(s)	Mitigation Measure	Main Findings	References
Hydration Technology Innovations	Produced water	Chemical cleaning	<ul style="list-style-type: none"> The claim that FO is a low fouling technology was proved wrong. Pretreatment of complex feeds is advised to avoid both the adverse fouling and degradation of the FO's module. 	[69]
Hydration Technology Innovations	Municipal wastewater	Physical and chemical cleaning	<ul style="list-style-type: none"> FO has higher reduction in water flux than osmotic membrane bioreactor due to biomass accumulation over the membrane's surface. Better water flux recovery results were obtained by FO after the cleaning process. 	[70]
Fabricated polyamide thin-film composite (TFC) and Hydration Technology Innovations' module	Filtered Shale Gas Drilling Flowback Fluid (SGDF)	Ultrafiltration pretreatment and TFC surface modification using ethylene glycol	<ul style="list-style-type: none"> The pretreatment technique couldn't reduce the fouling effect, but delayed it. The modified TFC had lower fouling rate along with a marginal reduction in water flux. 	[71]
Hydration Technology Innovations	Coal seam gas's produced water	Physical cleaning	<ul style="list-style-type: none"> The mitigation technique was found to be efficient in full recover the declined water flux. The FO process was found highly efficient in rejection of organic/inorganic compounds and biomass in the given feed. 	[72]
Fabricated hollow fiber membrane	Produced water	Different cleaning agents (sodium dodecyl sulfate (SDS), ethylenediaminetetraacetic (EDTA) and NaOH)	<ul style="list-style-type: none"> SDS was found to be the most effective cleaning agent over EDTA and NaOH. 	[73]

Membrane Type/Manufacturer	Feed Solution Composition(s)	Mitigation Measure	Main Findings	References
Hydration Technology Innovations	Coal seam gas's produced water	Physical cleaning	<ul style="list-style-type: none"> FO had a little decline in flux compared to pressure retarded osmosis (PRO). A complete restoration of the declined flux was achieved through the physical cleaning. 	[74]
Hydration Technology Innovations	MBR-treated landfill leachate	Chemical cleaning	<ul style="list-style-type: none"> Landfill leachate treated by the given draw solution (NH_4HCO_3) can be used directly for fertigation. 	[75]
Hydration Technology Innovations	Oily wastewater	Physical cleaning, osmotic backwash, oxidant, acid, surfactant, and complexant cleaning	<ul style="list-style-type: none"> Osmotic backwash was found to be the best mitigation technique with a high flux recovery efficiency (95%). 	[76]

2.2.2 Draw Solution Types and Draw Solution Recovery

Another dimension which affects the performance of the FO system is the selection of the draw solution. Many researchers tried out different draw solutions and studied the impact on the system's flux due to different parameters. Their main goal was to look into the sustainability of the FO processes, and how incorporating the FO unit might lead to lower operating costs. For instance, the recoverability of the draw solution was emphasized by different researchers. Different techniques were used in order to fully recover the experimented draw solution as shown in Figure 10.

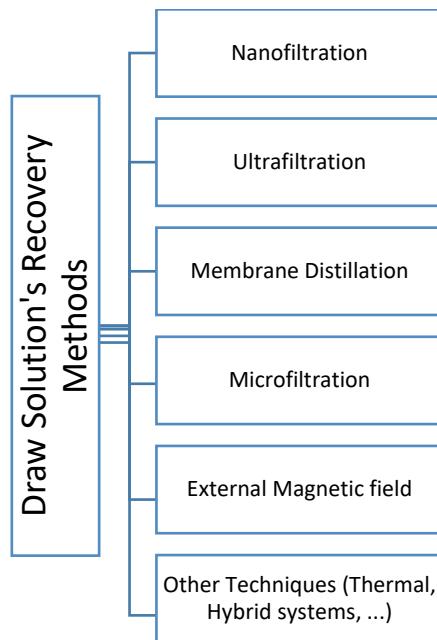


Figure 10. DS recovery methods.

Highlights of the advancements of FO's draw solutions along with the associated recovery methods, and main findings are tabulated in Table 3.

Table 3. DS Advancements and Recovery Methods for FO Applications

Membrane Type/Manufacturer	Draw Solution Type/Concentration	Recovery Methods	Main Findings	References
Hydration Technology Innovations and Fabricated Cellulose Acetate (Hollow Fiber)	PAA-Na polyelectrolytes	Ultrafiltration (UF)	<ul style="list-style-type: none"> • Due to their high solubility in water, they provide high osmotic pressure and they can be easily recovered with ultrafiltration aid. • Although PAA-Na polyelectrolytes have comparable flux results to the conventional ionic salts counterparts, they have much lower reverse diffusion affinity. 	[77]
Hydration Technology Innovations	Poly (sodium 4-styrenesulfonate)	UF	<ul style="list-style-type: none"> • Conductivity and viscosity values increase with an increase in the draw solution's concentration (Mw = 70,000 achieved the best FO's flux). • The draw solution has a lower reverse diffusion over the conventional ionic salts. • Ultrafiltration process requires low energy demand (>2 bar) for the recovery process. 	[78]
Hydration Technology Innovations	poly (aspartic acid sodium salt)	Nanofiltration (NF)	<ul style="list-style-type: none"> • The draw solution used has good solubility in water, non-toxicity nature, and high osmotic pressure. • The reverse diffusion of the draw solution has a positive impact on the organic matter fouling. 	[79]

Membrane Type/Manufacturer	Draw Solution Type/Concentration	Recovery Methods	Main Findings	References
Hydration Technology Innovations	Polymerization of Sodium Acrylate (SA) and Nisopropylacrylamide (NIPAM)	Hot Ultrafiltration (HUF): A technique used to transfer the nature of the draw solution chains from hydrophilic to hydrophobic	<ul style="list-style-type: none"> HUF was found to be an effective technique in recovering the draw solution with a low energy demand (> 2 bar) 	[80]
Hydration Technology Innovations	PSSS-PNIPAM copolymers	Membrane Distillation (MD)	<ul style="list-style-type: none"> The thermoresponsive nature of the draw solution improves the recovery tendency by MD where the reduction in osmotic pressure elevates the vapor pressure of water. Hence, increase the separation efficiency. 	[81]
Hydration Technology Innovations	Polyethylene glycol/Humic Acid	UF/NF	<ul style="list-style-type: none"> Both of the draw solutions are irrecoverable at higher concentrations using the given recovery methods. An optimization process is required for the draw solution's concentration to recovery. 	[82]
Hydration Technology Innovations	Polyethyleneimine	NF	<ul style="list-style-type: none"> Due to the lower viscosity and polarization, It was found that at high molecular weight of the draw solution (MW = 1200), the flux had dropped compared to a lower value (MW = 800). NF recovery rate of the draw solution was high (99.4%) 	[83]
Hydration Technology Innovations	P(MT _x EO _y)	Heating up the draw solution + Microfiltration (MF)	<ul style="list-style-type: none"> P(MT₂₀EO₈₀) draw solution's recovery value was 99.8% (70 °C heating temperature followed by MF) 	[84]
Hydration Technology Innovations	Poly (amidoamine) reacted with sodium carboxylate groups (PAMAM-COON _a)	MD	<ul style="list-style-type: none"> 2.5G PAMAM-COONa generation was found to be the optimal draw solution in different FO applications. 	[85]

Membrane Type/Manufacturer	Draw Solution Type/Concentration	Recovery Methods	Main Findings	References
Hydration Technology Innovations	Ethylenediamine Tetrapropionic	NF	<ul style="list-style-type: none"> pH value optimization of the draw solution enhanced the osmotic pressure. The higher concentration of the draw solution has an exponential relation with the osmotic pressure. Hence, higher flux rate. NF recovery technique fully recovered the draw solution. 	[86]
Hydration Technology Innovations	EDTA sodium salt	NF	<ul style="list-style-type: none"> At a pressure of 5.5 bar, NF-TS80 was the most efficient membrane in recovering the draw solution. Compared to conventional inorganic salts, the EDTA has a lower salt leakage value. 	[87]
Hydration Technology Innovations	Ferric CA complex (Fe-CA)	NF	<ul style="list-style-type: none"> Due to concentration polarization, the higher the concentration of the diluted draw solution, the lower the water flux and the draw solution rejection rate. Using the given draw solution, hollow fiber membrane outperforms the flat sheet counterpart in terms of water flux. 	[88]
Hydration Technology Innovations	2-methylimidazole	MD	<ul style="list-style-type: none"> The osmotic pressure is not the only factor which affects the performance of the draw solution. Other factors like ionic strength and external concentration polarization also affects the performance. The novel solutions have high water flux and low reverse diffusion values due to their high solubility, and ionic strength nature. 	[89]

Membrane Type/Manufacturer	Draw Solution Type/Concentration	Recovery Methods	Main Findings	References
Hydration Technology Innovations + Fabricated hollow fiber membrane	Different micellar solutions	UF/Krafft point method (by lowering the draw solution temperature below the Krafft temperature)	<ul style="list-style-type: none"> Micellar solutions with higher Krafft temperature are easier in regeneration, and require less refrigeration energy. 	[90]
Hydration Technology Innovations	Sodium lignin sulfonate (NaLS)	UF/NF unit	<ul style="list-style-type: none"> NaLS large molecules can be easily separated than other conventional salts. 	[91]
Hydration Technology Innovations	Super hydrophilic nanoparticles	UF	<ul style="list-style-type: none"> UF separation method is better than magnetic separation in recovering the draw solution. The draw solution can be recycled for 5 continuous cycles without affecting its osmotic pressure value. UF membranes with smaller pore diameter and fine pore distribution are preferable in the recovery process. 	[92]
Hydration Technology Innovations	Dextran coated Fe ₃ O ₄ magnetic nanoparticles (MNPs)	External magnetic field (EMF)	<ul style="list-style-type: none"> The dextran content has a positive impact on the water solubility of the draw solution which leads to a higher osmotic pressure. Due to the draw solution's superparamagnetic nature, it can be easily recovered by an external magnetic field. 	[93]
Hydration Technology Innovations	Synthesized magnetic nanoparticles (MNPs)	EMF	<ul style="list-style-type: none"> The draw solution can be recycled for 9 continuous cycles with a 21% drop in the water flux rate. 	[94]
Hydration Technology Innovations	Polyacrylic acid magnetic nanoparticles (PAA-MNPs)	EMF	<ul style="list-style-type: none"> Decreasing the diameter of nanoparticles, enhances the water flux value. 	[95]
Hydration Technology Innovations	Magnetic thermoresponsive ionic nanogels	Heating + EMF	<ul style="list-style-type: none"> The dual effect of the used recovery technique improves the recovery process efficiency, and results in lower energy consumption. 	[96]

2.3 Previous Works in Separating Colloidal Particles by FO

One of the drawbacks of the FO process is the membrane fouling over time. The cake layer formation over the membrane surface hinders the overall treatment efficiency overtime. FO membranes are prone to different fouling mechanisms based on the nature of the feed solution; it varies from a single fouling mechanism to multiple fouling mechanisms based on the nature of the feed solution. These fouling mechanisms have been studied extensively in previous works. For instance, the effect of organic fouling, inorganic fouling, biological fouling, and combined fouling can be seen elsewhere [97]. Nonetheless, there is a lack of research in investigating how FO process tolerates the colloidal fouling. Up to the author's knowledge, some studies have been carried out previously by [55, 60, 98-100], their goal was to assess how effective the FO process is in treating waters contain highly stable colloidal particles.

Choi et al. [98], have experimentally studied the colloidal fouling behavior of FO in four different modes, namely, FO mode in which the membrane faces the feed solution side; PAFO mode, which is an application of some extra pressure within the feed solution's loop; UAFO mode, in which ultrasound waves of an ultrasound transducer equipment were being utilized; PUFO mode where both PAFO and UAFO modes were combined. The stable colloidal model used in this study was silica colloid with a concentration of 5 g/L with an average diameter ranging from 10-20 nm. The main findings of their study are as follows: 1) Comparing to an inorganic scalant like calcium sulfate (CaSO_4) in FO mode, silica particles have attributed to a higher flux decline across the membrane. It was found that when the concentration factor (i.e. final/initial concentrations) has reached 2.5, the flux rate has dropped by 50%; 2) In PAFO mode, applying a pressure of 5.0 bar has enhanced

the initial flux by 20%, however, the pattern of flux decline afterwards was found to be matching the CaSO₄ scalants; 3) Ultrasound waves have lowered the declination of flux rate by around 30%, since this mitigation technique was able to effectively detach the cake layer formation all over the membrane; and 4) Combining both the ultrasound waves and the pressure within the FO setup have minimized the effect of colloidal fouling. This behavior was attributed to the elevated pressure within the feed solution loop, which results in an increase of the ultrasound intensity; 5) Physical cleaning technique was used to investigate the reversibility of the membrane's fouling, it was found that physical cleaning has recovered a minimum of 75% of the initial flux in all the studied modes.

Kim et al. [99], have investigated the effect of osmotic backwashing in mitigating the FO membrane fouling due to colloidal particles. In osmotic backwashing, the high-salinity draw solution is replaced with deionized water to switch the permeate flow direction from the draw solution side into the feed solution side instead of how FO usually operates. In other words, the osmotic pressure is higher in the feed solution side which forces the permeate to flow towards its direction. In this study, the experimental work was carried out over multiple stages as follows: a) Baseline experiments were performed to determine the flux pattern of deionized water; b) Multiple fouling experiments were run using different foulant concentrations; and c) the effect of osmotic backwashing on destabilizing the cake layer formation was assessed. The stable colloidal model used in this study was silica colloid with a concentration of 2 g/L with two different particle diameters: 20 nm and 100 nm. The main findings of their study are as follows: The fouling of 20nm silica particles for AL-DS was more severe than AL-FS, since the small silica particles were entrapped within the membrane pores in the AL-DS mode. Hence, the shear force

resulting from the crossflow had a negligible effect in scraping the cake layer formation. Nonetheless, osmotic backwashing was found to recover the initial flux of AL-DS mode to some extent. The fouling patterns of 100 nm silica particles for both operational modes were matching, and the decline in flux rates were more severe than 20 nm silica particles due to a thicker cake layer formation all over the membrane. However, osmotic backwashing has recovered 85% of the initial flux rate.

Lee et al. [60], have systematically investigated the difference in fouling behavior of colloidal particles between Reverse Osmosis (RO) and FO. They have studied the effect of different crossflow velocities on the flux rates over time, how salt diffusivity relies on the draw solution's salt, and the effect of the particle size on the process efficiency.

Boo et al. [55], have assessed the effect of the back diffusion of the draw solution's salt on FO. They have investigated the effect of salt concentration in the draw solution, the contribution of pH value in the feed solution with different colloidal particle concentrations of a single and multiple particle sizes. The stable colloidal model was with a concentration of 1 g/L and 2 g/L with an average diameter ranging from 10-20 nm and 70-10 nm, respectively. The same research group behind this study has also developed a code to model the separation process [100].

Table 4 below summarizes the previous studies on the colloidal particles' separation by FO.

Table 4. Previous studies of FO on colloidal particles separation

Feed Solution	Draw Solution	Parameter(s) Studied	Reference
5 g/L SiO ₂ + 0.5M NaCl (seawater condition)	4M NaCl	<ul style="list-style-type: none"> Flux rates in FO, PAFO, UAFO, and PUAFO modes. The effectiveness of physical cleaning in fouling reversibility. 	[98]
2 g/L SiO ₂ + 0.6M NaCl (seawater condition)	5M NaCl (AL-FS) 3.8M NaCl (AL-DS)* *Note: Two different salt concentrations were used to achieve a similar initial flux value in both operating modes.	<ul style="list-style-type: none"> Flux rates out of AL-FS and AL-DS modes were investigated. The Effect of osmotic backwashing on permeate flux recovery. 	[99]
1mM NaCl	5M NaCl	<ul style="list-style-type: none"> Varying crossflow velocities. Salt back diffusion. Different particle sizes effect. 	[60]
1 g/L, 2 g/L of silica colloids of varying particle size throughout the experimental work	5M, 3M, 2.5M of NaCl and LaCl ₃	<ul style="list-style-type: none"> Solutions chemistry. Fouling reversibility. 	[55, 100]

Although the aforementioned studies have assessed the FO technology's efficiency in separating highly stable colloids, they lacked the emphasis on the following conditions:

- 1) Different foulant model (the same model was used all the time);
- 2) The effect of coupled action of both the feed and draw solutions' concentration;
- 3) How different pretreatment approaches could affect the overall process efficiency.

FO given its many advantages in treating complex effluents over different treating techniques is assessed in this study using a different foulant model, which used in mimicking real industrial wastewater. For this purpose, the following objectives are set:

- 1) The coupled effect of variable Bentonite --as a highly stable colloidal model-- and salt concentrations on the efficiency of the Forward Osmosis process;
- 2) The effect of varying pH values of the Feed Solution on the separation of the stable colloids; and
- 3) the behavior of the treatment process after introducing a coagulant to the feed solution.

Chapter 3: METHODOLOGY AND EXPERIMENTAL PROTOCOL

3.1 Forward Osmosis Apparatus

The separation performance of highly stable colloids by utilizing the Forward Osmosis process is being investigated in this work. Figure 11 illustrates a bench-scale of the Forward Osmosis technology which is used in this work. Also, the actual crossflow unit (Model No. CF042D-FO) was provided by Sterlitech.

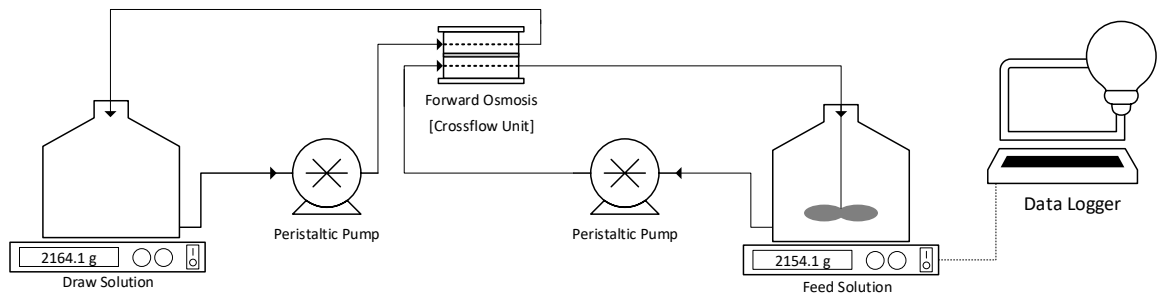


Figure 11. FO schematic diagram.



Figure 12. FO crossflow unit.

Aquaporin based FO flat sheet membranes were used in this work. It was provided by Sterlitech. According to the manufacturer official website, the provided FO membranes have an active membrane area $A = 42 \text{ cm}^2$, a thickness of $110 \mu\text{m}$ with an error range of $\pm 15 \mu\text{m}$. It operates on a temperature range of 5°C to 50°C , and a pH range of 2 to 11. Finally, it has a shelf life of 6 months.

Two peristaltic pumps were used to circulate both the feed and the draw solutions, it is worth mentioning that a silicone grease was applied around the pumps' shafts to lower the friction between them and the setup's tubes. #16 tubes were used to connect the containers with the crossflow unit. Both the pumps and the connecting tubes were provided by Cole-Parmer as seen in Figure 13.

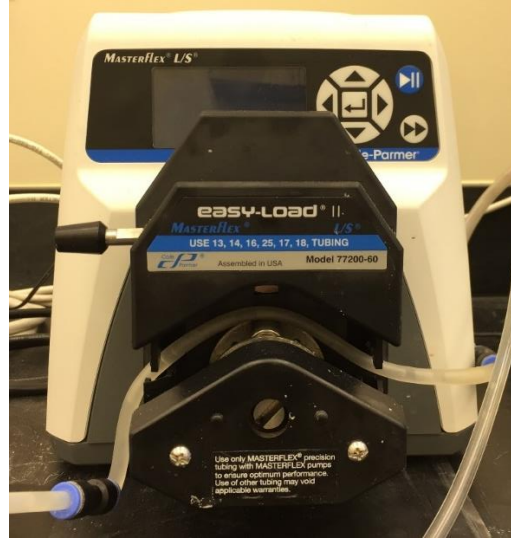


Figure 13. FO setup peristaltic pumps.

3.2 Feed Solution Preparation

Different feed solutions were prepared to serve the experimental work's objectives. We have lab prepared Bentonite water with varying concentrations, starting from pure water all the way up to 1.5 g/L of Bentonite. Table 5 summarizes feed solutions. Lab grade Bentonite powder was provided by Sigma-Aldrich.

Table 5. Feed Solution Concentrations

Feed Solution Concentrations		
Type	Concentration [g/L]	Volume [L]
Deionized Water	-	
Bentonite Water	0.5	2
	1	
	1.5	

In this objective, a constant concentration of Bentonite water was set against varying pH values to investigate its effect on the separation process as shown in Table 6.

Table 6. pH of Feed Solutions

Feed Solution pH			
Type	Concentration [g/L]	pH	Volume [L]
Bentonite Water	1	4	2
		7	
		9	

Finally, different concentrations of a coagulant (NaCl) were mixed with the Bentonite water to investigate their effect on the treatment process as seen in Table 7.

Table 7. Coagulant Concentration in Feed Solutions

Feed Solution – Coagulant Concentrations				
Type	Concentration [g/L]	Coagulant Type	Coagulant Concentration [M]	Volume [L]
Bentonite Water	1	NaCl	0.001 0.01 0.1	2

3.3 Draw Solution Preparation

The Draw Solution is an essential part of the Forward Osmosis process, it generates the osmotic pressure as a filtration driven force across the membrane due to the difference between their chemical strength. Hence, different concentrations were employed to serve this work's objectives as shown in Table 8. Lab grade NaCl salt with a purity of 99% was provided by Sigma-Aldrich.

Table 8. Experimental Draw Solutions

Draw Solution		
Type	Concentration [M]	Volume [L]
NaCl Solution	1	2
	1.5	
	2	

3.4 Mixing Tool

High speed stirrer was used throughout the experiments. It was incorporated in this work for different purposes. It was used to rapidly mix the Bentonite solution while it was mixed to make sure the solution was homogenous. Also, it was used while the fouling experiments were running to eliminate any pressure buildup around the feed solution side which was causing the tubes to get ruptured. Hence, the utilization of the stirrer at a lower mixing speed throughout the fouling experiments has assured the stability of the colloidal particles. The stirrer was provided by Caframo (Model No. BDC6015) as seen in Figure 14.



Figure 14. High rotational speed stirrer.

3.5 Weighing Scale

Two types of weighing scales were used in this work, namely, top loading balance and analytical balance. The top loading balance (Model No. PGW 4502e) as seen in Figure 15 was used to get the solution weighed and the difference in weight while the experiments were running was recorded using a computerized datalogging system.



Figure 15. Top loading balance.

The analytical balance (Model No. PW 214) Figure 16, on the other hand, was used to precisely weight the required amount of Bentonite colloidal particles as per the experimental aim. Both balances were provided by Adam Equipment.



Figure 16. Analytical balance.

3.6 Experimental Procedure

The efficiency of FO membrane system on separating highly stable colloidal particles was being investigated. A systematic approach was been incorporated in this work to report different flux rates as per the experimental requirement. The following phases were followed in order throughout this work: A brand-new FO membrane was soaked in deionized water for 30 min before initiating the fouling experiment, the required solids as per the experimental requirement for both the feed solution (FS) and draw solution (DS) were weighed. After weighing the solids, both solutions were prepared as follows: the solids of the NaCl solution were dropped in using a funnel into a 2L volumetric flask, and topped up to the mark with Deionized Water (DW). For the Bentonite solution, a starting amount of 500mL DW were poured in a beaker, the stirrer was run at 850 rpm speed, and a tiny amount of Bentonite were added slowly every 30 secs, the stirrer was left for an extra 10 min when the total required weight was added to the DW. Later, the concentrate was dropped in using a funnel into a 2L volumetric flask, and topped up to the mark with DW. Then, the main FO compartment was prepared for the experiment as follows: the FO cross-flow unit knobs were unfastened, the top part compartment was removed, then, some DW were sprinkled in between the two rings area of the base compartment to lower down the friction between the bottom compartment of the cross-flow unit and the membrane sheet. Later, the FO coupon where active layer is facing the FS (AL-FS Mode) was carefully inserted over the base compartment, again, some DW were sprinkled over the membrane surface, before sliding in the top part compartment once again, finally the knobs were tightened to close the FO unit. As for the circulating

pumps, the pump's head lever was rotated towards left to open the tube enclosure in order to insert the connecting tube, then, the pump's head lever was rotated towards right to close the tube enclosure. Before initiating the experiment, the rotating speed was set to either 300 mL/min for fouling experiments or 450 mL/min for physical backwashing. It is worth mentioning that the flow direction was set to co-current (i.e. towards the cross-flow unit) throughout this study. To conclude the unit preparation, the solutions were poured in their respected containers and both solutions were initially weighed, then, the data logger software was initiated to keep track of the feed solution weight during the experiment. To startup the system, both circulating pumps were initiated at the same time (3 hrs for fouling experiments, and 1 hr for physical backwashing experiments). After the suggested experiment duration has passed, both solutions were weighed again, the datalogging software session was ended, and finally both pumps were stopped at the same time. For physical backwashing experiments, DW were dropped in both containers, the membrane was facing the draw solution (AL-DS Mode). For pH adjusting experiments, the Bentonite solution was poured in a container, then, the solution was mixed using the stirrer while adding the acid (1M HCl), pH readings were taken constantly until the desired pH value was reached, the pH adjusted solution was poured in the feed solution container, and the FO fouling experiment was initiated. Finally, for coagulant doping experiments, the required amount of coagulant was weighted, then, the coagulant was then rapidly mixed (850 rpm) with the feed solution which contained the Bentonite colloidal particles for 10 min. Later, the stirrer rotational speed was set back to 500 rpm, and the FO fouling experiment was initiated.

Note: A datalog extract of datalogging software and sample calculations are provided in the appendices.

Chapter 4: RESULTS AND DISCUSSION

4.1 Effect of Bentonite Concentration and Draw Solution Salinity

4.1.1 Effect of Process Parameters

The main aim of this objective is to study the coupled effect of Bentonite and the salinity of the draw solution. The concentration of Bentonite suspensions was experimented out against different draw solution concentrations to figure out the impact of varying salinity on the process performance. At the early beginning, some baseline experiments were carried out to investigate the behavior of the FO process with a variable salt concentration. Pure water with no traces of Bentonite were dragged across the membrane to the draw solution side throughout this batch of experiments. The baseline experiments have shown that the higher the salinity is the higher the flux, which can be attributed to the osmotic pressure across the membrane. When the salt concentration was set to 1M, the flux value was 13.20 LMH, similarly the flux values of 1.5M and 2M were 16.20 LMH and 18.26 LMH respectively. The results have also shown that increasing the salinity from 1M to 1.5M has enhanced the water flux by 22.73%, also, increasing the salinity from 1.5M to 2M has enhanced the water flux by 12.72%. The varying concentration of salt starting from 1M up to 2M has enhanced the water flux by 38.33%. It is worth mentioning that all the related studies have utilized FO coupons provided by HIT, and in this study Aquaporin FO membranes were used, which shows a potential in protein-based membrane when comparing the reported data. In the second batch of experiments, the feed solution had a constant Bentonite concentration of 0.5 g/L over the course of multiple trials with varying salt concentrations. The experiments have shown that adding a

Bentonite concentration of 0.5 g/L has resulted in a similar trend to the baseline experiments (i.e. increase in salinity enhances the water flux). The starting salt concentration was 1M, the flux value was 12.89 LMH, similarly the flux values of 1.5M and 2M were 15.86 LMH and 18.01 LMH respectively. The results have also shown that increasing the salinity from 1M to 1.5M has enhanced the water flux by 23.04%, also, increasing the salinity from 1.5M to 2M has enhanced the water flux by 12.93%. The varying concentration of salt starting from 1M up to 2M has enhanced the water flux by 13.58%. However, the water flux values were found to be slightly lower than the baseline experiments when the Bentonite colloids were added to the feed solution. The water flux value of 1M salinity was lower by 2.35% compared to the baseline value, also, both values of 1.5M and 2M were lower than their respected baseline values by 2.10% 1.37% respectively. Despite the fact that the reported values are lower than the baseline experiments, the increase of draw solution salinity has contributed in a higher water flux in a similar manner to the baseline values with an increase of 23.02% when the salinity of the draw solution has elevated from 1M to 1.5M, and an increase of 13.58% resulted from the increase of the chemical strength of the draw solution from 1.5M to 2M. The flux was found to be enhanced by 39.72% when increasing the salinity of the draw solution from 1M to 2M which is comparable to reported value of the baseline experiments, meaning that dosing the feed solution with a trace amount of Bentonite colloids (0.5 g/L) has a minimal effect on the FO membrane performance. For 1 g/L Bentonite feed solution, at 1M NaCl draw solution the water flux value was found to be 12.34 LMH. Similarly, the flux values of 1.5M and 2M were 15.43 LMH and 15.94 LMH respectively. The results have also

shown that increasing the salinity from 1M to 1.5M has enhanced the water flux by 25%, also, increasing the salinity from 1.5M to 2M has enhanced the water flux by 3.36%. The first two values are in line with the reported values of both the baseline value and the second batch of experiments where 0.5 g/L of Bentonite colloidal particles were added to the feed solution. The water flux value of 1M salinity was lower by 6.5% compared to the baseline value, also, the value of 1.5M salinity was lower by 4.78% which fall in a comparable margin with their baseline counterparts. However, the value of 2M salinity is clearly showing a downgrade of the FO system performance. Despite the fact that the water flux value is improving at higher concentration values of NaCl, when this particular value is put in comparison against the baseline value, it can be clearly seen that the water flux has dropped this time round. Comparing to the reported value, the filtration rate has decreased by 12.69%. By running the next set of experiments, where the concentration of Bentonite colloidal particles in the feed solution is 2 g/L, it was found that the last experiment from the previous batch was a turning point, and the water flux rate continued to drop. Yet, comparing them at a local scale, the last batch of experimental results were increasing when the ionic strength of the draw solution is increasing. The water flux values of draw solution concentrations of 1M, 1.5M, and 2M are 10.95 LMH, 11.23 LMH, and 11.84 LMH respectively. Increasing the salinity from 1M to 1.5M of the draw solution has enhanced the flux rate by 2.52% and increasing the salinity from 1.5M to 2M has also continued to enhance the flux by 5.40%, meaning that increasing the salt concentration from 1M to 2M has elevated the filtration rate by 8.05%. However, on a global scale, all of these results are sharply lower than their baseline results counterparts. For 1M water flux, the value has

dropped by 20.55%, also, for both of 1.5M and 2M, the flux values have dropped by 44.26% and 54.22% respectively. It should be noted that for each set of experiments three runs were considered and the average value of the three runs was taken with an absolute relative error not exceeding 10%. The aforementioned results are illustrated in Figure 17.

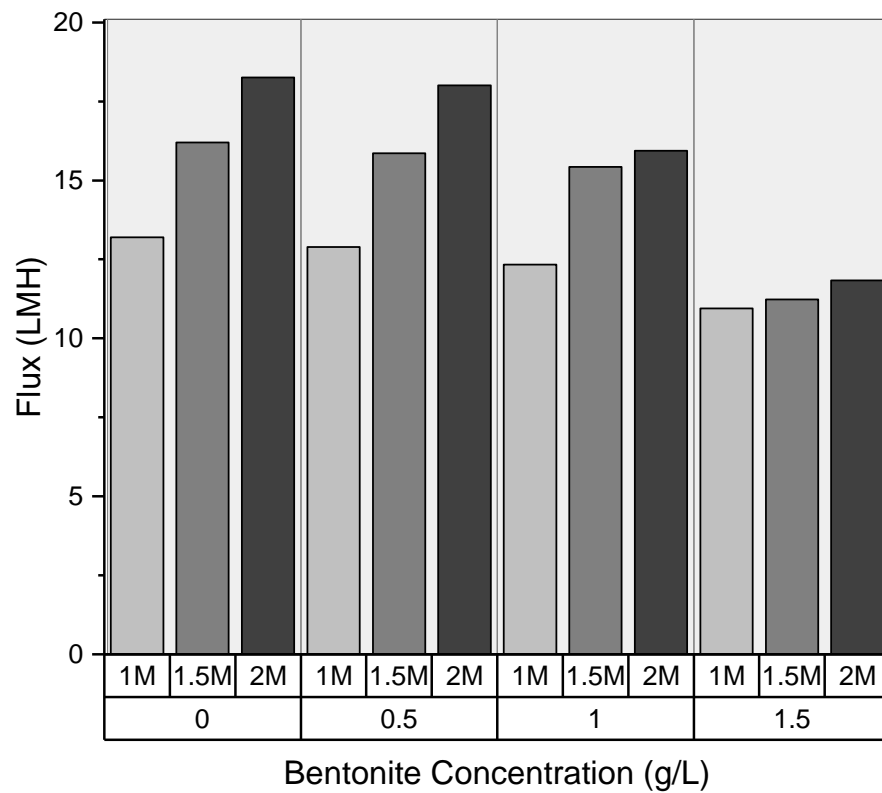
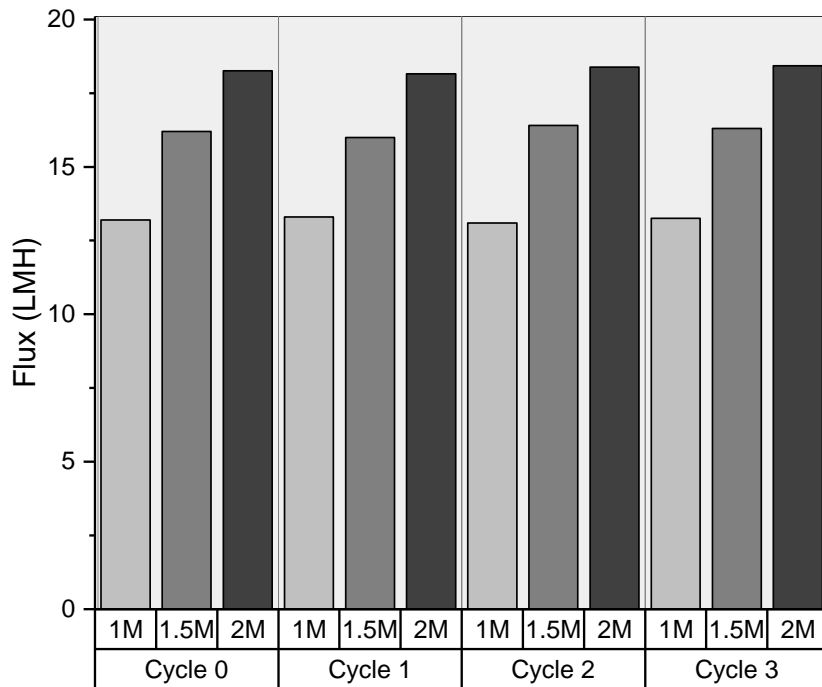


Figure 17. Bentonite-flux plot.

4.1.2. Physical Backwashing

Physical backwashing was found to perfectly recover the flux rates over multiple cycles after running the fouling experiments. The flux rate values were compared against the baseline water flux values. The values of water flux of 1M-draw solution over cycle 0 through cycle 3 were 13.20 LMH, 13.30 LMH, 13.10 LMH, and 13.26 LMH respectively. Cycle 1 was reported to have the highest flux rate among the other cycles in comparison to the baseline water flux. It has the lead over the baseline value with a marginal increase of 0.75%. Also, 1.5M-draw solution water flux values of cycle 0 through cycle 3 were 16.20 LMH, 16.00 LMH, 16.41 LMH, and 16.30 LMH. Cycle 2 was reported to have the highest flux rate among the other cycles in comparison to the baseline water flux. It has the lead over the baseline value with a marginal increase of 1.28%. Similarly, water flux values of 2M-draw solution of cycle 0 through cycle 3 were 18.26 LMH, 18.16 LMH, 18.38 LMH, and 18.43 LMH. Cycle 3 was reported to have the highest flux rate among the other cycles in comparison to the baseline water flux. It has the lead over the baseline value with a marginal increase of 0.92%. In cycle 0, the increase of the draw solution salinity has increased the water flux by 22.73% when elevating the draw solution strength from 1M to 1.5M. Also, the water flux was further enhanced by 12.72% when increasing the salinity from 1.5M to 2M. Hence, the water flux has increased by 38.33% due to altering the draw solution salinity from 1M up to 2M. Further, in cycle 1, the increase of the draw solution salinity has increased the water flux by 20.30% when elevating the draw solution strength from 1M to 1.5M. Also, the water flux was further enhanced by 13.50% when increasing the salinity from 1.5M to 2M. Hence, the water flux has increased by 36.54% due to altering

the draw solution salinity from 1M up to 2M. In cycle 2, the increase of the draw solution salinity has increased the water flux by 25.27% when elevating the draw solution strength from 1M to 1.5M. Also, the water flux was further enhanced by 12% when increasing the salinity from 1.5M to 2M. Hence, the water flux has increased by 40.30% due to altering the draw solution salinity from 1M up to 2M. Finally, in cycle 3, the increase of the draw solution salinity has increased the water flux by 22.92% when elevating the draw solution strength from 1M to 1.5M. Also, the water flux was further enhanced by 13.07% when increasing the salinity from 1.5M to 2M. Hence, the water flux has increased by 39% due to altering the draw solution salinity from 1M up to 2M. It can be clearly seen that physical backwash of the membranes after running the fouling experiments has fully restored the water flux with no signs of performance loss in any of the experimental cycles. Furthermore, the increase of water flux values due to changing the draw solution strength per cycle was matching to reported values of the baseline experiments as seen in Figure 18, meaning that the fouling experiments has neither changed the performance of the FO membrane nor altering its composition.

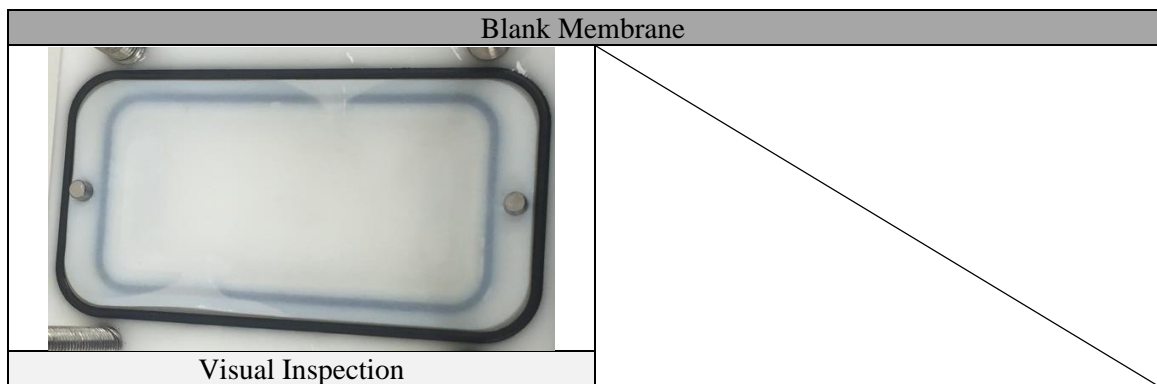


Different Concentrations of NaCl over Multiple Cycles

Figure 18. Physical backwash effect on FO.

4.1.3. Membrane Morphology

To further investigate the membrane morphologies before and after experiments, the membranes were cut and scanned by SEM (SEM, Philips model XL30E, USA). The SEM images of the blank membrane were taken beforehand to have an idea how a virgin FO membrane would look like, the membrane was cut and both the top view and cross section view were taken. From the images, it can be clearly seen how an aquaporin-based FO membrane looks like, and how the material matrix looks like all over the flat membrane sheet when looking at the top side. Also, a cross sectional image would help establishing a clear vision of the arrangement of the pore structure across the membrane layers, namely, active layer and support layer. Finally, a digital photo of the physical membrane was taken, and it was compared against the fouled membranes after the fouling experiments were running as seen in Figure 19.



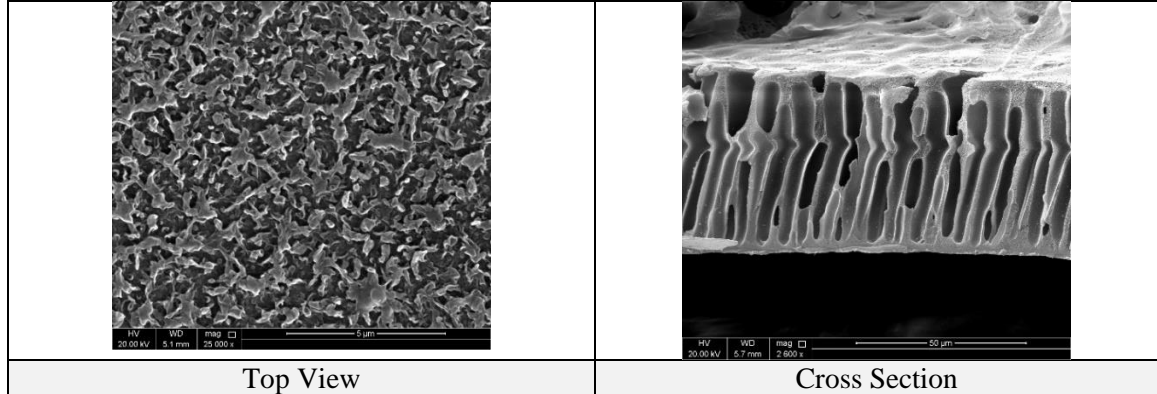
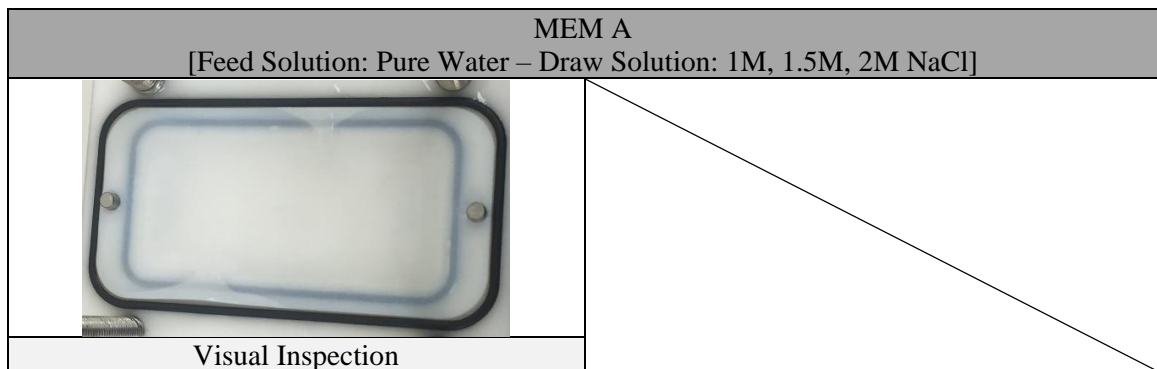


Figure 19. FO blank membrane morphology.

In MEM A, it was found that running fouling experiments with no traces of Bentonite in the feed solution has no visual impact on the membrane sheet. The digital photo when comparing against the virgin membrane looks similar with no precipitations of any kind on top of the membrane surface. Also, the top view SEM image of the fouled membrane has also confirmed the observation of the visual inspection, where the membrane surface of both the virgin membrane and the fouled membrane looks identical. Finally, the cross-sectional SEM image has revealed that the pores of the flat sheet membrane are clean and no signs of irreversible fouling can be observed.



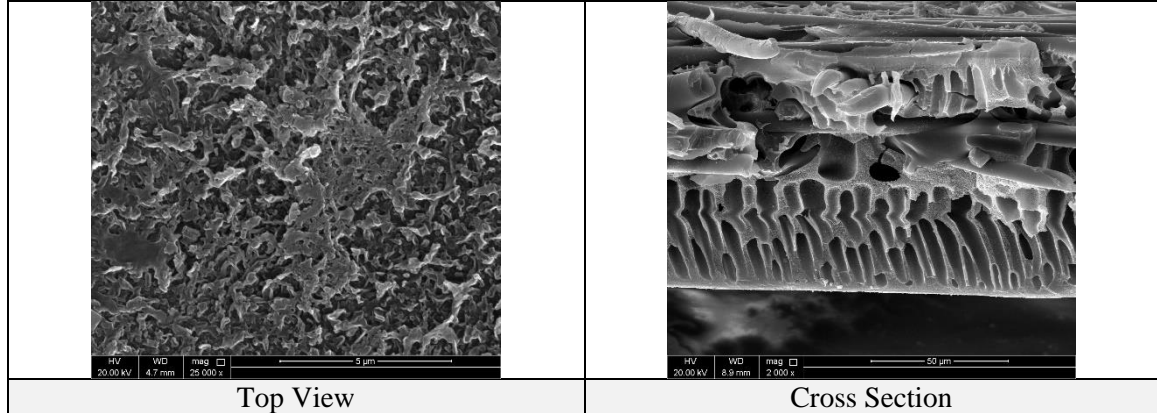
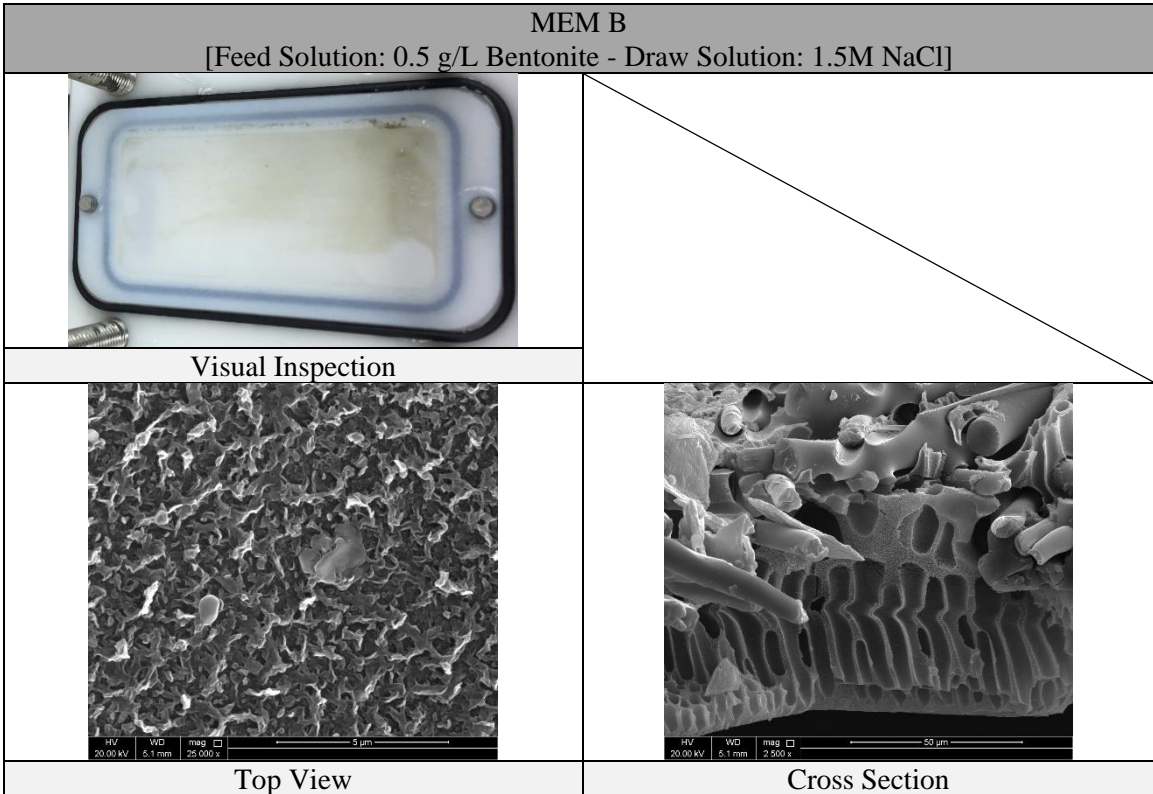
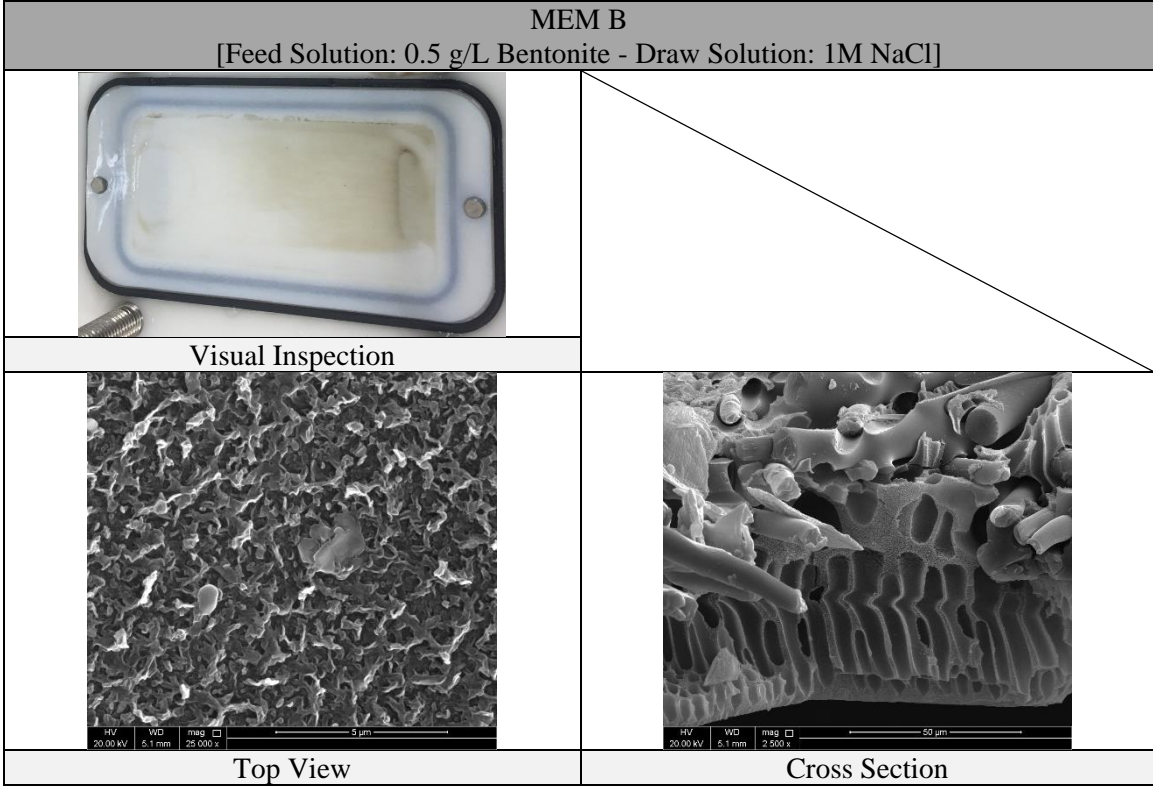


Figure 20. MEM A morphology.

In MEM B, Figure 21, a tiny amount of Bentonite colloidal particles has added to the feed solution (0.5 g/L), and the ionic strength were elevated after each fouling experiment, starting with 1M of NaCl all the way up to 2M of NaCl. it was found that running fouling experiments with 0.5 g/L of Bentonite in the feed solution has showed a precipitation of Bentonite suspensions over the flat sheet membrane surface. The digital photo when comparing against the virgin membrane has confirmed the effect of the introduction of Bentonite on top of the membrane surface. It was also found that the intensity of Bentonite colloids over the membrane surface has elevated when the salinity has increased. However, the top view SEM image of the fouled membrane compared to the membrane surface of both the virgin membrane looks identical which once again confirm the efficiency of physical back washing of the FO membranes. Finally, the cross-sectional SEM image has revealed that the pores of the flat sheet membrane are clean and no signs of permanent fouling can be observed.



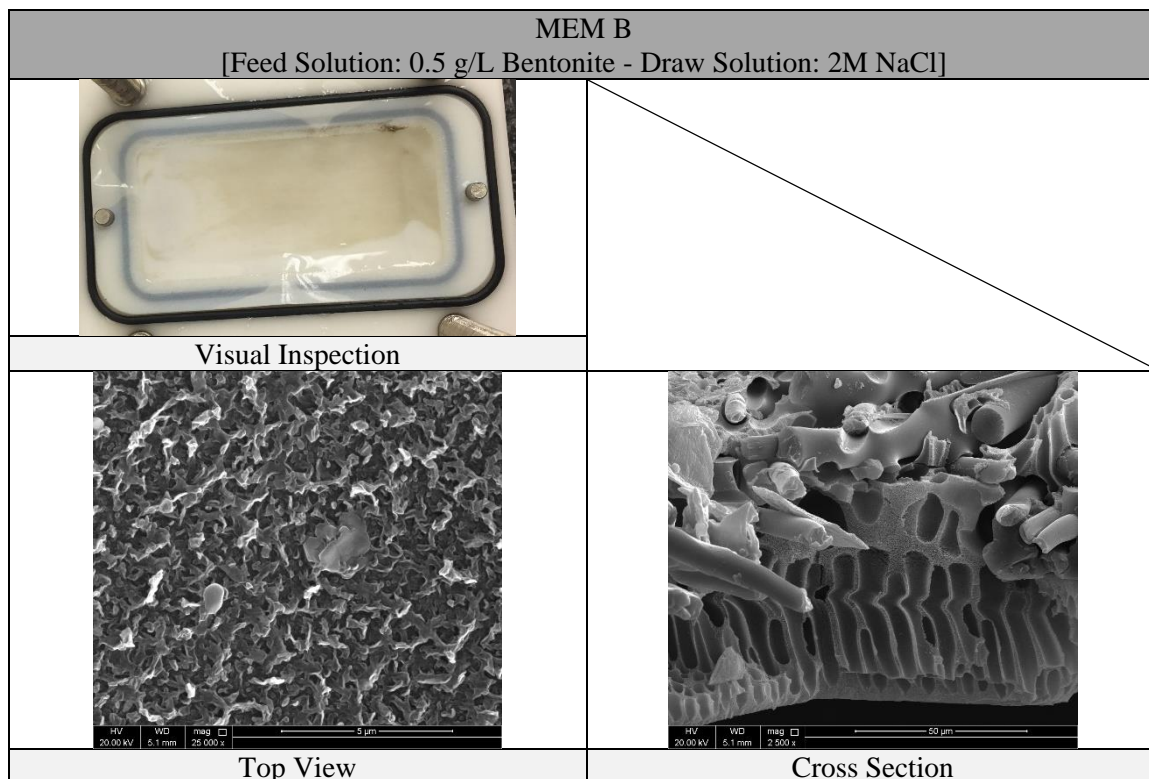
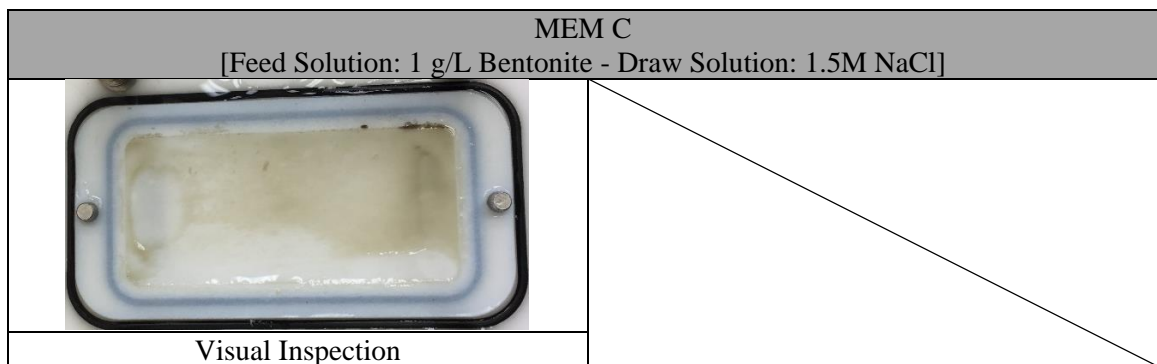
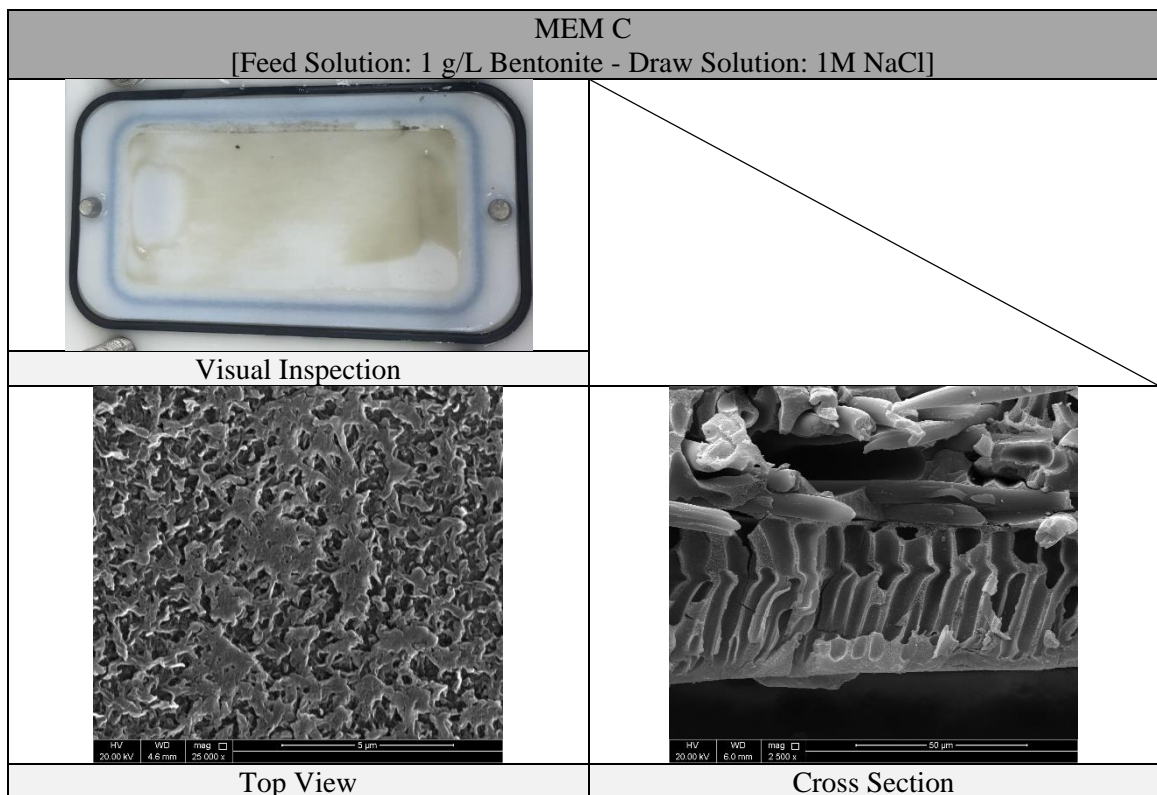


Figure 21. MEM B morphology.

In MEM C, Figure 21, an amount of Bentonite colloidal particles has added to the feed solution (1 g/L), and the ionic strength were elevated after each fouling experiment, starting with 1M of NaCl all the way up to 2M of NaCl. it was found that running fouling experiments with 1 g/L of Bentonite in the feed solution has showed a precipitation of Bentonite suspensions over the flat sheet membrane surface. The digital photo when comparing against the virgin membrane has confirmed the effect of the introduction of Bentonite on top of the membrane surface. It was also found that the intensity of Bentonite colloids over the membrane surface has elevated when the salinity has increased. However, the top view SEM image of the fouled membrane compared to the membrane surface of

both the virgin membrane looks identical which once again confirm the efficiency of physical back washing of the FO membranes. Finally, the cross-sectional SEM image has revealed that the pores of the flat sheet membrane are clean and no signs of permanent fouling can be observed.



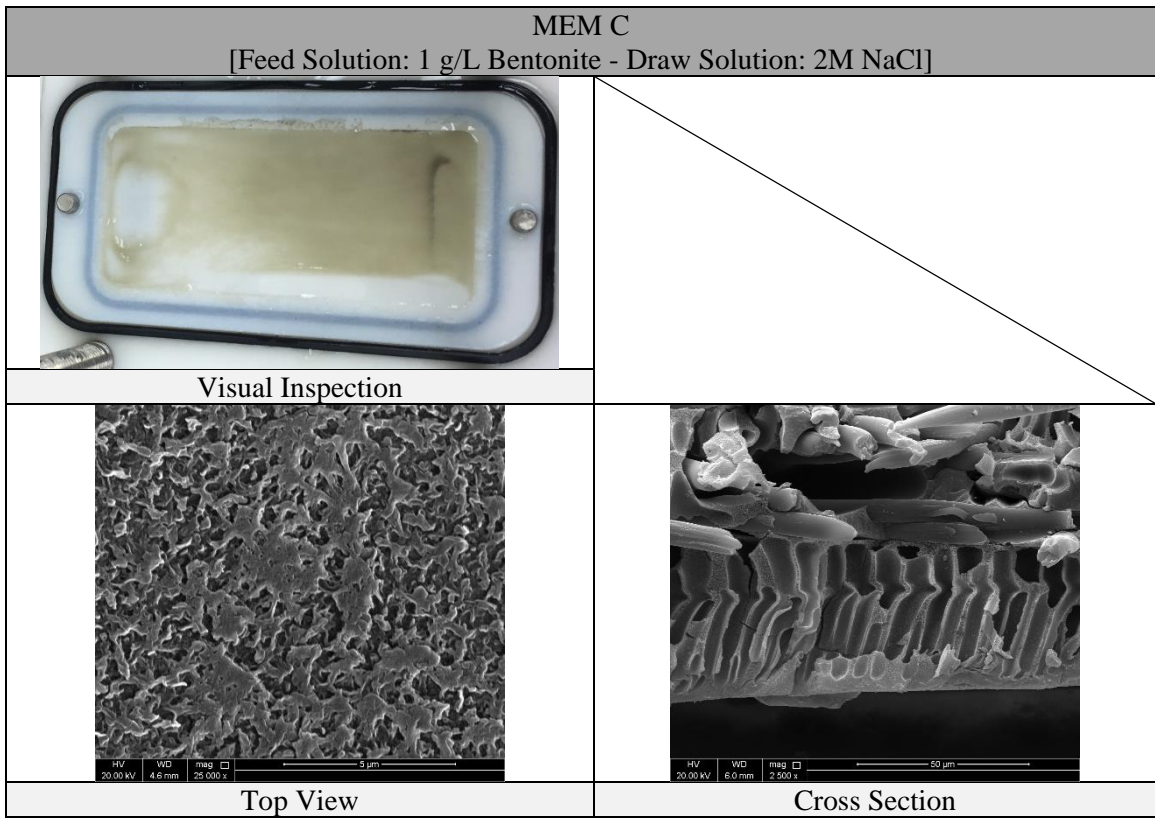
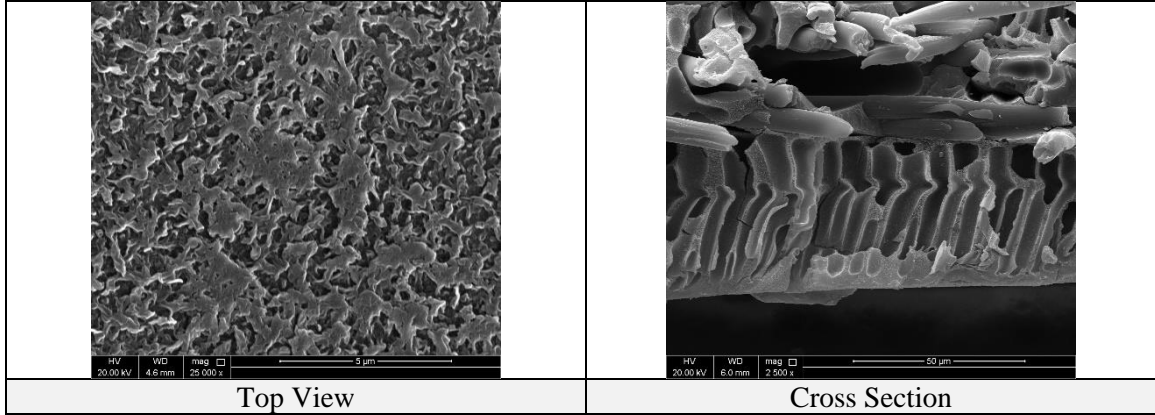
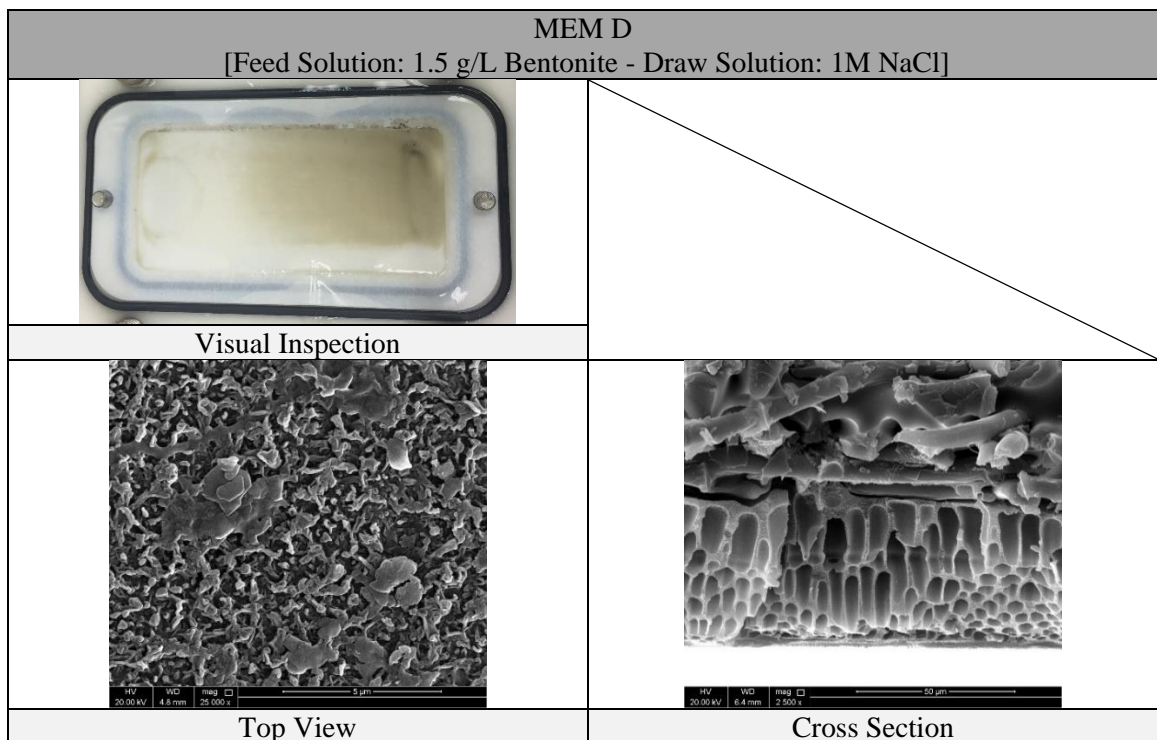


Figure 22. MEM C morphology.

In MEM D, Figure 23, an amount of Bentonite colloidal particles has added to the feed solution (1.5 g/L), and the ionic strength were elevated after each fouling experiment,

starting with 1M of NaCl all the way up to 2M of NaCl. it was found that running fouling experiments with 1.5 g/L of Bentonite in the feed solution has showed a precipitation of Bentonite suspensions over the flat sheet membrane surface. The digital photo when comparing against the virgin membrane has confirmed the effect of the introduction of Bentonite on top of the membrane surface. It was also found that the intensity of Bentonite colloids over the membrane surface has elevated when the salinity has increased. However, the top view SEM image of the fouled membrane compared to the membrane surface of both the virgin membrane looks identical which once again confirm the efficiency of physical back washing of the FO membranes. Finally, the cross-sectional SEM image has revealed that the pores of the flat sheet membrane are clean and no signs of permanent fouling can be observed.



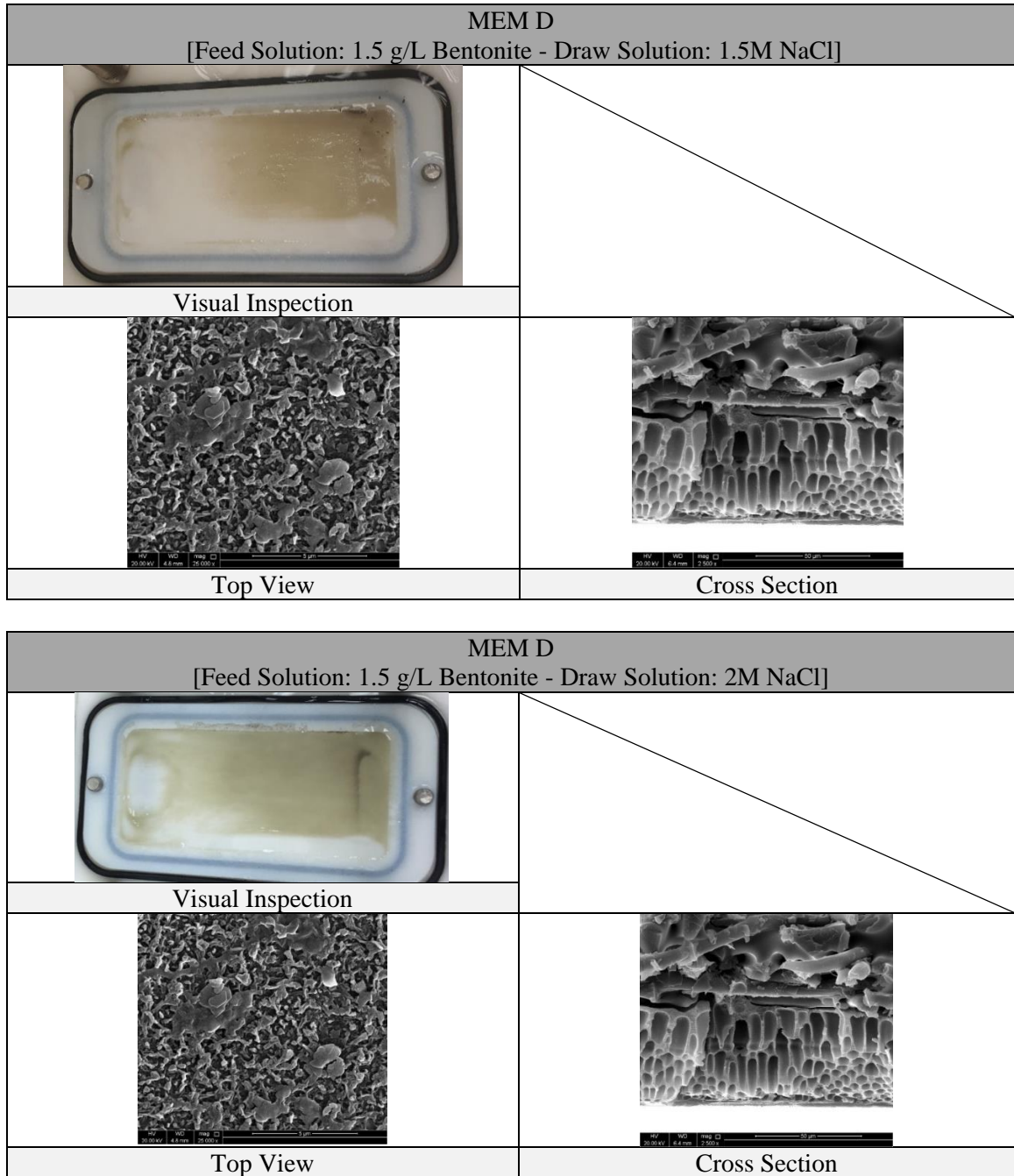


Figure 23. MEM D morphology.

Finally, the SEM images of used membranes when compared against virgin FO membranes have showed that the pores are clean and the Bentonite colloids have not been entrapped inside the pores causing an irreversible fouling. Starting with a Blank to Mem D has showed the visual inspection of each membrane by the end of fouling experiment and the SEM scan after running all the related batch of experiments. By comparing both the visual inspections and the SEM scans, it can be clearly seen that the drop in the flux rates throughout the experiments were due the Bentonite colloidal particles buildup over the membrane surface.

4.2 Effect of pH Value of Bentonite Colloidal Particles on FO Performance

4.2.1. Effect of Process Parameters

In this objective, the alteration of the pH value of Bentonite colloidal particles is investigated. Naturally, the pH value of Bentonite after mixing it with deionized water is around 9. Two additional fouling experiments were run for different pH values, namely, pH = 7 and pH = 4 in order to study the effect of varying pH values on the performance of FO membrane system. As reported earlier, the water flux value of separating 1 g/L Bentonite suspensions against 1.5M draw solution is 15.43 LMH. In the first experiment, the value was lowered to pH = 7, then, a fouling experiment was run to evaluate the separation performance of the system, the same operating conditions were set to compare the results against the natural pH value of Bentonite colloids. It was found that lowering the value to pH = 7 has affected the water flux rate, the separation rate of this fouling experiment is 14.91 LMH. Changing the pH value to 7 has dropped the water flux rate by 3.37%. Similarly, in the second experiment, the value was lowered to pH = 4, then, a fouling experiment was run to evaluate the separation performance of the system. It was found that lowering the value to pH = 4 has also affected the water flux rate in a similar pattern to pH = 7, the separation rate of this fouling experiment is 13.97 LMH. Changing the pH value from 7 to 4 has further dropped the water flux rate by 6.30%. Altering the pH value from 9 to 4 was found to drop the initial water flux value by 9.46%. In Figure 24, it can be clearly seen that the flux rate pattern against the pH value is related, meaning that the higher the pH value, the higher the flux. In other words, altering the natural chemical nature of Bentonite suspensions has not improved the performance of the FO system.

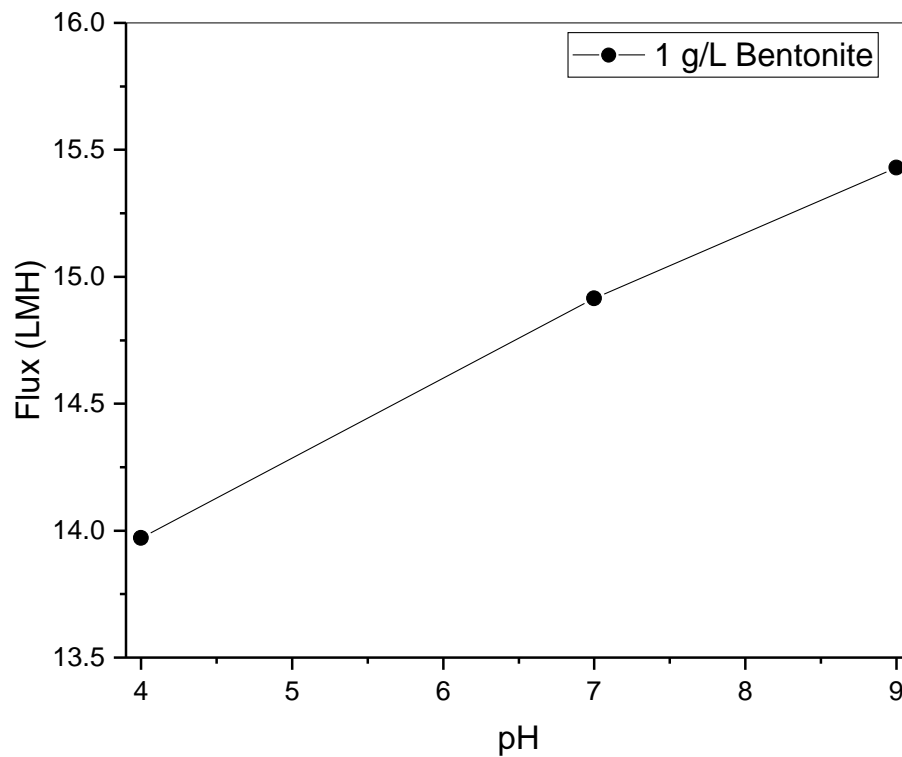


Figure 24. pH effect on FO performance.

This phenomenon has been reported in the literature, altering the chemical nature of the colloidal particles was found to change their interaction between each other. At lower pH values, the particle-particle interaction was found to be face-edge configuration (instead of face-face configuration found at higher pH values) where this particular arrangement leads to floc the colloidal particles which becomes challenging to separate them from the

given solution as the pH value approaches more acidic nature, hence, resulting in lower filtration rate as seen in Figure 25 [101].

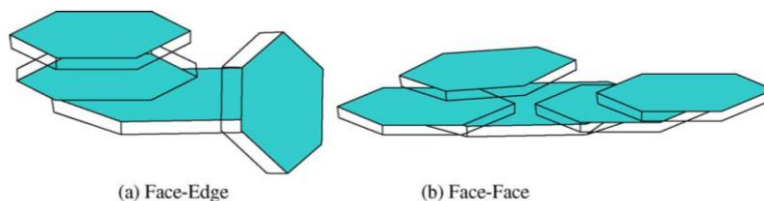


Figure 25. Colloidal particle main configurations [101].

4.2.2. Membrane Morphology

After running the fouling experiments, the membranes used were cut and their morphologies were directly analyzed by Scanning electron microscopy. Starting with a Blank, MEM C, MEM E, and MEM F, is showing the visual inspection of each membrane by the end of fouling experiment and the SEM scan after running all the related batch of experiments.

In MEM C, Figure 26, an amount of Bentonite colloidal particles similar to MEM C case has added to the feed solution (1 g/L), the pH value has reported to be pH = 9, and the ionic strength was set to 1M of NaCl. It was found that running fouling experiments with 1 g/L of Bentonite in the feed solution has showed a precipitation of Bentonite suspensions over the flat sheet membrane surface. The digital photo when comparing against the virgin membrane has confirmed the effect of the introduction of Bentonite on top of the membrane surface. However, the top view SEM image of the fouled membrane

compared to the membrane surface of both the virgin membrane looks identical which once again confirm the efficiency of physical back washing of the FO membranes. Finally, the cross-sectional SEM image has revealed that the pores of the flat sheet membrane are clean and no signs of permanent fouling can be observed.

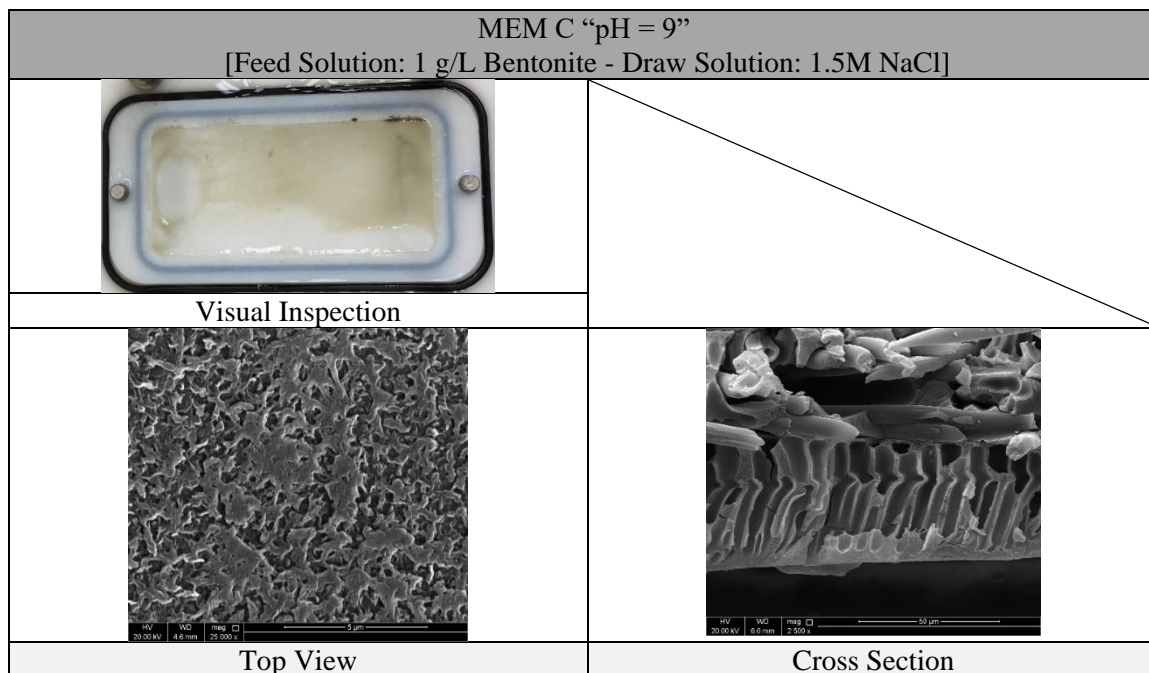


Figure 26. MEM C morphology.

In MEM E, Figure 27, an amount of Bentonite colloidal particles similar to MEM C case has added to the feed solution (1 g/L), the pH value has lowered down to pH = 7, and the ionic strength was set to 1M of NaCl. It was found that running fouling experiments with 1 g/L of Bentonite in the feed solution has showed a precipitation of Bentonite suspensions over the flat sheet membrane surface. The digital photo when comparing against the virgin membrane has confirmed the effect of the introduction of Bentonite on

top of the membrane surface. It was also found that the intensity of Bentonite colloids over the membrane surface has elevated when the pH value has increased compared to MEM C. However, the top view SEM image of the fouled membrane compared to the membrane surface of both the virgin membrane looks identical which once again confirm the efficiency of physical back washing of the FO membranes. Finally, the cross-sectional SEM image has revealed that the pores of the flat sheet membrane are clean and no signs of permanent fouling can be observed.

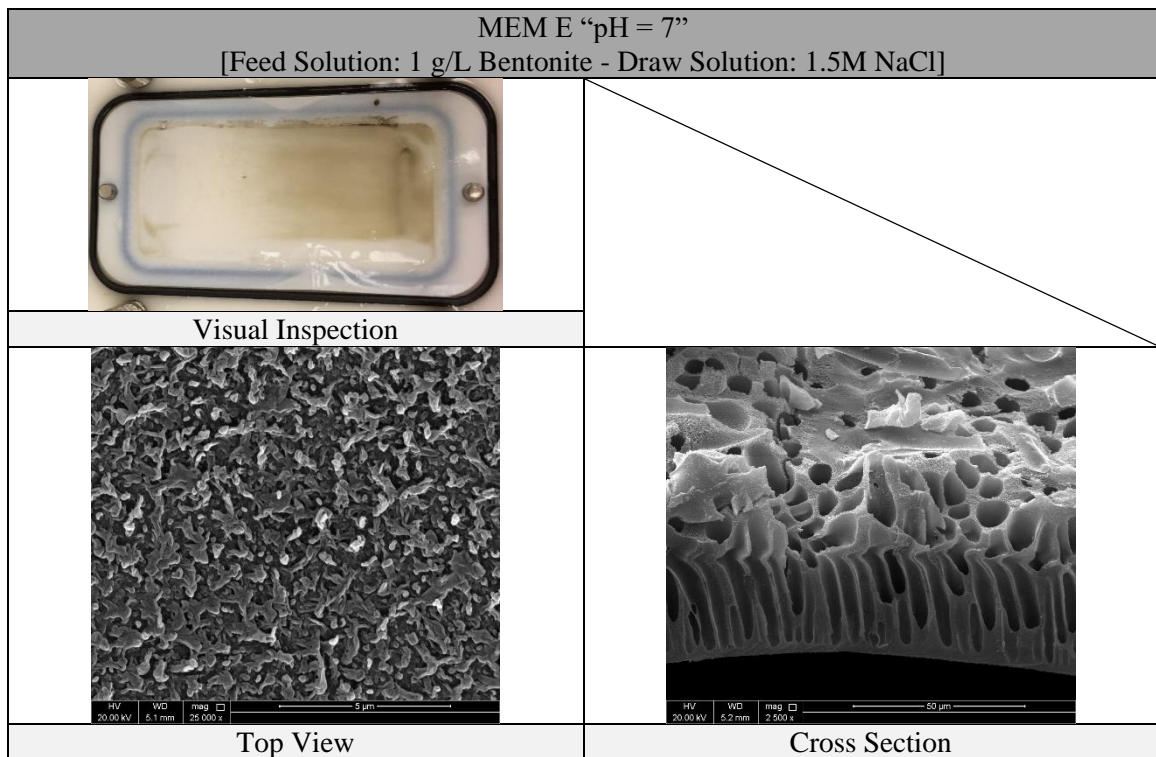


Figure 27. MEM E morphology.

In MEM F, Figure 28, an amount of Bentonite colloidal particles similar to MEM C case has added to the feed solution (1 g/L), the pH value has lowered down to pH = 7, and the ionic strength was set to 1M of NaCl. It was found that running fouling experiments with 1 g/L of Bentonite in the feed solution has showed a precipitation of Bentonite suspensions over the flat sheet membrane surface. The digital photo when comparing against the virgin membrane has confirmed the effect of the introduction of Bentonite on top of the membrane surface. It was also found that the intensity of Bentonite colloids over the membrane surface has elevated when the pH value has increased compared to MEM C. However, the top view SEM image of the fouled membrane compared to the membrane surface of both the virgin membrane looks identical which once again confirm the efficiency of physical back washing of the FO membranes. Finally, the cross-sectional SEM image has revealed that the pores of the flat sheet membrane are clean and no signs of permanent fouling can be observed.

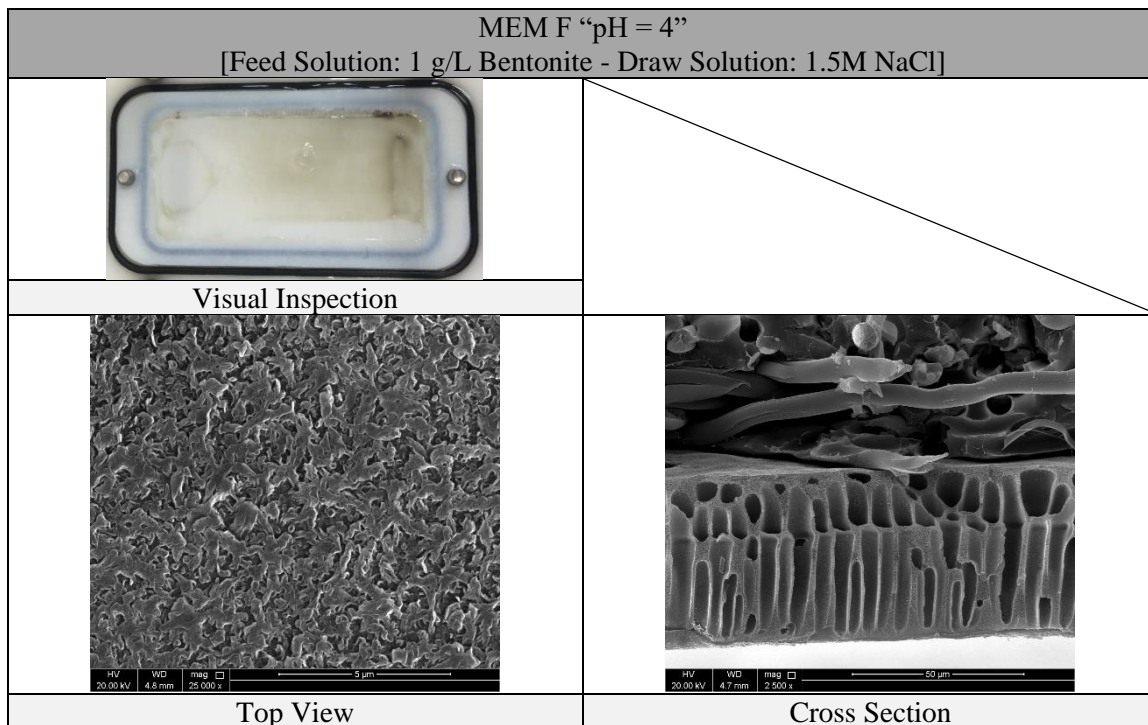


Figure 28. MEM F morphology.

Finally, the obtained images were compared against virgin FO coupon and have showed that the pores are clean and the Bentonite colloids have not been entrapped inside the pores causing an irreversible fouling.

4.3 Effect of Coagulant Addition to the Feed Solution

4.3.1. Effect of Process Parameter

In this part of work, the effect of adding a coagulant to the feed solution is studied. Three different doses of Sodium Chloride have been added to the feed stream which contained Bentonite colloids prior initiating the fouling experiments. The three doses were 0.001M, 0.01M, and 0.1M of NaCl. Each of these fouling experiments are then compared to the baseline experiments where no traces of coagulant are mixed with the Bentonite solution. As reported earlier, the filtration rate of 1 g/L of Bentonite suspensions against 1.5M NaCl draw solution is 15.43 LMH. In the first experiment, 0.001M of NaCl was rapidly mixed with 1 g/L of Bentonite solution for 10 min, then, the fouling experiment was started. The water flux resulting from doping the feed solution with a coagulant is 13.89 LMH, this has dropped the flux rate by 10.01%. Secondly, the coagulant has stepped up to 0.01M of NaCl which has rapidly mixed with 1 g/L of Bentonite solution, the water flux resulting from doping the feed solution with a coagulant is 11.23 LMH, this has dropped the flux rate by 19.14% compared to the first case. Finally, 0.1M of NaCl was added to 1 g/L of Bentonite solution, the filtration rate of this case is 9.43 LMH, a further drop of 16.03% was caused by adding the coagulant. Ultimately, the highest dose of NaCl coagulant has dropped the water flux rate comparing to the baseline experiment by 38.89%. It can be clearly seen that adding a coagulant to the feed solution has affected the FO system performance, the doping process has resulted in a trend where the higher the coagulant dose, the lower the flux rate. Further, the values of Zeta Potential and Turbidity of the feed solution were also investigated. Both values have reinforced the aforementioned findings

of the efficiency of doping the feed solution with a coagulation agent and its impact on the flux rate, and how adding a coagulant with similar characteristics to the draw solution might lead to. All the values with their respected coagulant dose are tabulated in Table 9.

Table 9. Effect of Coagulant Concentration on Membrane Performance

Coagulant Concentration [M]	Flux [LMH]	Zeta Potential [mV]	Turbidity [NTU]
0	15.43	-33	182
0.001	13.89	-30	177
0.01	11.23	-27.6	177
0.1	9.43	-18	57.7

Both values were also matching with their water flux counterparts. For Zeta Potential values, Figure 29, it was found that adding a coagulant has elevated the ZP values, where the higher the coagulant dose, the higher the ZP of the feed solution which means that the coagulation process as a pretreatment aid was found to have a positive impact on the colloidal particles. In other words, NaCl as a coagulation agent is efficient on destabilizing the Bentonite suspensions, and improving the feed solution quality. Nonetheless, at higher ZP values the water flux has dropped. Also, the Turbidity measurements of the feed solution can be linked to the water flux values as seen in Figure 30. It was found that these values were in sync with the filtration rate values, where the higher the turbidity of the solution, the higher the flux rate. This pattern can be attributed to the net osmotic pressure across the membrane sheet, meaning that mixing NaCl with

Bentonite suspensions in the feed solution has resulted in lower net osmotic pressure, consequently, the filtration rate has declined since the driving force of the FO process is the resultant osmotic pressure across the membrane.

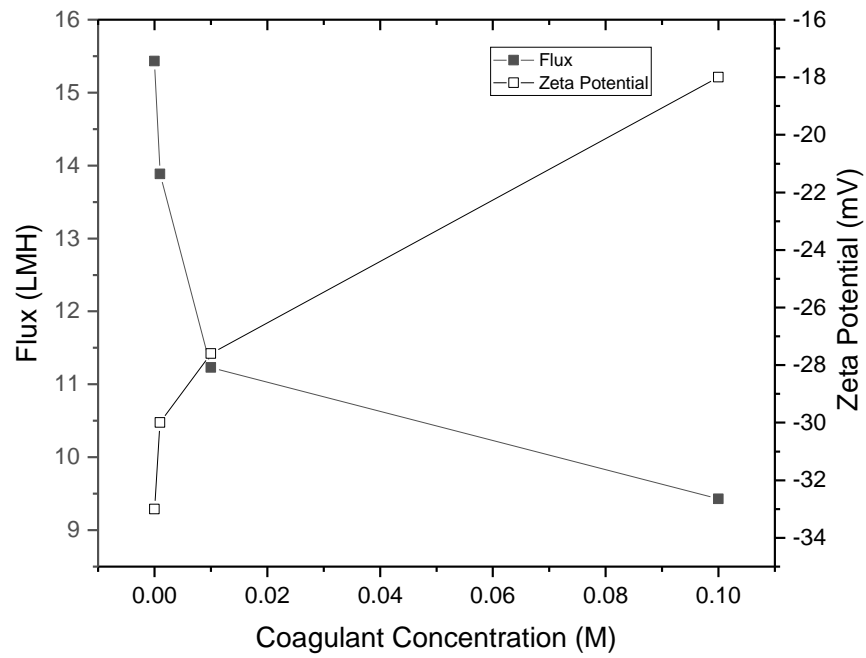


Figure 29. Flux-coagulant-ZP effect.

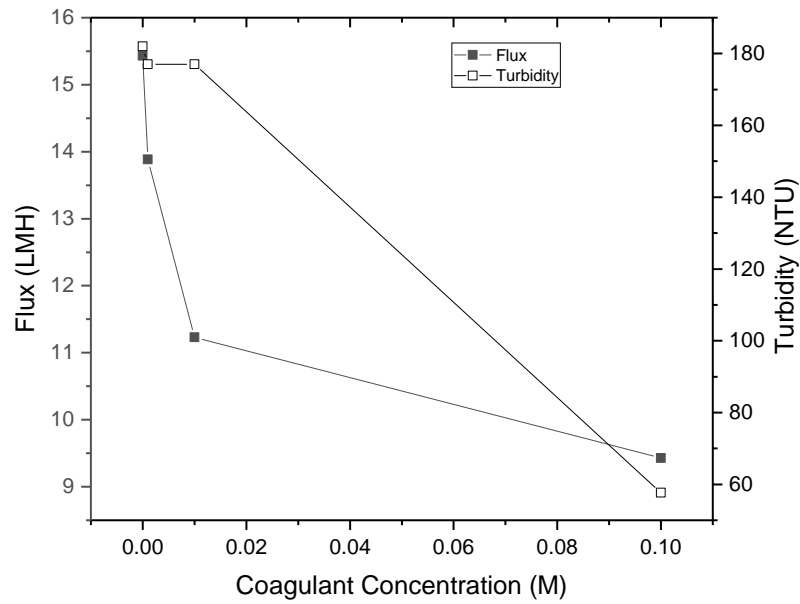
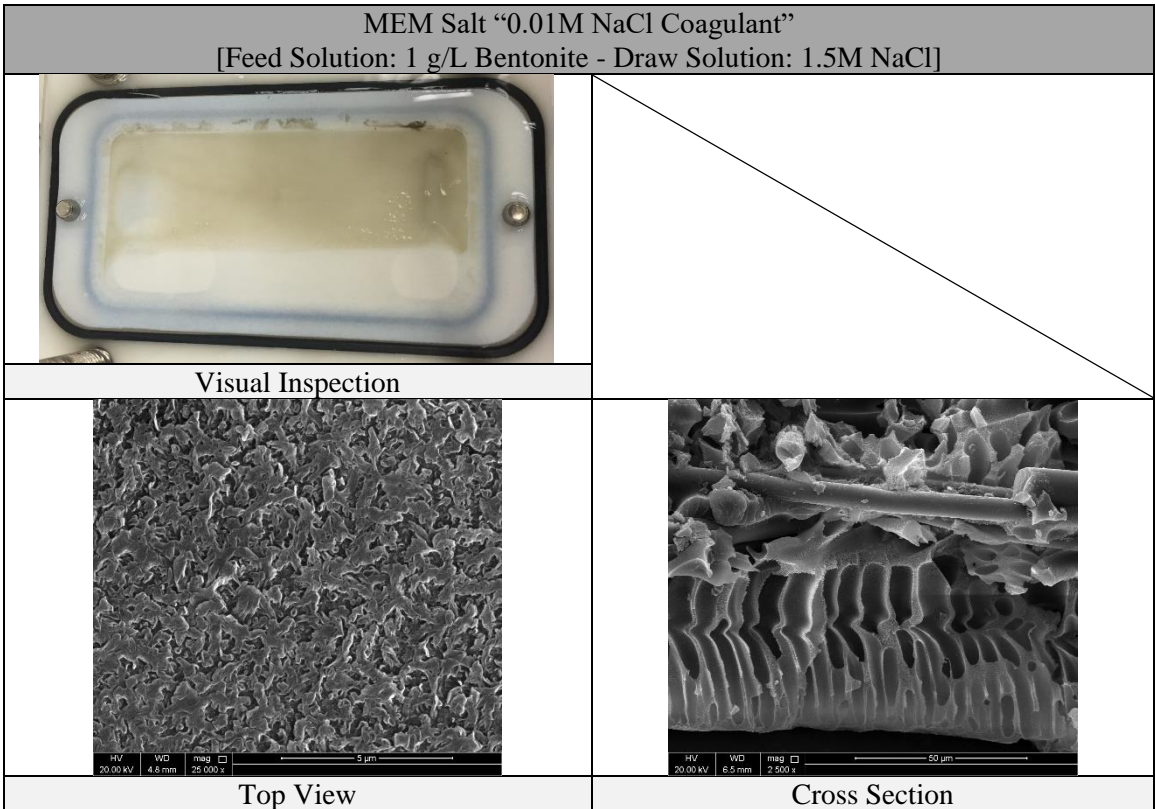
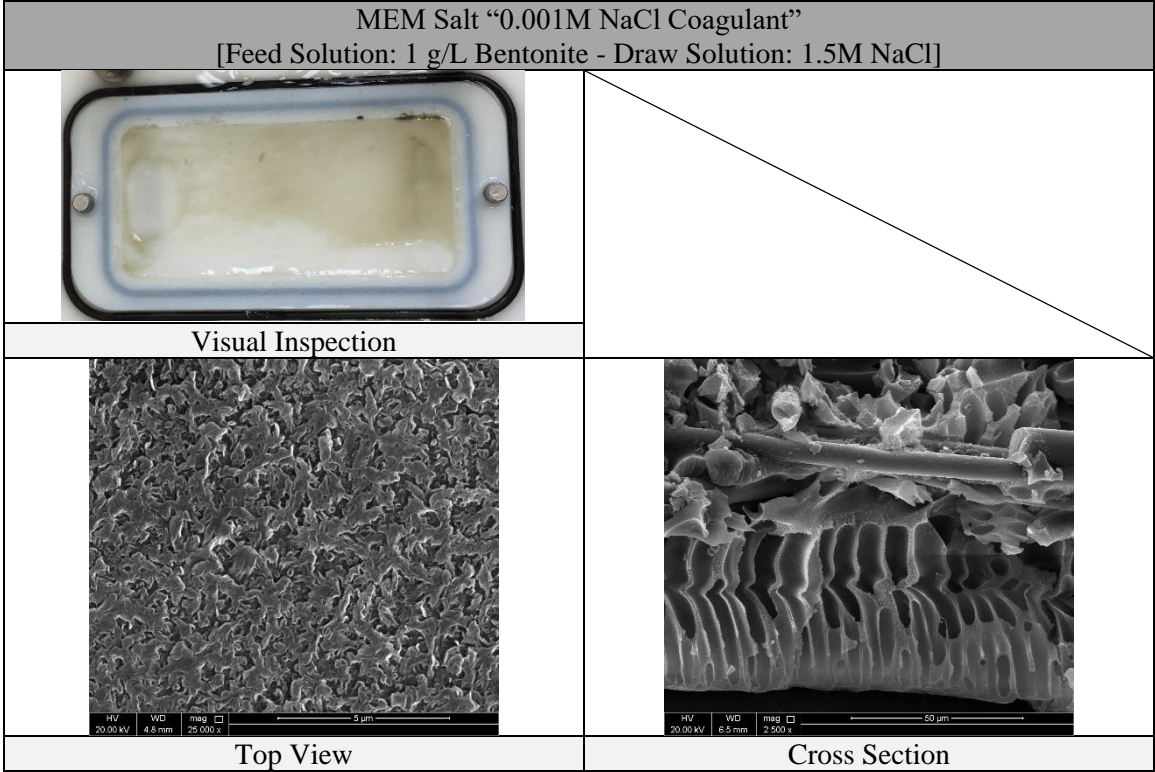


Figure 30. Flux-coagulant-turbidity effect.

4.3.2. Membrane Morphology

After running the fouling experiments, the membranes used were cut and scanned by SEM. In Figure 31, the visual inspection of each membrane by the end of fouling experiment and the SEM scan after running all the related batch of experiments are presented. In MEM Salt, Figure 30, an amount of Bentonite colloidal particles similar to MEM C case has added to the feed solution (1 g/L), adding different doses of NaCl coagulant, and the ionic strength was set to 1.5M of NaCl. It was found that running fouling experiments with 1 g/L of Bentonite in the feed solution has showed a precipitation of Bentonite suspensions over the flat sheet membrane surface. The digital photo when comparing against the virgin membrane has confirmed the effect of the introduction of Bentonite on top of the membrane surface. It was also found that the intensity of Bentonite colloids over the membrane surface has elevated when the coagulant dose is lowered, meaning that adding a coagulant is found to hinder the treatment process. However, the top view SEM image of the fouled membrane compared to the membrane surface of both the virgin membrane looks identical which once again confirm the efficiency of physical back washing of the FO membranes. Finally, the cross-sectional SEM image has revealed that the pores of the flat sheet membrane are clean and no signs of permanent fouling can be observed.



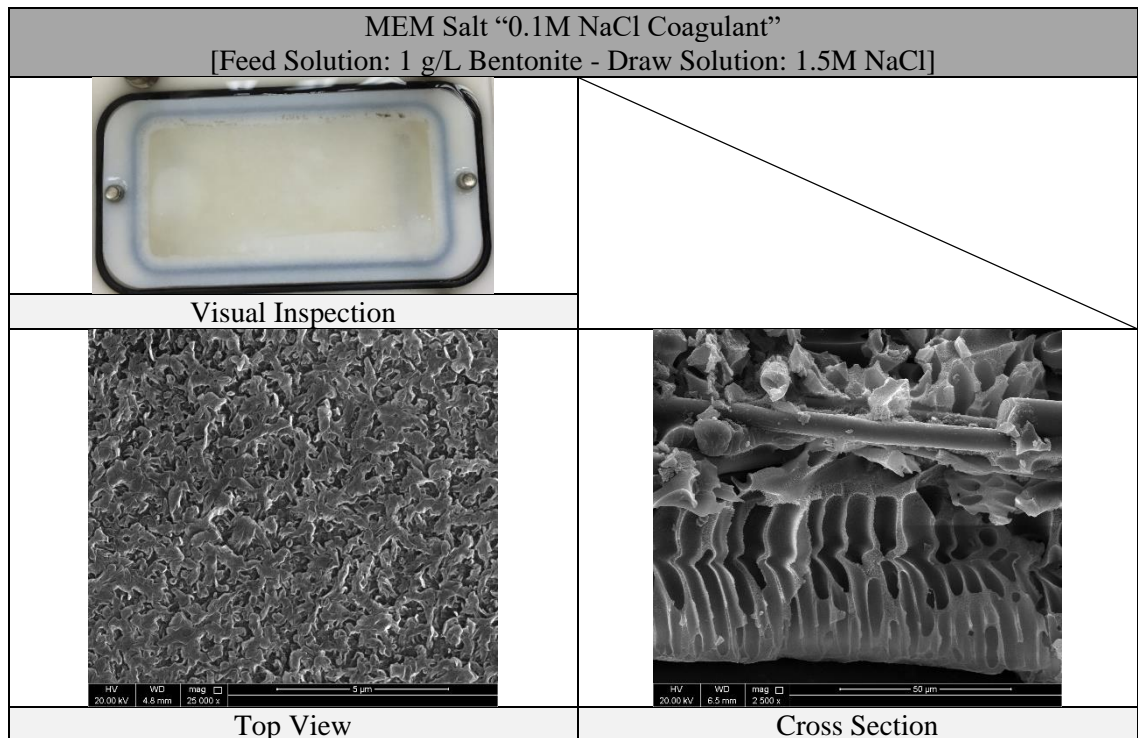


Figure 31. MEM Salt morphology.

The SEM images of used membranes when compared against virgin FO coupon have showed that the pores are clean and the Bentonite colloids have not been entrapped inside the pores causing an irreversible fouling.

Chapter 5: CONCLUSION AND RECOMMENDATIONS

In this work, the performance of FO system in separating highly stable colloidal particles has been investigated. Three main aims were set out of this work, namely, 1) the coupled effect of variable Bentonite (as a highly stable colloidal model) and salt concentrations on the efficiency of the FO process; 2) The effect of varying pH values of the Feed Solution on the separation of the stable colloids; and 3) the behavior of the treatment process after introducing a coagulant to the feed solution. The main findings of this study were as follows:

- Adding a tiny amount of Bentonite colloidal particles (0.5 g/L) has not affect the overall performance of the FO system.
- Physical backwash of FO membranes has completely restored water flux recovery (100% water flux recovery were achieved over 4 cycles).
- Altering the chemical nature of Bentonite suspensions has steadily decreased the flux (i.e. lowering the pH value has affected the filtration rate).
- Adding a coagulant to the feed solution has hindered the separation process (the overall efficiency has sharply declined to 38.89% due to 0.1M NaCl dose).

We would highly recommended investigating different colloidal models e.g. Kaolinite, and further examine the reported results under different operating conditions e.g. feed spacer, variable circulation speeds, and different draw solutions for future research.

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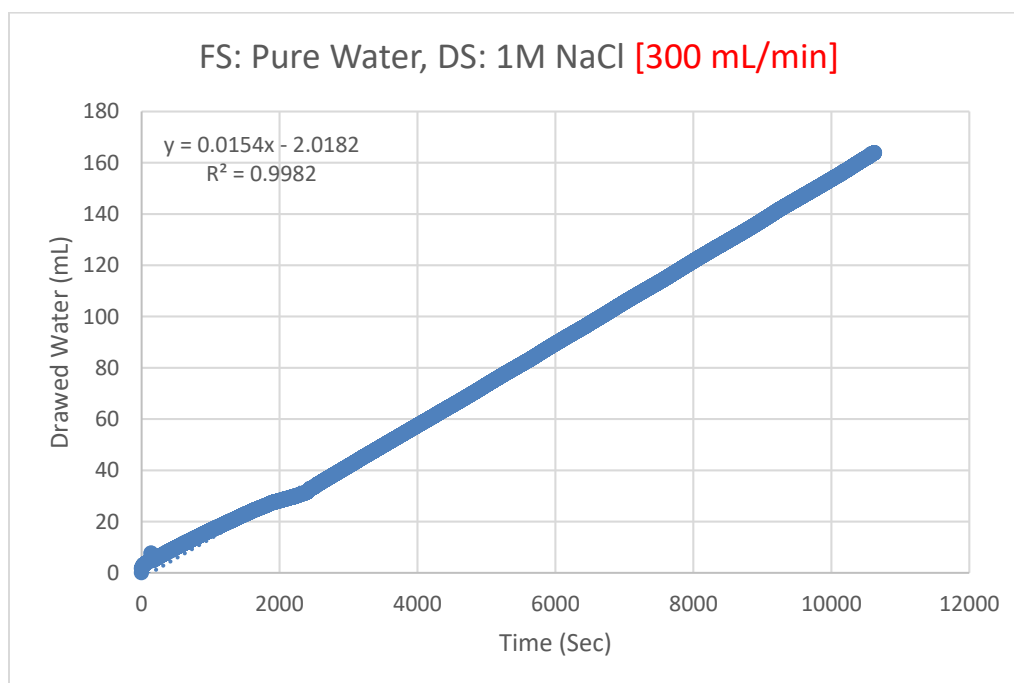
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Appendix A: DATALOG EXTRACT OF EXPERIMENT 1

Reading #	Time (sec)	Weight (g)	Accumulation (mL)
0	0	2153.58	0
1	1	2151.88	1.72
2	2	2152.01	1.59
3	3	2151.91	1.69
4	4	2151.85	1.75
5	5	2151.76	1.84
...
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...
...
...
10625	10625	1991.4	163.82
10626	10626	1991.39	163.83
10627	10627	1991.36	163.86
10628	10628	1991.35	163.87
10629	10629	1991.34	163.88



Appendix B: SAMPLE CALCULATIONS OF WATER FLUX

After performing Experiment A (Data extracts are provided in Appendix A), it was found that the flowrate = $0.0154 \frac{mL}{sec}$, also, the active membrane area $A = 42 \text{ cm}^2$.

$$\text{Water Flux} = 0.0154 \frac{mL}{sec} \times 3600 \frac{sec}{hr} \times \frac{1 \text{ L}}{1000 \text{ mL}} \times \frac{1}{0.0042 \text{ m}^2} = 13.2 \text{ LMH}$$