Flux recovery of a forward osmosis membrane after a fouling process

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Wastewater treatment through Forward Osmosis (FO) membranes is a process that has been evaluated in the past years as an innovative technology for the Next Generation Life Support Systems. FO technologies are cost effective, and require very low energy consumption, but are subject to membrane fouling. Membrane fouling occurs when unwanted materials accumulate on the active side of the membrane during the wastewater treatment process, which leads to a decrease in membrane flow rates. Membrane fouling can be reversed with the use of antifoulant solutions. The aim of this study is to identify the materials that cause flow rate reduction due to membrane fouling, as well as to evaluate the flux recovery after membrane treatment using commercially available antifoulants. 3D Laser Scanning Microscope images were taken to observe the surface of the membrane. Fourier Transform Infrared (FTIR) spectrometry results identified possible compounds that cause membrane fouling and FO testing results demonstrated flow rate recovery after membrane treatment using antifoulants.

Nomenclature

*FO* = Forward Osmosis
*FTIR* = Fourier Transform Infrared
*LSS* = Life Support Systems
*OA* = Osmotic Agent
*RO* = Reverse Osmosis
I. Introduction

FO SYSTEMS are the base for next generation LSS, because it is the most reliable, low-cost technology for its utilization in future space missions.

Since it would be impractical, in terms of volume and cost, to completely stock a spacecraft with oxygen or water for long duration missions, it is indispensable to create a lightweight water recycling system that will provide astronauts with the water supply they need for as long as the mission lasts.

Conventionally, osmosis is defined as the net movement of water across a selectively permeable membrane driven by a difference in osmotic pressure across the membrane. The selectively permeable membrane allows water to pass through it, but it rejects the solutes and contaminants. This approach is used to recycle wastewater, humidity condensate and urine into drinking water to provide astronauts with a reliable water source.

The greatest advantages of FO water treatment systems, are the low consumption of energy and the reduction in fouling compared to RO systems. However, fouling is still a major issue in the long term performance of the system. The aim of this project is to find an efficient cleaning system using commercially available antifoulants that will restore the system’s flow rate after fouling occurs, and extend the membrane’s lifespan.

II. Background

A. Forward Osmosis (FO)

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 (Fig. 1). 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 or ions. [1]

As the feed water comes through the active side of the membrane, leaving contaminants behind, the membrane tends to catch the contaminants in its active surface, eventually reducing or blocking the water flux. When this occurs, the membrane must be cleaned in order to restore its optimal function. In this work, two different methods have been tested and measured to find the most practical cleaning method for fouled membranes.

![Forward Osmosis](image)

Fig. 1. Forward osmosis
B. Membrane Fouling

The organic fouling of a membrane is caused by a deposition of biopolymers. Previous studies have demonstrated that the major percentage of those polymers are proteins and polysaccharides [2] more specifically polysaccharides and other non-settetable organic matter with a molecular weight larger than 120 000 Da [3]. Biological precipitation can be another contribution to inorganic fouling. The biopolymers contain ionisable groups (COO-, CO32-, SO42-, PO43- and OH-) which are easily capturable by metal ions. Metal ions play a significant role in the formation of fouling layers, which can bridge the deposited cells and biopolymers and then form a dense cake layer. There exists a synergistic interaction among biofouling, organic fouling and inorganic fouling [4].

In the filtration process of wastewater, different fouling mechanisms may occur, expressed by the filtration resistance $R$ (Fig. 2). The retained components can form a cake layer (C) on top of the membrane surface, block the membrane pores (P) or adsorb (A) at the membrane surface or in the membrane pores, depending on their chemical and physical properties. [3]

Fig. 2. Schematic drawing of filtration resistances

Antiscalants acts as threshold inhibitors of growth of scales from supersaturated brine. In minute concentrations, they complex with the surfaces of seed crystals, preventing them to grow in the super-saturated brine. Some antiscalants also inhibit the precipitation of inorganic gels such as hydroxide/oxides of aluminum, iron, manganese and other heavy metals and silica and silicates. Certain antiscalants also inhibit the polymerization of reactive silica that result in membrane fouling by polymeric hydrated silica and silicates. [5]

C. Fouled membrane

The FO membrane (Fig. 3) was used for treating wastewater at NASA Johnson Space Center. The feed consisted of humidity condensate, hygiene water and urine pretreated in a bioreactor. The feed was circulated through the FO membrane until the system failed, which indicated membrane fouling (accumulation of unwanted materials on the surface of the membrane).

The membrane was then brought to NASA Ames Research Center to determine the fouling composition and to evaluate the flow rate recovery after cleaning it with commercially available antifoulants.
III. Materials and Methods

We want to compare the flow rate difference in the fouled membrane before and after the cleaning process, and in the control membrane. We also submitted the fouling composition to a FTIR analysis to know which fouling agents were present in the membrane.

First, the flow rate in the fouled membrane was measured. Then, it was cleaned with the King Lee 1000 antiscalant, and after that, another run with DI water as feed was performed to see if there was any improvement in the flow rate. Following that, it was cleaned with King Lee 2000, measured and then a final DI water run was performed.
A. Fouling composition

FTIR sample analysis protocol

Membrane fouling agents were subjected to FTIR analysis. Samples of the fouling agents were collected with a Corning® Small Cell Scraper from fouled Porifera FO membrane. The samples (Fig. 5) were placed in a weight boat, and were dried in the desiccator for 72 hours. Following the drying process, the samples were placed in an Eppendorf tube, and were delivered to Evans Analytical Group, which performed the FTIR analysis.

![Fig. 5. Dessicated fouling sample](image)

B. Membrane Performance Testing

The experiment set (Fig. 6) consisted in a set of two graduated cylinders (1 and 2), each connected to a pump (5) (for fluid recirculation purposes) and to the test cell (3) between them. The membrane (4) was installed between two acrylic plates; the active layer of the membrane facing the feed (1) and the membrane support facing the osmotic solution (2). Test cell has a membrane area of 4.25X10^-4 m².

![Fig 6. Testing cell diagram](image)

The left cylinder was filled with 70ml of DI water (Feed), and the right one was filled with 70ml of an Osmotic Agent (OA), a 3.5% NaCl Solution. Two tubes were set next to a ruler in order to measure and control the pressure of the fluids. The pressure in the Feed side was always higher than in the OA side.

After ten minutes of starting the pumps, and the system was stabilized, the cylinders were adjusted again with 70ml of fluid each, since some of the fluid was inside the tubes.
The system was controlled and measured every hour for a period of five hours to calculate the flux rate. For test reproducibility, the experiment was made in triplicate. First we tested the new Porifera membrane and then the fouled membrane. We compared the flux rate difference afterwards.

C. Cleaning methods
Two different antifoulants were used to clean the membrane: King Lee 1000 (hardness scale removal) and King Lee 2000 (organic removal).

The antifoulants were ran through the testing cell at 10\% concentration using the same procedure as the membrane performance testing (Fig. 7). After each cleaning process, the membranes were tested using DI water and NaCl solution as feed and OA respectively, in order to verify any improvement in flux rate.

Fig 7. Testing cell

IV. Results

A. FTIR Analysis
The components from the FO membrane were identified as a biological polyamide such as the protein in skin and/or a synthetic polyamide such as a polymeric resin; inorganic silicate such as silica, and relatively smaller amounts of an ester and possibly aliphatic hydrocarbons.

A representative sample of the dried residue was transferred to an infrared transmitting substrate and examined by the Fourier Transform Infrared Spectrometer (FTIR, Thermo Nicolet 6700) with the FTIR Continuum microscope in transmission mode.

Fig. 7 shows FTIR spectrum of two micro-pieces of the components from the osmosis membrane, in an overlay format, demonstrating its homogeneity (i.e., the match of the bands between the two measurements). The components were identified as:
- Biological polyamide such as the protein in skin and/or synthetic polyamide such as a polymeric resin (e.g., bands at ~ 3292, 2921, 2851, 1657, 1544, 1463 and 1381 cm⁻¹);
- Inorganic silicate such as silica (e.g., bands at ~ 1102 and 805 cm⁻¹);
- A small amount of ester (weak band at ~ 1734 cm⁻¹), and
- Possibly aliphatic hydrocarbon (intensity of the bands at ~ 2921 and 2851 cm⁻¹).

Fig. 8. FTIR spectrum of dried components from a FO membrane used in wastewater treatment
B. Flow rates

Fig. 9 shows the difference between the flow rates of the fouled and the control membrane. The control membrane obtained a flow rate of 96 ml after a five hour run, while the fouled membrane reached a flow of 88 ml after the same time.

![Fig. 9. Flow rate difference between fouled and control FO membrane](image)

Fig. 10 shows the differences between the flow rates of the fouled and control membranes, as well as the flux after each cleaning procedure. The flow rate after the first cleaning remained the same, and after the second cleaning it improved, reaching 92 ml after the run. However, it did not reach the flow rate of the control membrane.

![Fig. 10. Flow rate of the Control and fouled FO membranes, and the FO membranes after cleaning procedures.](image)
V. Conclusion

There was an improvement in the flow rate after both cleaning processes. However, the original performance was not completely restored. The flow rate with the control membrane increased in a 37%, while the flow rate after the KL1000 cleaning was improved by 24%, and after the KL2000 cleaning it increased by 31%.

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References