Testing of synthetic biological membranes for forward osmosis applications

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Commercially available forward osmosis membranes have been extensively tested for human space flight wastewater treatment. Despite the improvements achieved in the last decades, there is still a challenge to produce reliable membranes with anti-fouling properties, chemical resistance, and high flux and selectivity. Synthetic biological membranes that mimic the ones present in nature, which underwent millions of years of evolution, represent a potential solution for further development and progress in membrane technology. Biomimetic forward osmosis membranes based on a polymeric support filter and coated with surfactant multilayers have been engineered to investigate how different manufacturing processes impact the performance and structure of the membrane. However, initial results of the first generation prototype membranes tests reveal a high scatter in the data, due to the current testing apparatus set up. The testing apparatus has been upgraded to improve data collection, reduce errors, and to allow higher control of the testing process.

Nomenclature

ARC	=	Ames Research Center
CAD	=	computer aided design
CFD	=	computational fluid dynamics
FO	=	forward osmosis
GPM	=	gallons per minute
LPM	=	liters per minute

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NaCl	=	sodium chloride
NASA	=	National Aeronautics and Space Administration
OA	=	osmotic agent
PID	=	proportional derivative integrative
PSI	=	pounds per square inch
P&ID	=	piping & instrumentation diagram
TOC	=	total organic carbons
VOC	=	volatile organic carbons

I. Introduction

MEMBRANE contactors are devices that keep in contact two phases and allow liquid/liquid mass transfer without dispersion of one phase within another. This is accomplished by passing the fluids on opposite sides of a semi-permeable membrane. Being the two phases separated by the membrane, they cannot mix and dispersion phenomena do not occur. The species are transferred from one phase to the other only by diffusion. Membrane contactor technology is used in a range of liquid/liquid and gas/liquid applications in the production of pharmaceuticals, in wastewater treatment, in semiconductor manufacturing, in carbonation of beverages, in metal ion extraction, in volatile organic carbons (VOC) removal from waste gas, and in protein extraction. Membrane contactors are an essential piece of equipment for testing new synthetic biological membranes that have been engineered at NASA Ames Research Center (ARC) to investigate how different manufacturing processes impact the performance and the structure of the membrane itself. These new membranes are intended for water reclamation within forward osmosis (FO) recycling systems. They have been tested using in-house made contactors. However, initial results of the first generation of prototype membranes tests reveal a high scatter in the data, which has been determined to be caused by both the manufacturing technique and the current testing apparatus set up. Thus, a Langmuir Blodgett trough has been purchased to fabricate the new membrane prototypes with precise control of the deposition of multi-layer films of surfactants/lipids/fatty acids on the support layer by controlling the packing density of the monolayer film at the air-solvent interface. In fact, the initial results showed a large difference in the performances of the prototype membranes depending on the person who fabricated them, despite the manufacturing protocol has been carefully followed and reproduced. The critical step of the manufacturing process has been identified in the coating of the support layer, which was done manually. The use of a computer-controlled deposition system should attenuate the human error.
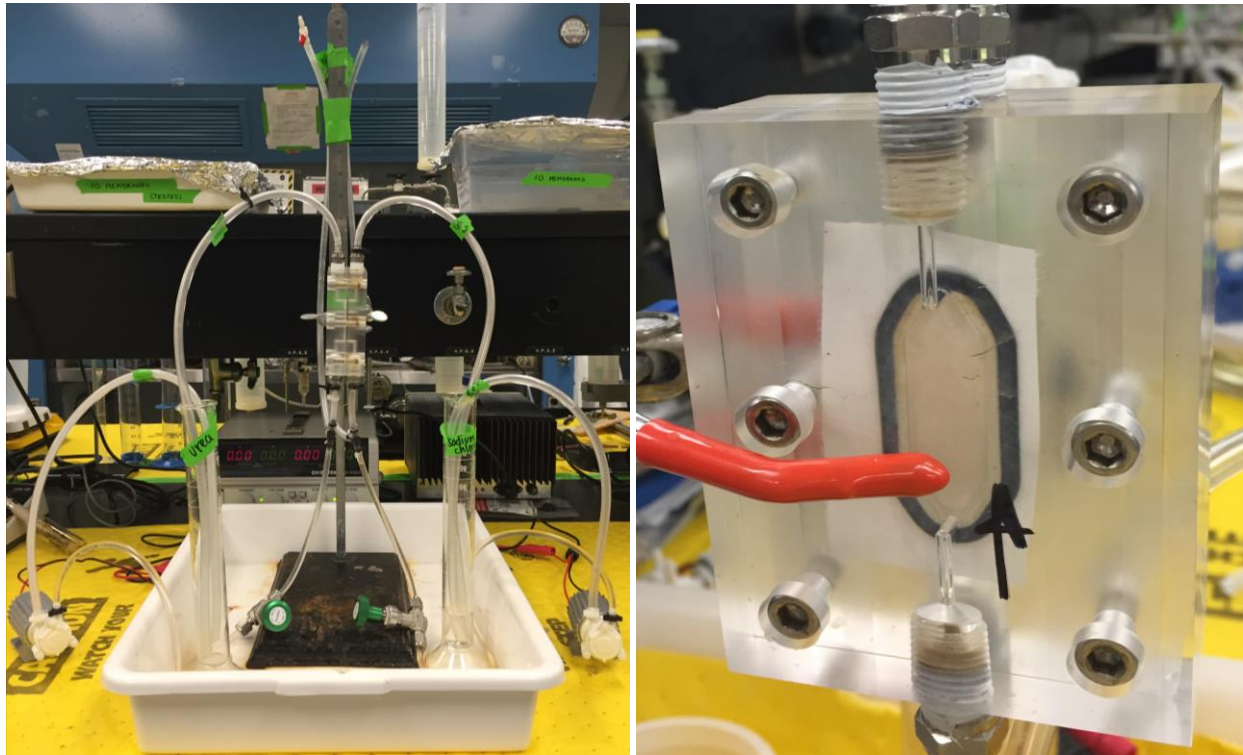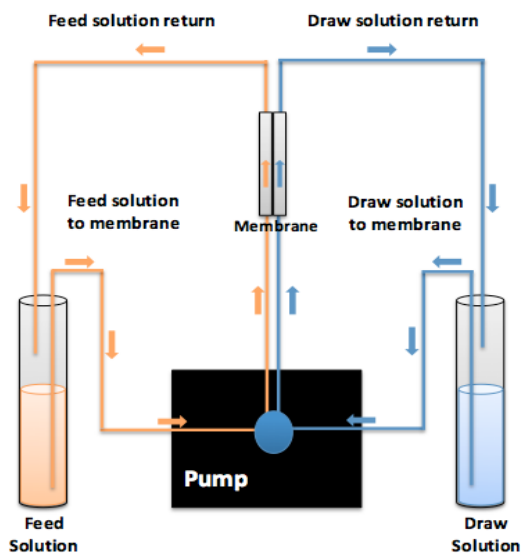

Figure 1. FO testing apparatus at NASA ARC.

Figure 2. Simplified testing apparatus flow diagram.

Moreover, the testing apparatus has been upgraded to improve data collection, reduce reading errors, and to allow a higher control of the testing process by automating the pumps control and data acquisition, and by modifying the membrane contactor. This paper is focused on the upgrade of the testing apparatus.

II. Background

Forward osmosis is a physical phenomenon that allows the transport of water across a selectively permeable membrane from a region of higher water chemical potential to a region of lower water chemical potential. It is driven by a difference in solute concentrations across the membrane itself, which causes a difference in osmotic pressure that allows passage of water but rejects most solute molecules. Reverse osmosis uses hydraulic pressure to oppose, and exceed, the osmotic pressure of an aqueous feed solution to produce treated water. Thus, in reverse osmosis, the applied hydraulic 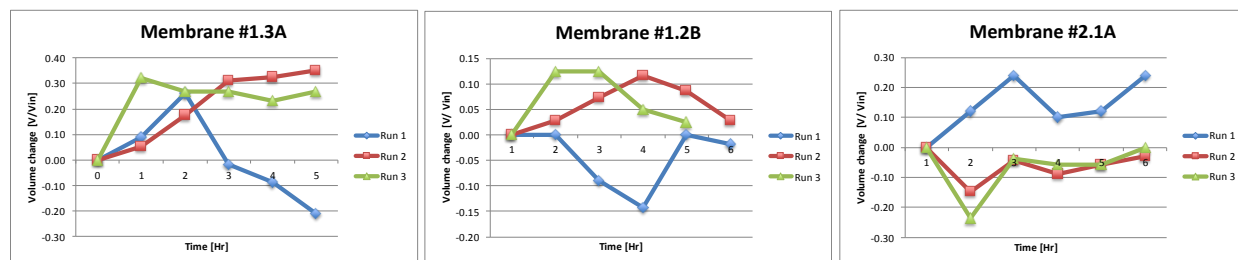pressure is the driving force for mass transport through the membrane. In forward osmosis, the osmotic pressure itself is the driving force for mass transport.

The main advantages of using FO are that it operates at low or no hydraulic pressures, it has high rejection of a wide range of contaminants, and it has a lower membrane fouling propensity compared to pressure-driven membrane processes. The source of the driving force in the FO process is the concentrated solution on the permeate side of the membrane. We used Urea solutions as the feed and a sodium chloride (NaCl) solution as the osmotic agent (OA) because of their high solubility and the simplicity to measure total organic carbons (TOC) rejection and back-flow salt rejection.

Forward osmosis membranes are tested using small laboratory-scale testing apparati, comprised of a membrane contactor, two parastaltic pumps, and two scales that measure the weight of the feed and of the draw solution. The testing appatus used at NASA Ames Research Center are showed in Figure 1 and their flow diagram is showed in Figure 2. The membrane contactor is composed of two almost-symmetric acrylic plates, milled at the NASA Ames machine shop. One of the two acrylic plates has an exagonal groove that hosts an o-ring. The shape of this groove, which has a perimeter of a standardized 1"-diameter o-ring, requires two users to put it in place because of the elasticity of the o-ring that tends to aquire its original circular shape. In some membranes, damage has been visually observed at the end of the test in correspondence of the extremities of the groove, where the o-ring bents the most. Another dameage that has been visually identified on some membranes at the end of their testing was a hole in correspondence of the outlet of the fluid within the membrane contactor. This systematic failure on different membranes in different testing apparati has been attributed to the both the design of the membrane contactor itself and to the peristastic pumps used in the testing apparatus. In fact, the peristaltic pump creates a flow that is not linear but sinusoidal and thus creates fatigue on the membrane surface in correspondence to the outlet of the flow. The design of the membrane contactor, the inner ports of which are perpendicular to the membrane, creates an outlet flow of the fluid that hits the membrane also perpendicularly. Thus, the total pressure on the surface of the membrane in correspondence of that outlet will be maximum , and the gradient of the total pressure around that spot will be extremely high. Five testing apparatus set ups are used in parallel when testing coupons of new forward osmosis membranes. Two of them were initially equipped with peristaltic pumps. The others were equipped with ultra-compact gear pumps from Greylor,

Figure 3. Flow rates, organic of first gen prototype membranes.

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which are rated for a maximum flow rate of 0.38 gallons per minute (GPM) and a maximum pressure of 25 PSI. The membranes tested in the apparati having the gear pumps did not show the failure mode previously described. However, the Greylor gear pumps showed very different flow rates from one unit to the other when operated at the same voltage and amperage. Their performance also varied in function of time, affecting the results of the experiments. The scatter of the flow rates during the run is summarized in Figure 3.

FO membranes are ususaly microporous and hydrophilic. The area of contact of the two phases is ideally located in correspondence of the pores mouths. It is important to carefully control of the pressure difference between the fluids to keep the fluid/fluid interface at the mouth of each pore. The interfacial area can be established at the pore mouth only if the penetration of the draw phase into the pores is prevented. If a critical value of the pressure, called the breakthrough pressure, is exceeded, the draw phase starts to leak into the feed phase. For a particular material, the breakthrough pressure depends on the pore radius, the tortuosity of the membrane along its thickness, the contact angle between the membrane and the fluid, and the osmotic pressure differential. Since membrane pores have an undefined shape related to the tortosity and non-constant pore size along its thickness, the interfacial area is often established within the pores themselves. To keep this configuration and avoid dispersion between the phases, it is necessary to work with pressures of the feed side equal or higher than the draw phase pressure.

III. Materials and Methods

The testing apparatus has been redesigned including automated control of the feed and of the osmotic agent pump based on pressure feedback. The new P&ID is showed in Figure 4. The system is automated using National Instruments' Compact DAQ with LabVIEW software. The PID controller is used to control the pump on the product side (output), based on the differential pressure (input) in between the feed and product membrane inlets, with respect to the feed pump speed. The PID gains (controller gain, integral time, and derivative time) are manually adjusted and then auto-tuned until the error between the setpoint and process variable reaches less than 5%.

The design of the membrane contactor has been investigated using computational fluid dynamic (CFD) simulations looking at how different parameters affect the flow within the contactor. A module with a flat membrane has been chosen because it is easier to build at lab scale and because the membrane replacement is simple and fast. A single flat sheet of membrane is located between two plates that are equipped with inlets and outlets for the feed and draw solution, respectively. For industrial applications, where higher membrane area per volume ratio are desirable, spiral wound configurations and hollow fibers are preferred, due to their higher packing density. The goals pursued when designing the new membrane contactor are to guarantee a constant performance of the module for all its lengh and to
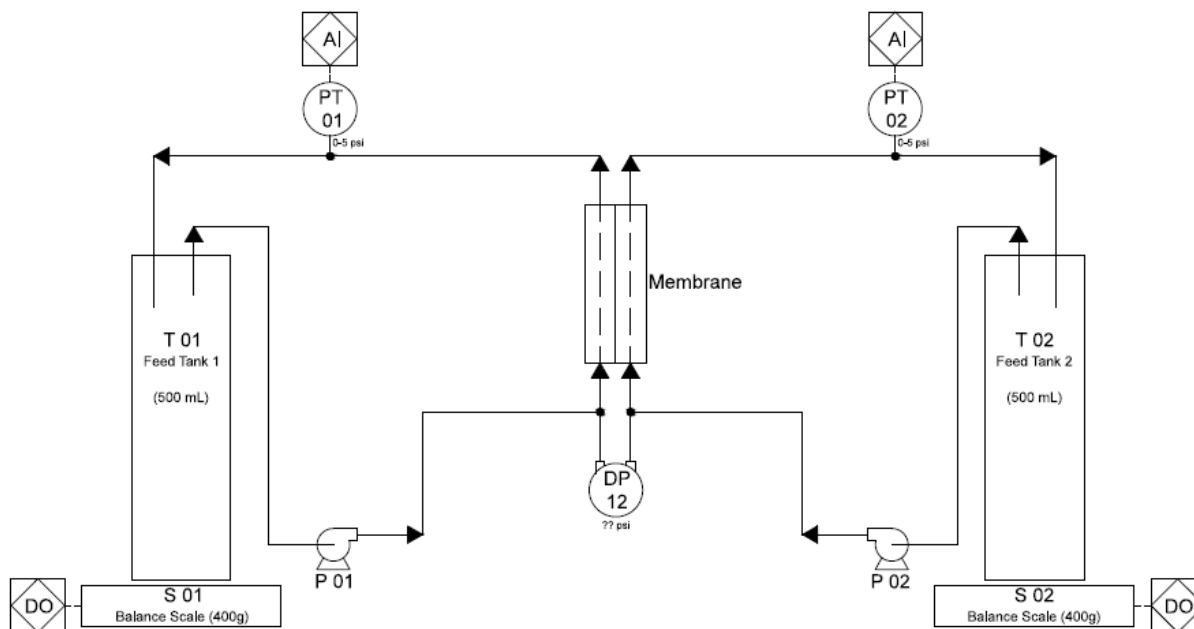

Figure 4. P&ID.

work with low pressure drops. One major limitation in membrane contactors is the non-uniform flow that occurs because of channeling, bypassing, mixing, entry region phenomena, and presence of stagnant zones, which lead to the calculation of mass transfer coefficients that often differ from scale-up systems. The type of flow inside the module plays also an important role. In fact, crossflow designs lead to higher mass transfer coefficients than co/counter-current flows, but pressure drops increase too. In our case, the most conservative case of co-current flow has been chosen in order to calculate the minimum performances of the membranes and to minimize the formation of air bubbles within the module during the run. Often turbulence promoters are added to reduce the boundary layer resistance, however, from past observations, it has been noticed that they often become traps for air bubbles, which affect the interfacial area of the membrane. Thus, they have not been included in the CFD model. An extensive literature exists on hollow fiber module design, and on how different parameters such as packing density, fibers lengh and diameters, operative flowrates and pressures affect the performances of the module. Several mathematical models have also been developed to analyze the performance of hollow fiber modules. However, this models cannot be used for flat sheet membrane contactors. Due to the lack of mathematical models, a computer-aided analysis has been performed to simulate the behavior of the tri-dimensional flow within the membrane contactors that have been used during all the membrane testing performed until now, using as boundary conditions of the CFD simulation the same flow rates and pressures used in the lab during the actual testing. The results of this CFD analysis have then been compared to visual observations of the tri-dimensional flow of the fluid within the module, adding a blue methylene dye to the feed solution when the system was running at full regime. This comparison has the objective of verifying that CFD software can be used as a reliable tool to design a new membrane contactor with desired performances.

Figure 5 shows the result of the CFD simulation of the membrane contactor used for all lab membrane testing at NASA ARC. The boundary conditions imposed in the simulation are the actual flow rate at the inlet of the contactor, which is 0.4 liters per minute (LPM) and atmospheric pressure at the outlet, which corresponds with good approximation to reality. The software used for this CFD simulations has the ability to adapt the computational mesh to the solution during the calculation. This means that the mesh cells are splitted in the high-gradient flow regions and are merged in the low-gradient flow regions, ensurig better accuracy during the calculation. However, since the model geometry is mostly composed of areas of curvature, thin walls, and narrow channels, the total number of cells created
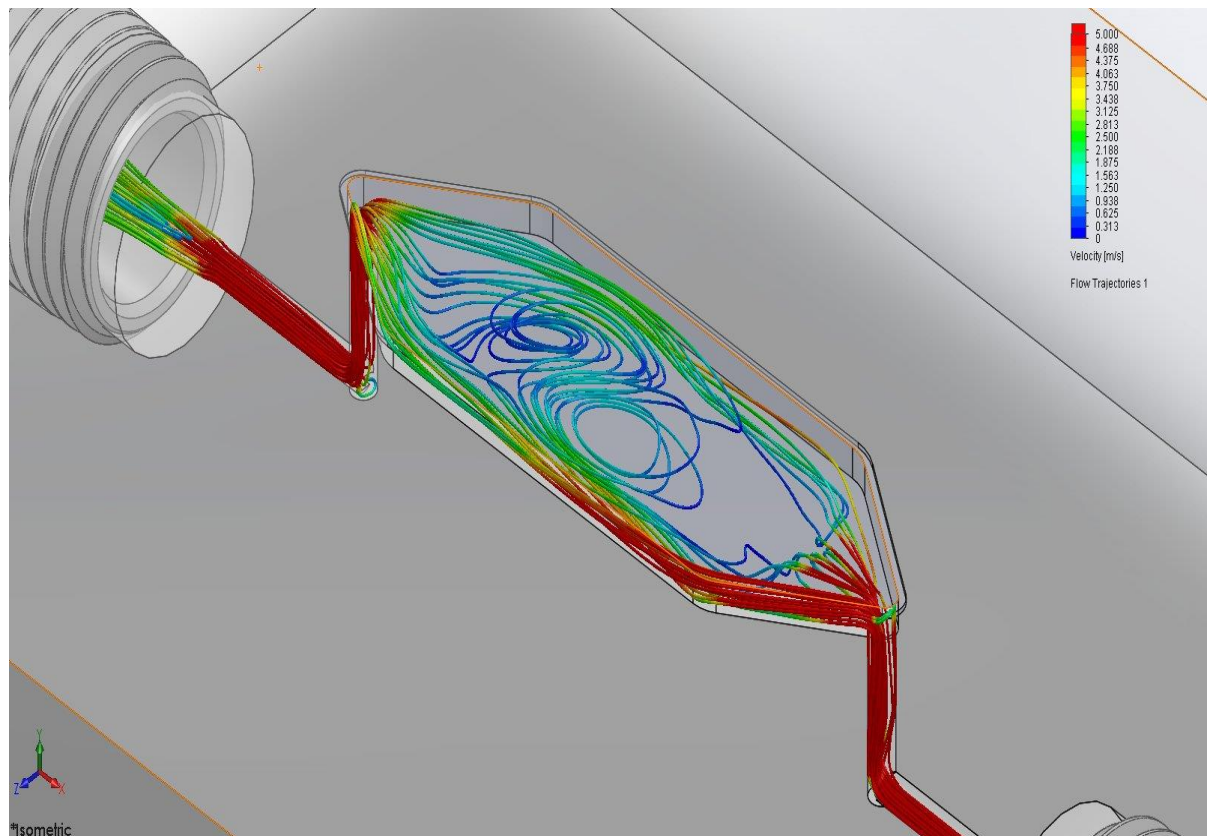

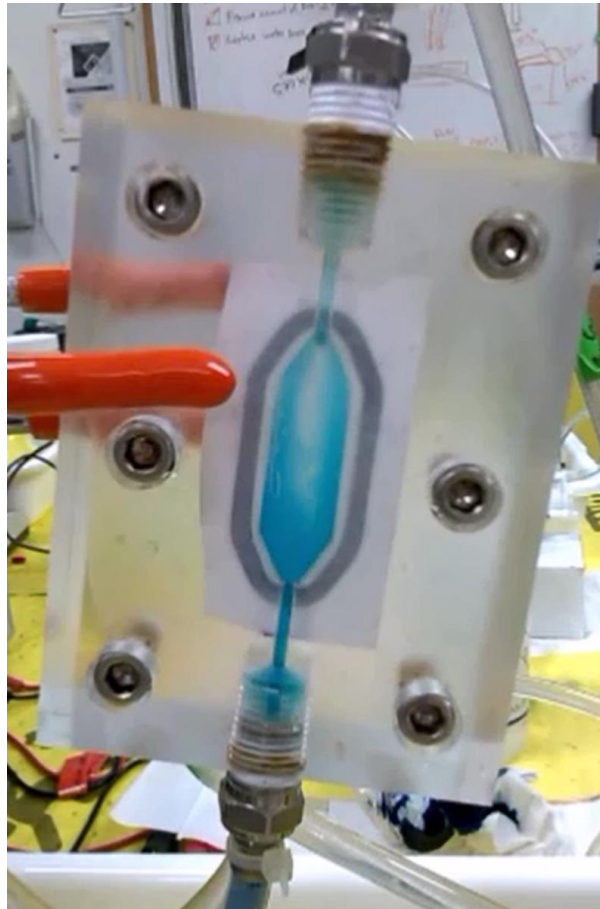**Figure 5. Results of CFD simulation of old membrane contactor.**

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Figure 6. Dye test of old membrane contactor.

during the refinement process is extremely high even at the lowest refinement level, requiring a great amount of physical RAM on the computer during the calculation. For this reason, the calculations took sever hours each and were usually run overnight.

A snapshot of the video of the dye test performed to verify the results of the CFD simulation is showed in Figure 6, demonstrating that the tri-dimensional simulation represents with good approximation the real behavior of the fluid within the memebrane contactor and that CFD software can be used as a tool to study how different design parameters affect the performances of the membrane contactor.

IV.   Results

Several computer aided design (CAD) models have been designed using SolidWorks and for each of them a CFD analysis has been performed to investigate the uniforminty of the flow for all the lengh of the interfacial area. The parameters investigated include pressure, velocity, total pressure, turbulence intensity, and vorticity.

The boundary conditions imposed at the inlet of the hose barb threaded to the membrane contactor are different flow rates, starting at 0.4 LPM, which is the value used during the previous membrane tests, and up to 9 LPM, which is the maximum value that can be reached when operating the DM412.VS pump at its maximum speed. The fluid used in all the simulations is water at 25 ^{O}C. A fully developed pipe flow is imposed at the inlet of the hose barb, which allows to estimate the turbulence level in function of the Reynolds number. Turbolence intensity, which is expressed as a percentage, is the ratio between the standard deviation of the velocity fluctuations at a particular location and the average of the velocity at the same location over a specified period of time. Vorticity of a three-dimensional flow is a pseudovector field defined as the rotational of the velocity field describing a continuum motion. It describes the local spinning motion of a particle along a trajectory.

The results of CFD simulations led to the selection of the design of a new membrane contactor, which has a bigger diameter of the inlet and outlet ports, inclined at a 30 degrees angle with respect to the surface of the membrane. The diameter of the hose barb fitting at the outlet port is bigger compared to the one at the inlet in order to minimize any built-up pressure within the membrane chamber. The membrane chamber is much longer than the previous version,

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almost four times. This allows the fluid to remain uniform for a longer tract of the interfacial area. The depth of the main chamber of the contactor has slightly increased to reduce the gradient of the velocity of the fluid between the center of the chamber and the surface of the membrane. The simulations show uniformity of the flow at all the different flow rates simulated between 0.4 LPM and 9LPM. Figure 7 and Figure 8 show the velocity, the pressure, the turbulence intensity, and velocity contours within the chamber of the down-selected membrane contactor. Figure 9 shows the snapshot of the video of a dye test performed on a prototype of the new contactor next to the results of the CFD simulation. Blue methylene was used again as a dye and it was injected in the feed solution when the system was running at regime. The dye test demonstrated again that the CFD simulation represents with good approximation the
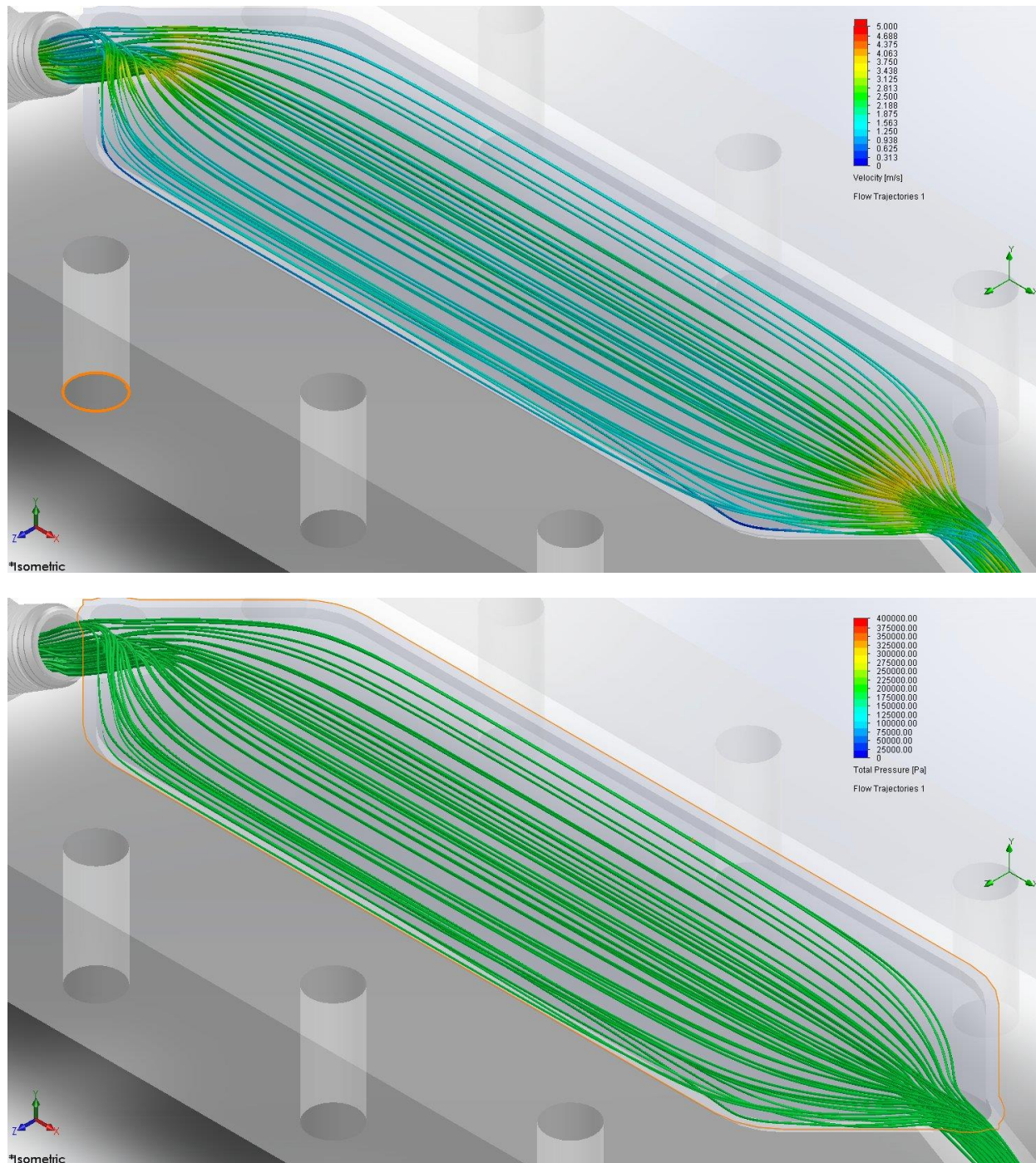

Figure 7. CFD simulation of new membrane contactor, velocity and total pressure.

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real behavior of the fluid within the memebrane contactor. Following the results of the CFD simulation and the dye tests, the final design has integrated larger fillets that reduce stagnant zones and areas affected by an excessive gradient of velocity, pressure, turbulence, and vorticity of the fluid, and include some optimizations needed to simplify the manufacturing process using both laser cutting and milling. The three dimensional model of the final design is showed in Figure 10.
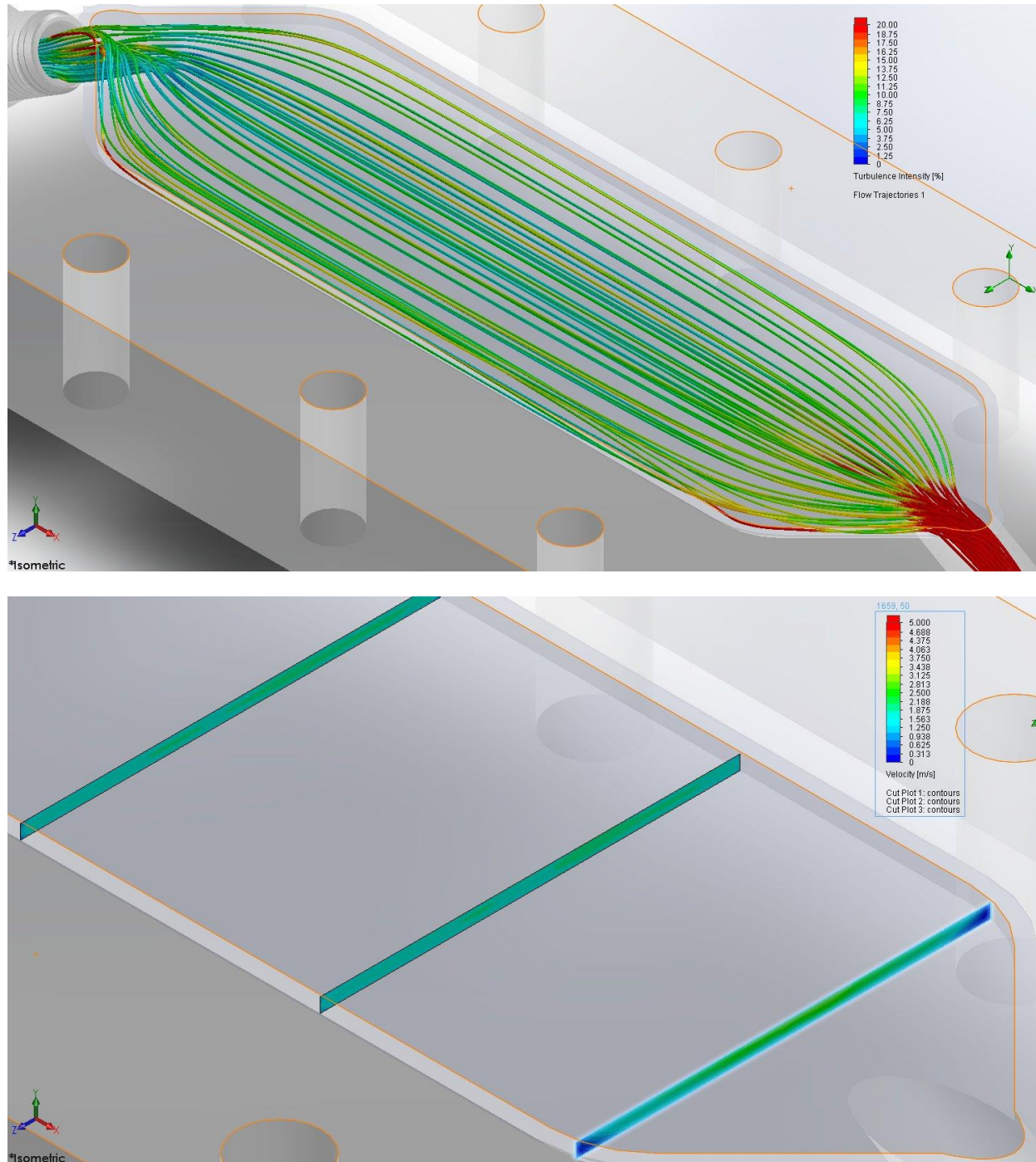

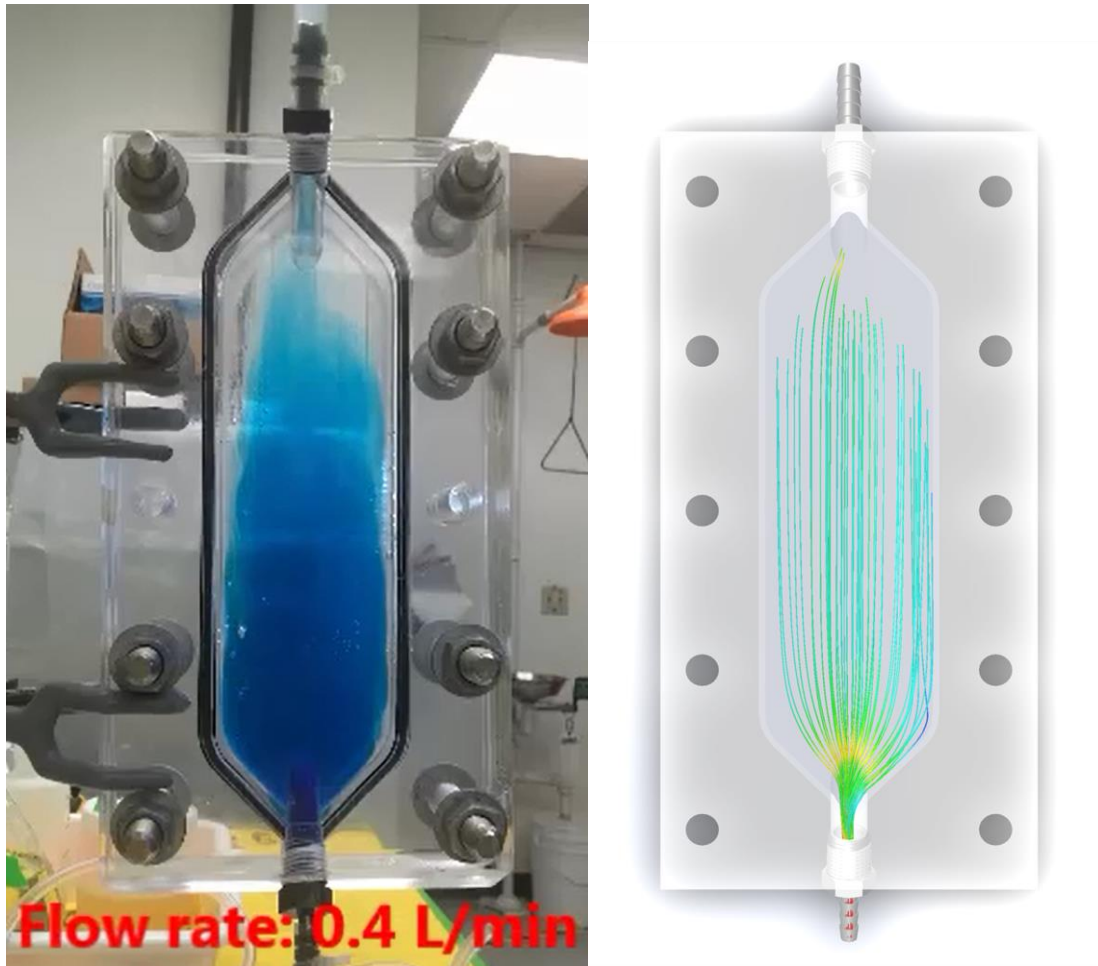**Figure 8. CFD simulation of new membrane contactor, turbulence intensity and velocity cut plots.**

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Figure 9. Comparison between dye test and CFD simulation of new membrane contactor.
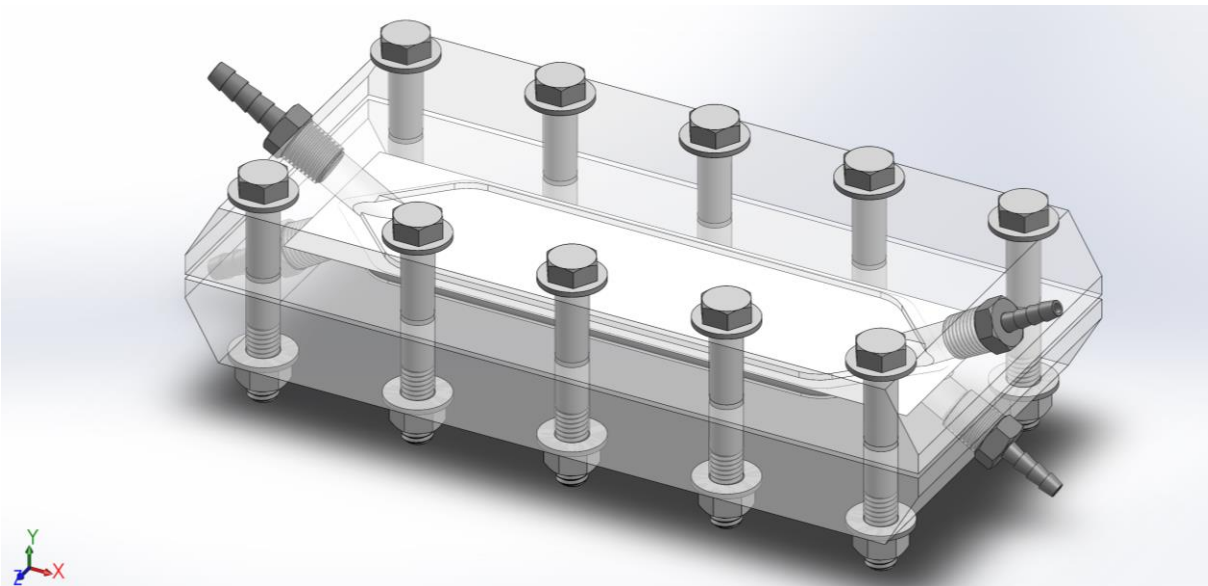

Figure 10. CAD of new membrane contactor.

V. Conclusion

Computational fluid dynamic simulations have been used to optimize the design of the membrane contactor used to test forward osmosis membranes with the objective of achieving a uniform performance of the module for all its lengh. Dye tests done on prototypes built to verify the outcomes of the simulations have proven with very good approximation the reliability of the software tool.

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Acknowledgments

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