

Appendices

Appendix A: Membrane Porosity

The porosity of the membrane was measured by weighting the dry and wet mass of the membrane. First, the dry membrane was weighted once received from the manufacturer (W_d), then the membrane was soaked for 24 hours in ultrapure deionized water. The water at the membrane surface was removed with a cloth before weighing it (W_w). The porosity is calculated based on the volume of water held by the dry membrane. The membrane porosity was obtained as follows:

$$Porosity = \left(\frac{W_w - W_d}{\rho_w v_m} \right) \times 100\%$$

where W_d , W_w , ρ_w , and v_m are the dry and wet membrane weight, the ultrapure water density at room temperature, and the membrane's volume, respectively.

Appendix B: Membrane cleaning in place method

After each filtration experiment (of 2 hours each), the LabBrain filtration unit and ceramic membrane element were drained, and all oily wastewater was flushed out of the system. First, RO water was circulated to clean the membrane (inside the membrane module) for 15 minutes at 50 °C to remove the accumulated oil at the membrane surface. Second, permeability measurements, using DI water, were done for the cleaned membrane to measure the regeneration level of the ceramic. The measured resistance after the permeability test was equivalent to membrane resistance (R_m) and the internal pore blockage (R_i). Third, for complete regeneration, the ceramic element was cleaned with alkaline NaOH (15-20g/L, 85°C, 30 minutes) and Phosphoric acid (85%, 5ml/L, 50°C, 15 minutes). Then, RO water was circulated for 15 minutes. Finally, the RO water was flushed and drained to rinse the filtration system until neutrality. Then another

permeability DI water test was carried out to measure the resistance of the membrane. The regeneration was repeated until 99% was achieved (Table S1).

Table S1: ceramic membrane cleaning cycle required by the manufacturer

Cleaning time, min	Temperature, °C	Chemical cleaning agent	Concentration
15	50	Water, H ₂ O	
30	85	Sodium hydroxide, NaOH	15 to 20 g /L
15	50	Water, H ₂ O	
15	50	Phosphoric acid 85%, H ₃ PO ₄	5 ml /L
15	50	Water, H ₂ O	

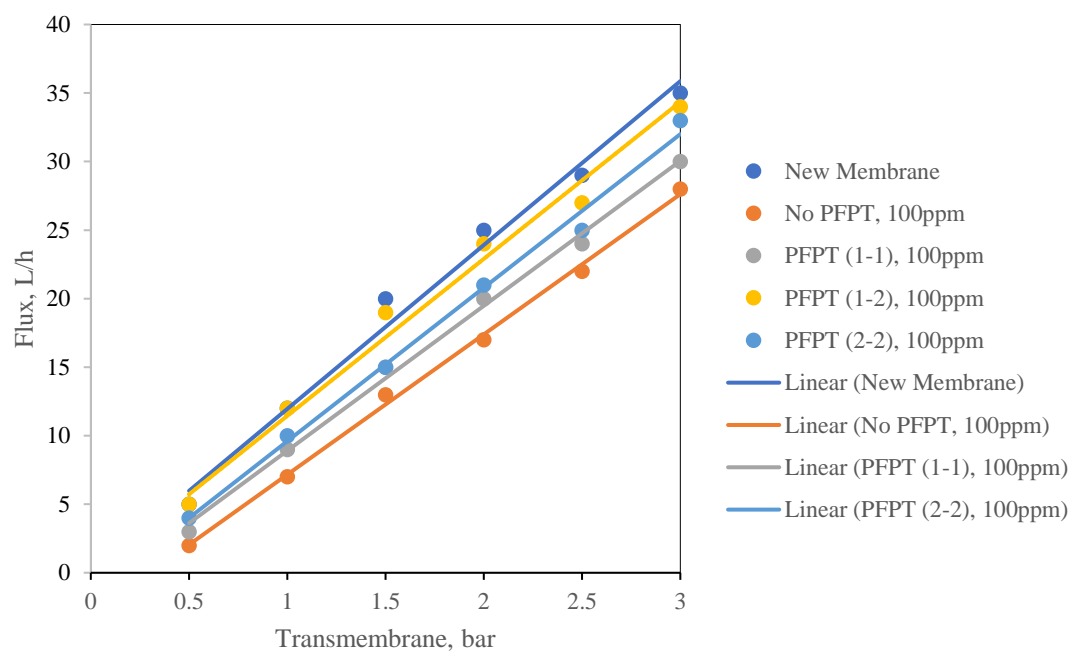


Figure S1: Permeability test at the end of each filtration process for feed 100ppm

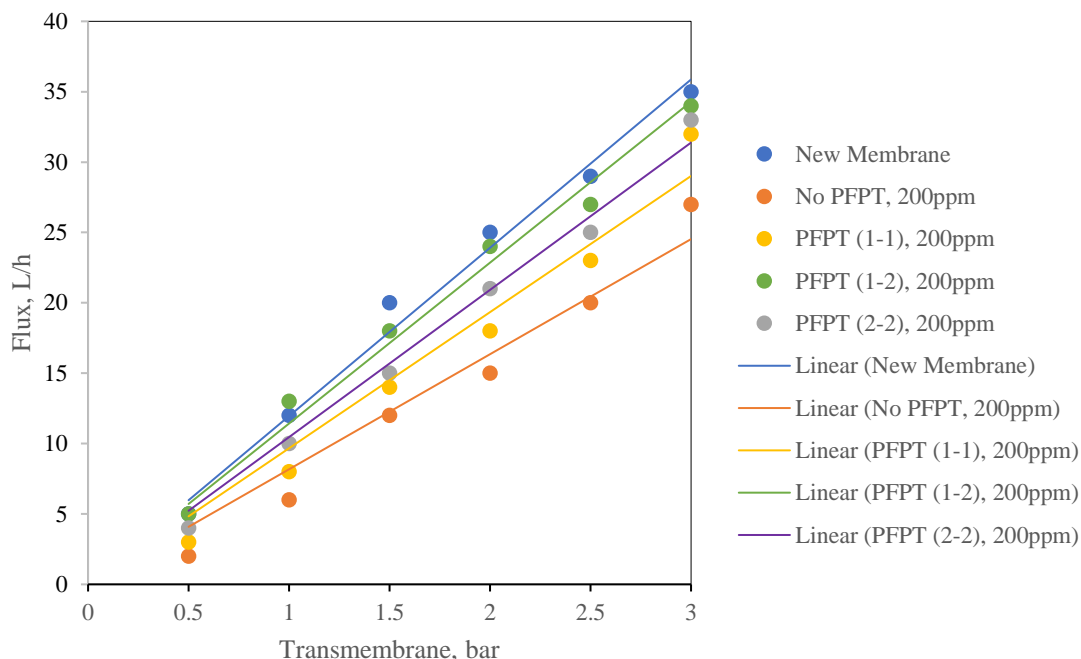


Figure S2: Permeability test at the end of each filtration process for feed 200ppm

Appendix C: Water Permeability

Before each experiment, the ceramic membrane is prepared by swamping it in deionized water (DI) to evacuate the air from it. Then, the water crossflow filtration was performed on the new membrane as a function of the applied transmembrane pressure. The permeability test was examined using Reverse osmosis (RO) water feed. For each TMP value (bar), the permeation flux was identified in liters per hour after 15 minutes of water filtration. Similarly, the applied TMP increases and the permeate flux is obtained. The increase of the applied TMP generates an increase in the permeation drag force toward the membrane surface. This explains the positive linear association between the permeate flux and transmembrane pressure variation (Figure S1). Figure S1 illustrates the ceramic membrane RO water flux as a function of applied transmembrane pressure.

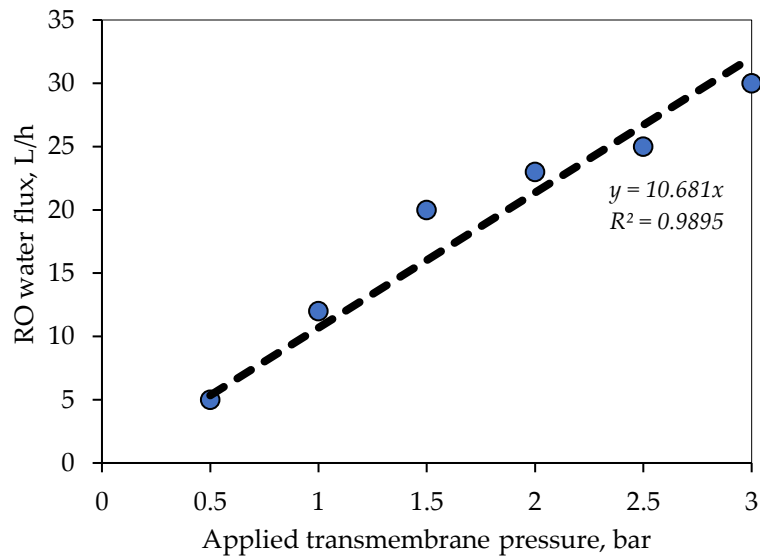


Figure S3: RO water permeates flux versus transmembrane pressure of the ceramic membrane at CFV 1 m/s

Supplementary materials

Figures

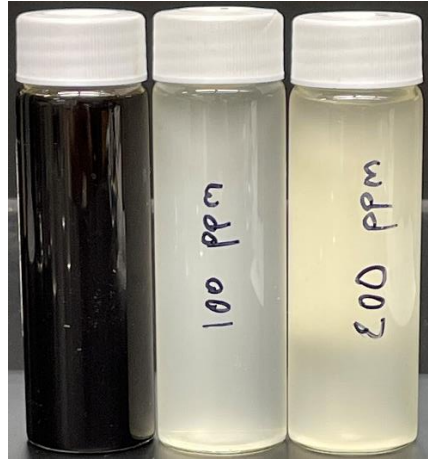


Figure A1: From left to right, Bakken oil, feed 100ppm, and feed 200ppm.

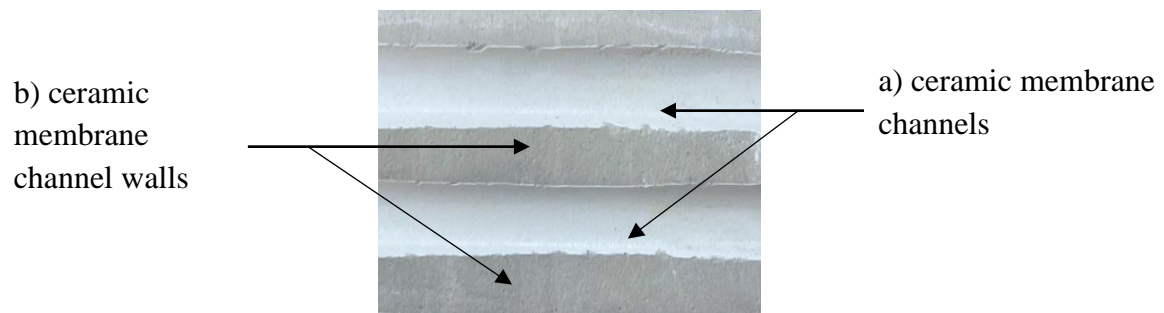


Figure A2: New ceramic membrane: a) Ceramic membrane channels, b) ceramic membrane channel walls.

Oil droplet

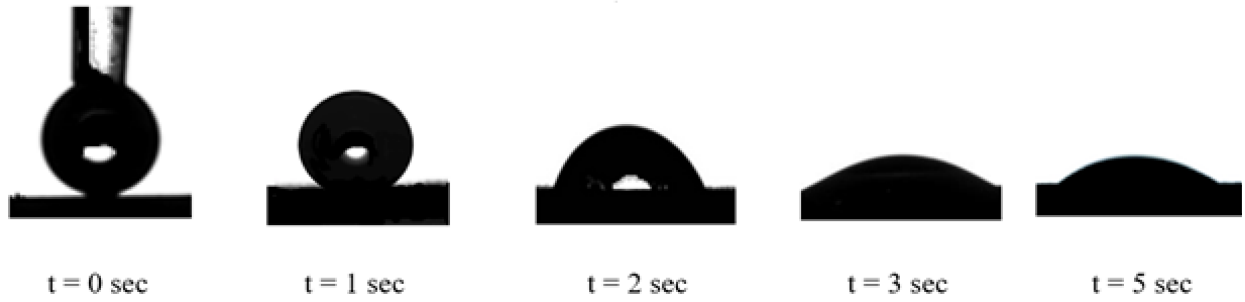


Figure A3: Sequential Images of the contact angle of a Bakken oil droplet at the ceramic membrane surface

Water droplet

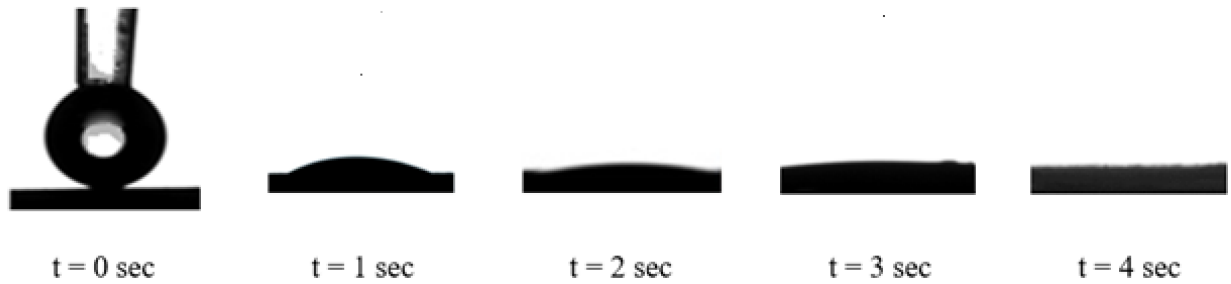


Figure A4: Successive Images of the contact angle of a water droplet at the ceramic membrane surface



Figure A5: LabBrain ceramic membrane filtration unit

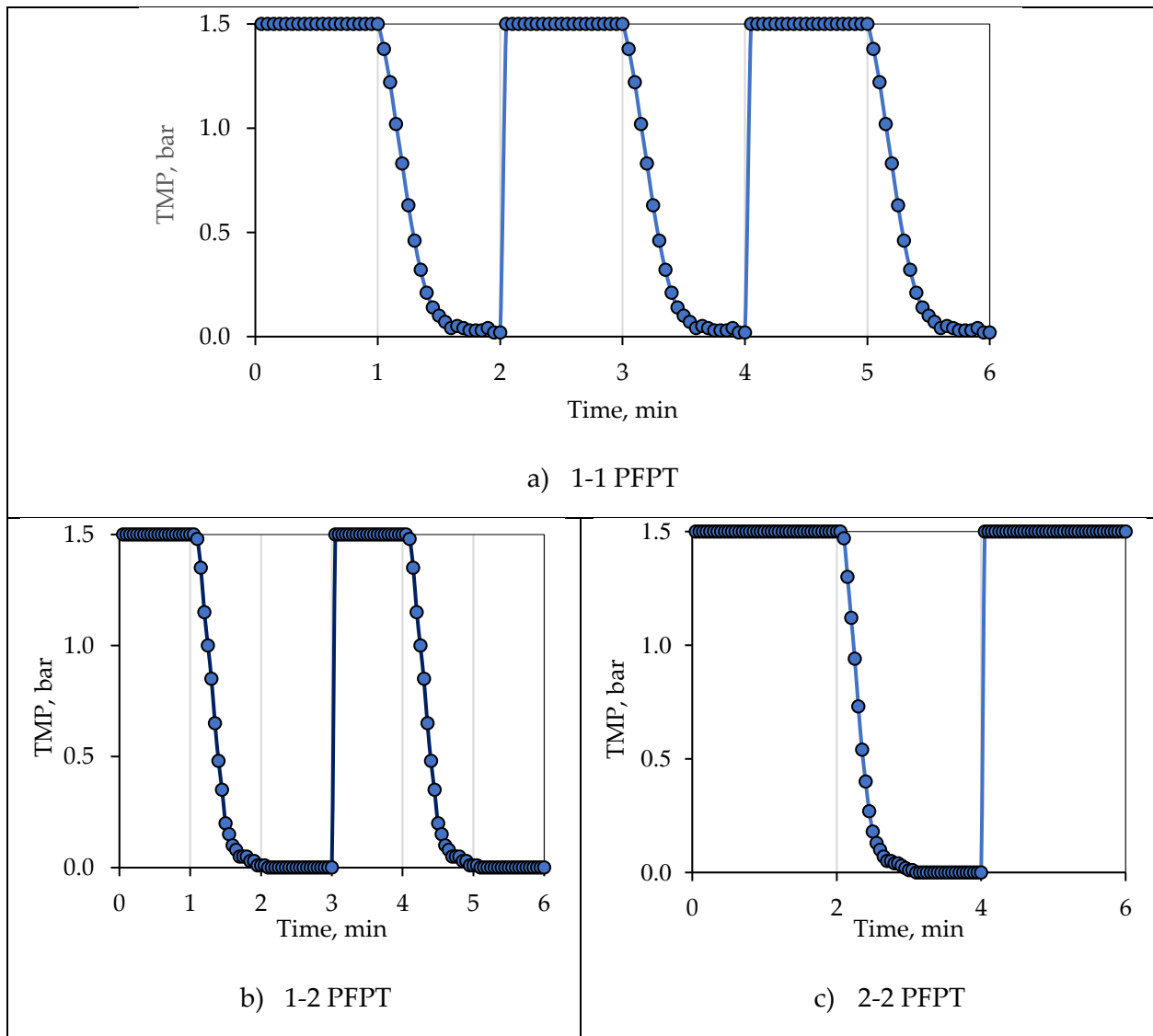


Figure 6A: Different pressure patterns of the PFPT. a) 1-1 PFPT, b) 1-2 PFPT, and c) 2-2 PFPT

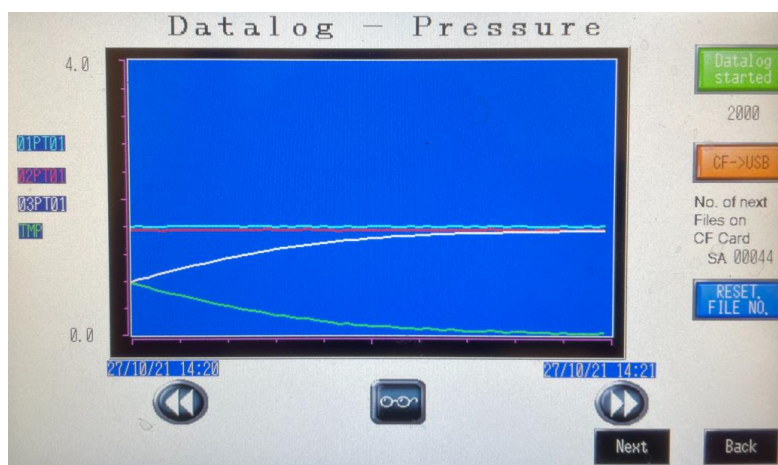


Figure A7: Control panel in the LabBrain filtration unit and Data log Pressure during the cleaning cycle (TMP (green line) tends to 0 bar)

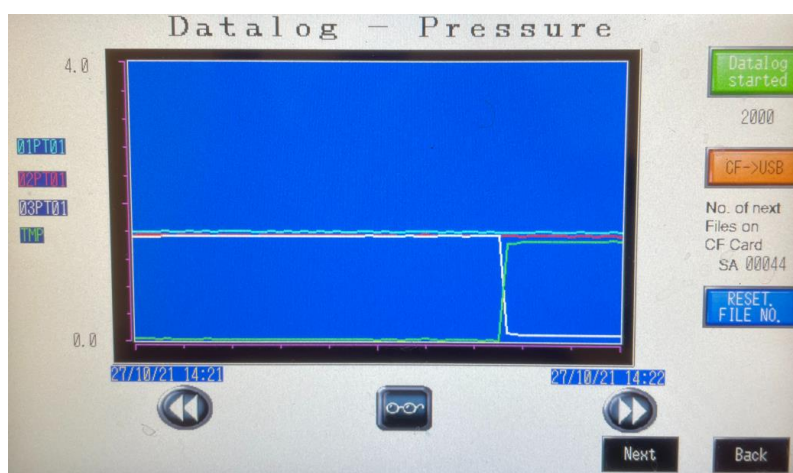


Figure A8: Control panel in the LabBrain filtration unit and Data log Pressure during the PFPT Filtration cycle (TMP (green line) =1.5 bar)

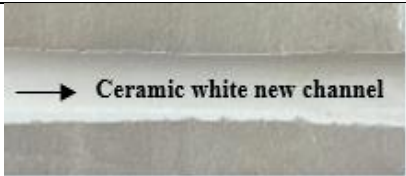
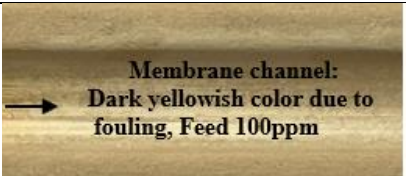
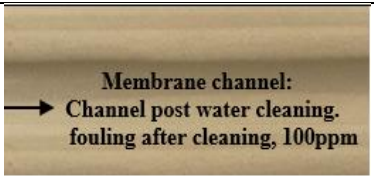
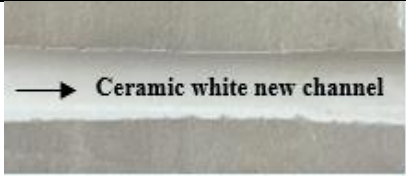
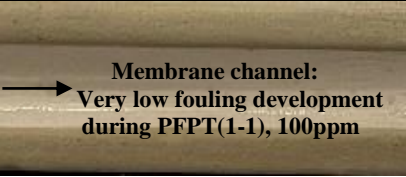
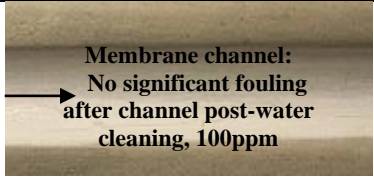
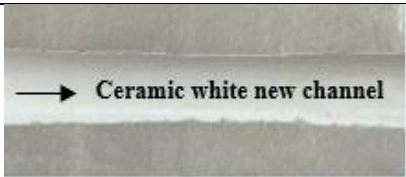
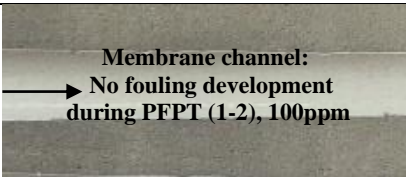
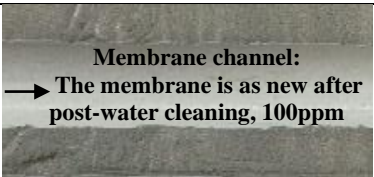
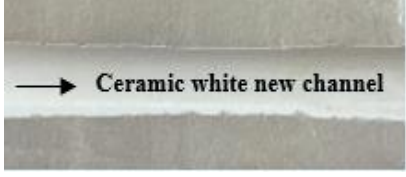
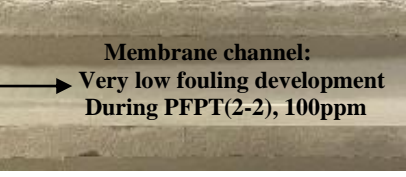
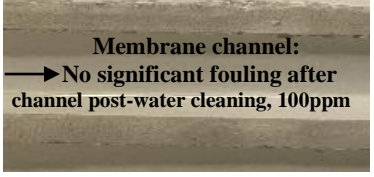
	Internal channel ceramic membrane		
Feed 100ppm	At, t = 0 min new ceramic membrane is used for the experiment	At, t = 120 min Fouled membrane At the end of the experiment	Water post-cleaned Ceramic membrane
No PFPT	 → Ceramic white new channel	 → Membrane channel: Dark yellowish color due to fouling, Feed 100ppm	 → Membrane channel: Channel post water cleaning. fouling after cleaning, 100ppm
PFPT (1-1)	 → Ceramic white new channel	 → Membrane channel: Very low fouling development during PFPT(1-1), 100ppm	 → Membrane channel: No significant fouling after channel post-water cleaning, 100ppm
PFPT (1-2)	 → Ceramic white new channel	 → Membrane channel: No fouling development during PFPT (1-2), 100ppm	 → Membrane channel: The membrane is as new after post-water cleaning, 100ppm
PFPT (2-2)	 → Ceramic white new channel	 → Membrane channel: Very low fouling development During PFPT(2-2), 100ppm	 → Membrane channel: No significant fouling after channel post-water cleaning, 100ppm

Figure A9: Ceramic membrane cross-sectional view before and after filtration and PFPT cycles, TMP=1.5 bar, CFV=1.0 m/s, and oil content of 100ppm

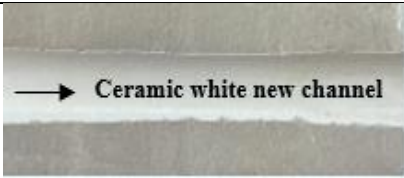

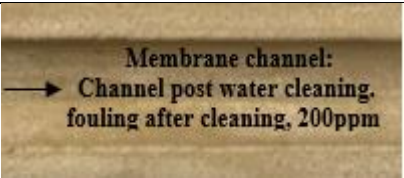
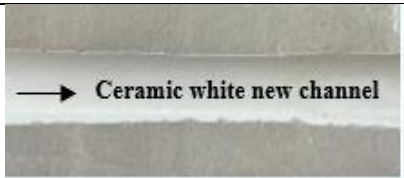
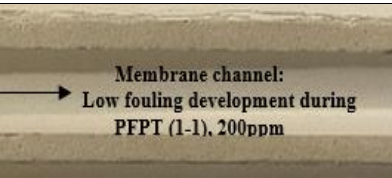
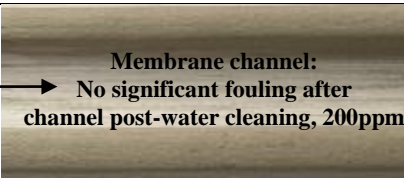

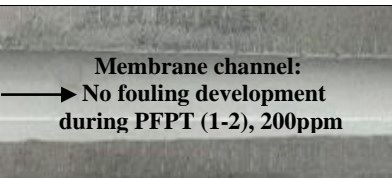
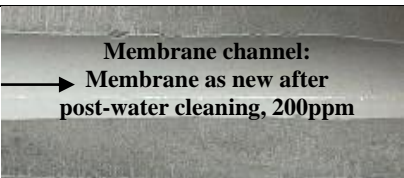
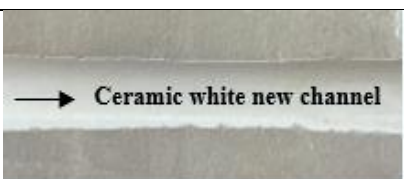
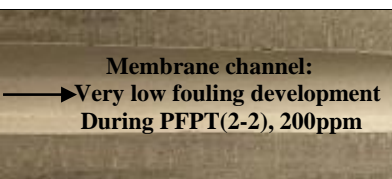
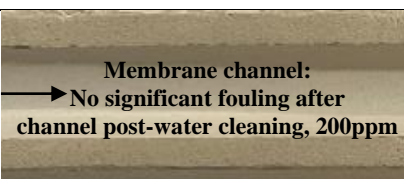
	Internal channel ceramic membrane		
Feed 200ppm	At, t = 0 min new ceramic membrane is used for the experiment	At, t = 120 min Fouled membrane At the end of the experiment	Water post-cleaned Ceramic membrane
No PFPT	 Ceramic white new channel	 Membrane channel: Dark yellowish color due to fouling, Feed 200ppm	 Membrane channel: Channel post water cleaning. fouling after cleaning, 200ppm
PFPT (1-1)	 Ceramic white new channel	 Membrane channel: Low fouling development during PFPT (1-1), 200ppm	 Membrane channel: No significant fouling after channel post-water cleaning, 200ppm
PFPT (1-2)	 Ceramic white new channel	 Membrane channel: No fouling development during PFPT (1-2), 200ppm	 Membrane channel: Membrane as new after post-water cleaning, 200ppm
PFPT (2-2)	 Ceramic white new channel	 Membrane channel: Very low fouling development During PFPT(2-2), 200ppm	 Membrane channel: No significant fouling after channel post-water cleaning, 200ppm

Figure A10: Ceramic membrane cross-sectional view before and after filtration and PFPT cycles, TMP=1.5 bar, CFV=1.0 m/s, and oil content of 200ppm

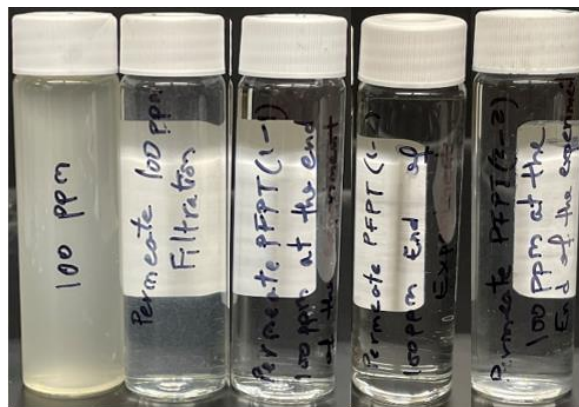


Figure A11: From left to right, feed 100ppm, no PFPT permeate, PFPT (1-1) permeate, PFPT (1-2) permeate, and PFPT (2-2) permeate



Figure A12: From left to right, feed 200ppm, no PFPT permeate, PFPT (1-1) permeate, PFPT (1-2) permeate, and PFPT (2-2) permeate

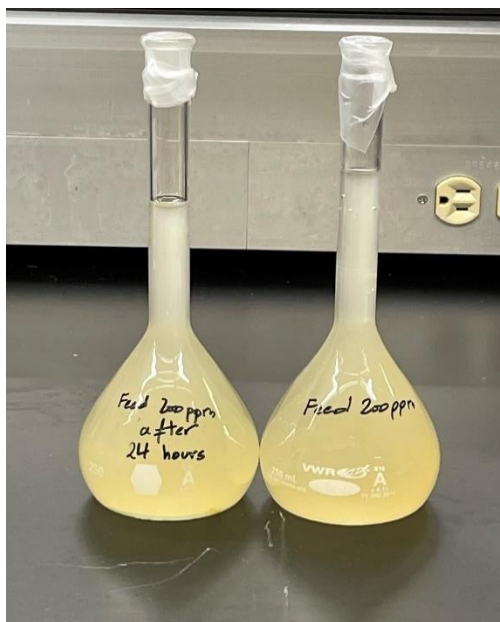


Figure A13: Oily wastewater emulsion (oil, water, and surfactant) at the synthesis time and after 24 hours: left volumetric flask shows no oil/water phase separation after 24 hours A16

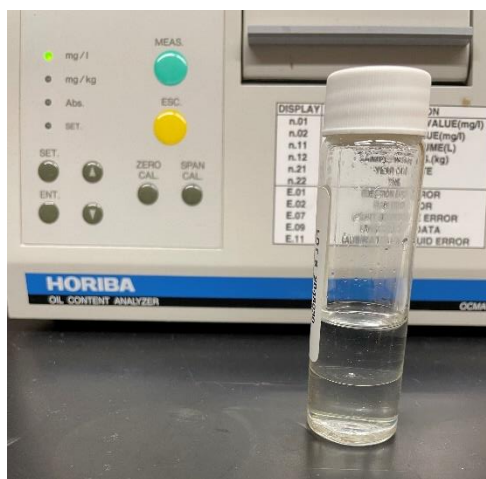


Figure A14: Oil phase extraction method, two-phase separation.
Top layer: water. Bottom layer: solvent-316 and oil A17


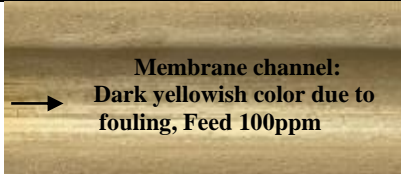
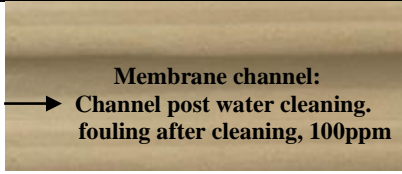
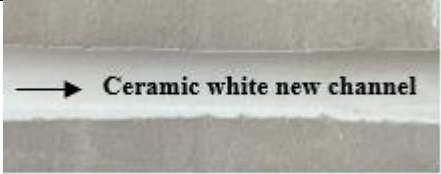
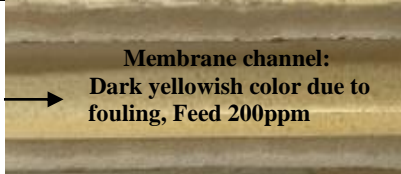
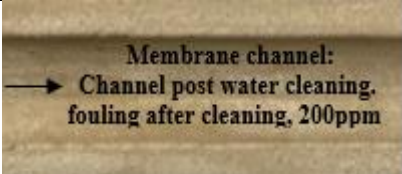
	Internal channel ceramic membrane		
Normal Filtration (no PFPT)	At t = 0 min new ceramic membrane is used for the experiment	At t = 120 min Fouled membrane At the end of the experiment	Water post-cleaned Ceramic membrane
Feed 100ppm	 Ceramic white new channel	 Membrane channel: Dark yellowish color due to fouling, Feed 100ppm	 Membrane channel: Channel post water cleaning. fouling after cleaning, 100ppm
Feed 200ppm	 Ceramic white new channel	 Membrane channel: Dark yellowish color due to fouling, Feed 200ppm	 Membrane channel: Channel post water cleaning. fouling after cleaning, 200ppm

Figure A15: Ceramic membrane internal channel surface oil deposition after normal filtration without PFPT cycles at TMP:1.5 bar, CFV: 1.0 m/s

Tables

	Feed Oil content, ppm	Permeate Oil content, ppm	Rejection, %
No PFPT (Normal filtration)	100	10.1	91
PFPT (1-1)	100	4.3	96
PFPT (1-2)	100	1.1	99
PFPT (2-2)	100	2.2	98

Table A1: The characteristics of the feed and permeate of the filtration and PFPT cycles at TMP=1.5 bar and CFV=1.0 m/s

	Feed Oil content, ppm	Permeate Oil content, ppm	Rejection, %
No PFPT (Normal filtration)	200	16.3	92
PFPT (1-1)	200	5.4	97
PFPT (1-2)	200	1.4	99
PFPT (2-2)	200	3.5	98

Table A2: The characteristics of the feed and permeate of the filtration and PFPT cycles at TMP=1.5 bar and CFV=1.0 m/s

Feed 100ppm	Feed 100ppm Turbidity, NTU	Permeate Turbidity, NTU
No PFPT (Normal filtration)	430.9	1.65
PFPT (1-1)	430.9	0.53
PFPT (1-2)	430.9	0.25
PFPT (2-2)	430.9	0.36

Table A3: The Turbidity of the feed 100ppm and permeate of the filtration and PFPT cycles at TMP=1.5 bar and CFV=1.0 m/s

Feed 200ppm	Feed 200ppm Turbidity, NTU	Permeate Turbidity, NTU
No PFPT (Normal filtration)	562	2.49
PFPT (1-1)	562	1.55
PFPT (1-2)	562	0.44
PFPT (2-2)	562	0.63

Table A4: The Turbidity of the feed and permeate of the filtration and PFPT cycles at TMP=1.5 bar and CFV=1.0 m/s