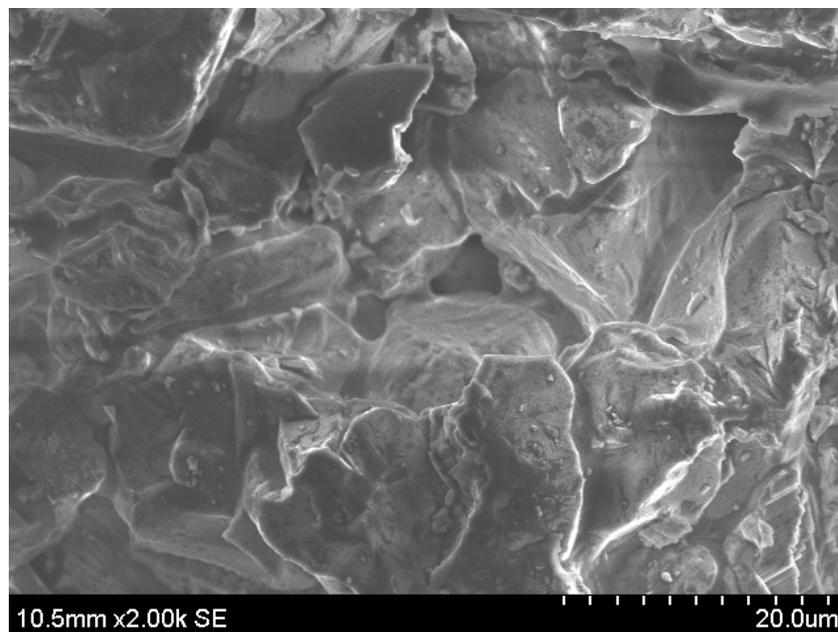




**NM WRRI Student Water Research Grant
Final Report**

**Student, Lin Chen, New Mexico State University
Faculty Advisor, Dr. Pei Xu, Department of Civil Engineering, NMSU**



SEM Image of Modified Ceramic Disc Membrane

June 3, 2019



1. Student Researcher: Lin Chen (New Mexico State University)
Faculty Advisor: Dr. Pei Xu (New Mexico State University)

2. Project title: Water Reuse and Desalination with Self-cleaning Photocatalytic Membrane Distillation

3. Description of research problem and research objectives.
 - (i) Research Problem: Conventional membranes are susceptible to membrane biofouling and organic fouling during treatment of impaired waters. Membrane fouling will cause increased treatment costs, decreased water production, and reduced membrane lifetime.
 - (ii) Research Objectives: This research aims to develop multi-functional hydrophobic microporous membrane with anti-fouling and self-cleaning properties for application of photocatalytic membrane distillation (PMD). Coating a superhydrophobic layer with titania photocatalytic nanoparticles on membrane surface could render membranes more robust, stable and chemical-resistant after modification.
 - (iii) Methods for Membrane Modification: Ceramic membranes are hydrophilic in nature. However, they can be hydrophobic when modified by adding hydrophobic groups via grafting with perfluoroalkylsilanes (FAS). Hence, they can be applied for membrane distillation (MD) after hydrophobic coating. The advantages of ceramic membrane for MD includes chemical-resistance, more stable with higher temperatures, and mechanical stability compared with conventional polymeric membranes [1-3]. Larbot et al. [4] initiated ceramic membrane modification for MD application in 2004. One type of FAS called 1H, 1H, 2H, 2H-perfluorodecyltriethoxysilane (normally noted C8) with a concentration of 0.01 mol/L was used as grafting agent. They observed a contact angle (CA) of 143° for alumina membrane with a pore size of 200 nm as well as a salt rejection (SR) of approximate 100% in their experiments. In addition, they suggested that a total soaking time of 118 hours was enough for obtaining a desired hydrophobic ceramic membrane. Water vapor flux was independent on the salt concentration when it is less than 0.1 M. Although this emerging method for grafting is feasible, the process for grafting and solvent used and concentration of the grafting agent varies. Additionally, the corresponding grafting time differs as well. For example, Lu et al. [5] grafted the γ -Al₂O₃ ceramic membranes on a α - Al₂O₃ support using 0.01 mol/L of tridecafluorooctyltriethoxysilane (noted as C6) and reported that an optimum grafting time of 48 hours with the highest CA of more than 150° when the coated times were five. Moreover,

hexane was used as the solvent for preparation of FAS solution whereas chloroform [4, 6] and ethanol [7] were also reported as the solvents.

4. Description of methodology employed.

The research was initiated by building customized membrane distillation (MD) and PMD reactors using polycarbonate plates and quartz glasses. Ceramic membranes modified using perfluoroalkylsilanes (FAS) and commercial hydrophobic polyvinylidene (PVDF) membranes from Pall Corporation were used to investigate the MD. Besides, PVDF membranes were modified using titania nanoparticles by spraying and coated by polydopamine (PDA) via brush methods. PDA can anchor TiO_2 nanoparticles on the surface of the PVDF membranes.

The membrane performance was evaluated in terms of product water flux and salt rejection at different temperature gradients. The temperature of hot/warm water side ranged from $38.4\text{ }^\circ\text{C}$ to $82.4\text{ }^\circ\text{C}$ while the temperature of the cold/permeate side was maintained at $21\text{ }^\circ\text{C}$. Synthetic solution of 1000 mg/L NaCl was used in the experiments simulating brackish water. A multi-flow channel peristaltic pump was used to pump feed and permeate into the MD system. The flow rate of feed and permeate remained the same as 100 ml/min . The water temperature of the hot and cold sides was controlled separately by a heater and a chiller manufactured by Poly-Stat.

Throughout the experiments, salt rejection was measured by the reduction of conductivity determined by a conductivity meter (HACH sension 5). Temperature at both sides was determined using a thermometer. Water flux was measured by the production of water as distillate. Membrane hydrophobicity was determined by contact angle measured by a goniometer (NRL C.A. Goniometer 100-00-115, ramé-hart instrument co.).

Two PMD systems were designed and constructed in the study. PMD system1 was used to test the performance of desalination using two types of ceramic membranes. One was Al_2O_3 ceramic membrane with an average pore size of 200 nm and diameter of 50 mm purchased from Hefei Shijie Membrane Engineering Inc. (China). The other one is the TAMI $\text{TiO}_2\text{-ZrO}_2$ disc membrane with an average pore size of 450 nm and diameter of 47 mm which is purchased from Sterlitech Inc. (US). The schematic diagrams of PMD system1 and system2 are the same as shown in Figure 1. Figure 2 shows the picture of the PMD system1 in the lab. Flow rates at hot feed side and cold permeate side in this report are the same as 800 ml/min while the corresponding flow velocity in PMD system1 is equal to 0.015 m/s or 90 cm/min .

PMD system2 was shown in Figures 1 and 3. It was used to test the performance of desalination using hydrophobic PVDF membranes and another type of ceramic membranes with an average pore size 100 nm and diameter of 25 mm which were acquired from European Institute of Membranes (EIM). This type of ceramic discs which are smaller in diameter were made using $200\text{ nm Al}_2\text{O}_3 + 50\text{ nm ALD TiO}_2$ with and without 5 nm Pd nanoparticles (marked as 50T and 50T5P in this report respectively).

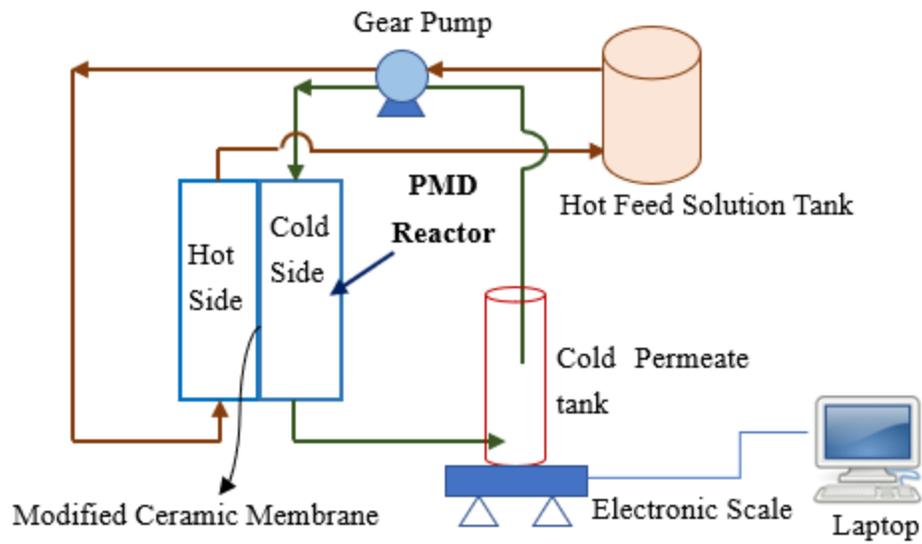


Figure 1. Schematic diagram of the PMD system

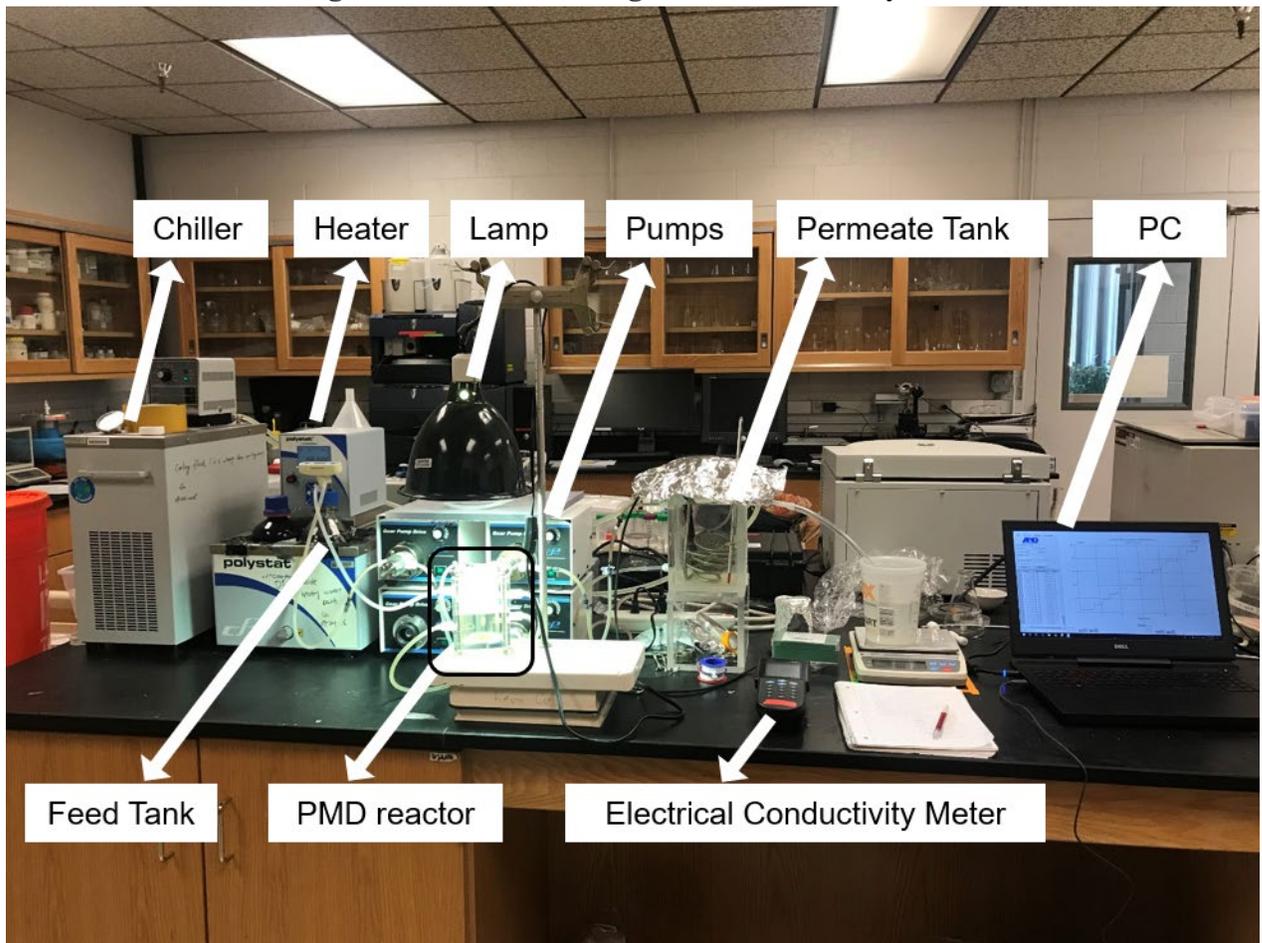


Figure 2. Picture of PMD system1

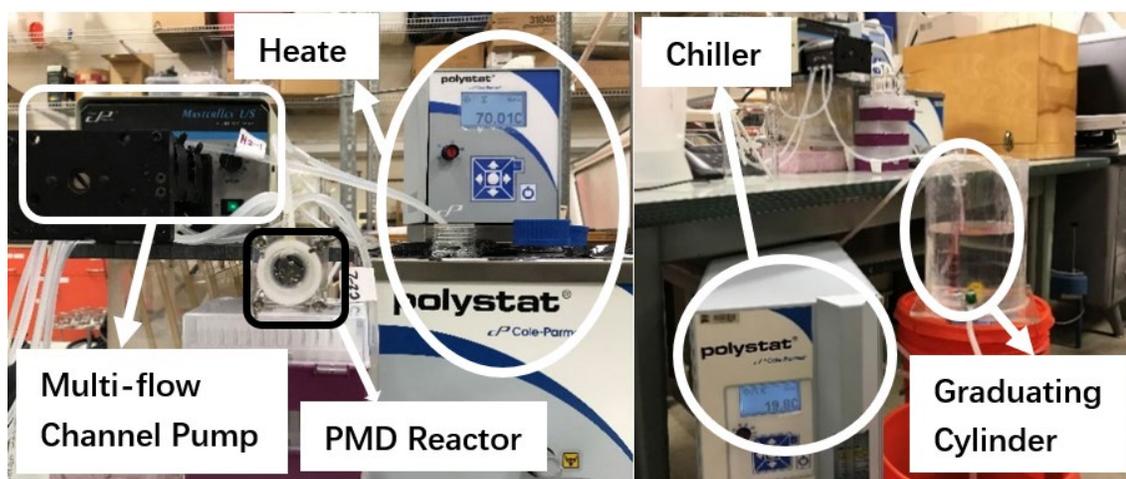


Figure 3. Picture of PMD system2

Table 1 shows the membranes used in this study. G1 and G2 were applied in PMD system1 while PVDF membrane and 50T and 50T5P ceramic membranes were applied in PMD system2. The hydrophobic modification of 50T5P was conducted using sol-gel with silica nanoparticles via spraying method. The preparation of silica sol-gel was reported by Wang et al [8]. Modification of G1 and G2 was finished by dip-coating using FAS solution.

Table 1. Membrane information

| Membranes | Types | Materials | Pore size | Thickness | CAs (°) before coating | CAs (°) after coating | Coating |
|-------------------|-----------|--|-----------|-----------|------------------------|-----------------------|---|
| FluoroTrans® PVDF | polymeric | PVDF | 0.2 μm | 127 μm | 122 | 122 | No coating |
| 50T | ceramic | 200nm Al ₂ O ₃ +50 nm ALD TiO ₂ | 0.1 μm | 1.5mm | 108(*) | 38(**) | No coating |
| 50T5P | ceramic | 200nm Al ₂ O ₃ +50 nm ALD TiO ₂ + 5 nm Pd NPs | 0.1 μm | 1.5mm | NA(***) | 134 | Using Silica sol-gel by spraying method |
| G1 | ceramic | Al ₂ O ₃ | 0.2 μm | 5 mm | NA(***) | 135 | FAS grafting for 56 hrs |
| G2 | ceramic | Al ₂ O ₃ | 0.2 μm | 5 mm | NA(***) | 138 | FAS grafting for 80 hrs |

Notes: ALD-atomic layer deposition; (*) 108 is the CA before tests, and (**) 38 is the CA value after experiments;

(***)NA means the membrane surface is superhydrophilic, CA is not measurable.

Methods for hydrophobic modification:

G1 and G2:

(i) Pretreatment of ceramic membrane: Commercial ceramic membrane is hydrophilic and needs to be pretreated prior to hydrophobic coating. The pretreatment process includes: A pristine ceramic membrane was firstly eroded by a 3 M NaOH solution for 5 min and then was cleaned via ultrasonication in acetone, ethanol and DI water for 10 min respectively. After that, dry the ceramic membrane at 110 °C in an oven for 12 hrs. Finally, cool the membrane to room temperature for subsequent use.

(ii) Preparation of FAS solution: The concentration of the FAS solution was 10 mM and chloroform was used as solvent. The FAS solution was prepared at an atmosphere of Argon gas because even a trace amount of humidity is harmful for the preparation. The solution should be vigorously mixed for 12 hours or dispersed for 4 hours using ultrasonication.

(iii) Grafting process: The pretreated and dried ceramic membranes were thoroughly soaked into the FAS solution for 4 hours; the grafted membranes were dried for 12 hours at 110 °C in an oven. The grafting process was then repeated three more times by immersing the membranes for 8 hours, 12 hours and 24 hours. The total grafting time can be modified depending on the desalination testing results.

50T5P membrane:

The sol solution containing silica nanoparticles was prepared by co-hydrolysis and condensation of two silane precursors, tetraethyl orthosilicate (TEOS) and tridecafluorooctyl triethoxysilane (FAS), in $\text{NH}_3 \cdot \text{H}_2\text{O}$ – ethanol solution. The detailed procedure information of the preparation of silica sol solution can be found from [9]. The coated 50T5P ceramic membrane was coated by spraying method.

Calculation methods:

Salt rejection was calculated using equation ①. Permeate flux ($\text{kg/m}^2/\text{h}$, KMH) was calculated using equation ②.

$$\text{Salt rejection, } r = 1 - \frac{C_p}{C_f} \quad \text{①}$$

where C_p and C_f are the salt concentration (or electrical conductivity) of the permeate and feed, respectively.

$$\text{Permeate flux, } J = \frac{\Sigma m}{At} \quad \text{②}$$

where Σm is the total weight of the product water (kg); A is the effective surface area of the membranes (m^2); t is the operating time (hour).

5. Description of results; include findings, conclusions, and recommendations for further research.

(1) Findings

(i) Flux and SR using commercial PVDF

Figure 4 shows an exponential growth of permeate flux with increasing feed temperatures using PVDF membranes when all other operating parameters remained stable and constant. Higher permeate flux can be obtained at higher temperature at feed stream indicating that higher temperature difference gives rise to greater production of distillate.

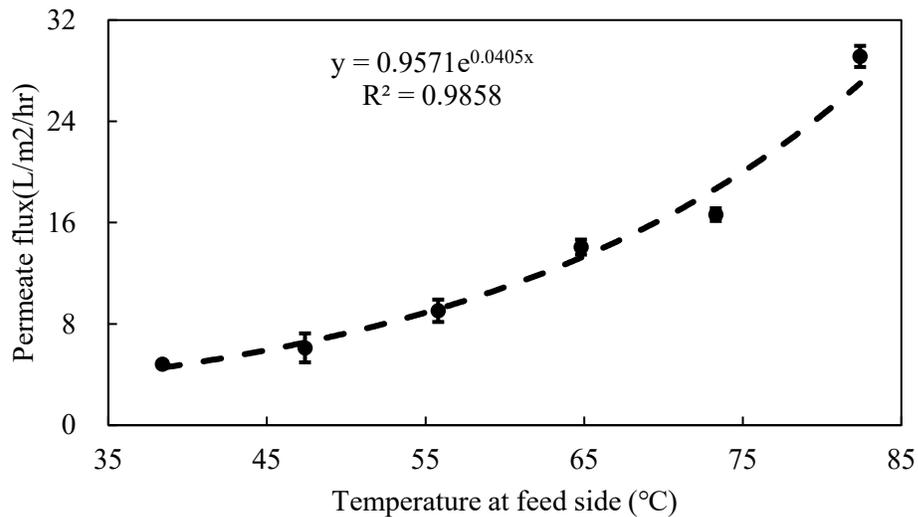


Figure 4. Permeate flux of hydrophobic PVDF membranes at different feed water temperatures (flow rate was 100 ml/min at both sides, temperature at cold side was constant at 21 °C, and feed salt concentration was ~1000 ppm). Each experiment was conducted for 3 hours and the standard deviation represents triplicate experiments.

This study also demonstrates that temperature difference does not affect SR. PVDF membrane reached. SR greater than 99.7% at various feed temperatures ranging from 38.4°C to 82.4°C at a flow rate of 100 ml/min and with the same cold permeate side temperature of ~21 °C (Figure 5).

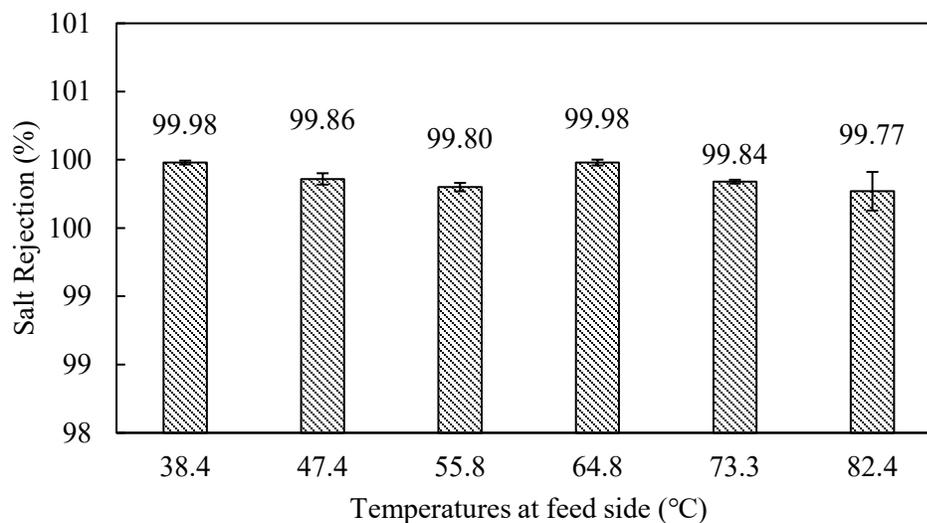


Figure 5. Salt rejection of PVDF membrane at different feed temperatures (flow rate was 100 ml/min at both sides, temperature at cold side was constant at 21 °C, and feed salt concentration was ~1000ppm). Standard deviation represents triplicate experiments.

Figure 6 and 7 show the trends of real-time permeate flux and average permeate flux (APF) and SR of PVDF membranes with and without TiO₂ modification during desalination of real wastewater collected from Las Cruces Wastewater Treatment Plant (LCWWTP) under the same operating conditions. Coating TiO₂ nanoparticles to PVDF membranes increased the APF by 33.9%, from 5.9 KMH of uncoated membrane to 7.9 KMH of modified membrane. Besides, the permeate flux with both PVDF membranes remained stable within 48 hours operation. The average SR and APF of PVDF membranes with and without TiO₂ modification were 97.9% and 85%, and 7.9 KMH and 5.9 KMH, respectively. SR of modified PVDF is 15.2% higher than that of unmodified PVDF. In addition, the minimum SR of modified PVDF is 96.9% which is 21.7% higher than that of 79.6% using unmodified PVDF. Therefore, desalination using PVDF membrane modified by TiO₂ nanoparticles shows a stable and higher performance of permeate flux and SR under the same long-term (~48 hours) operating conditions.

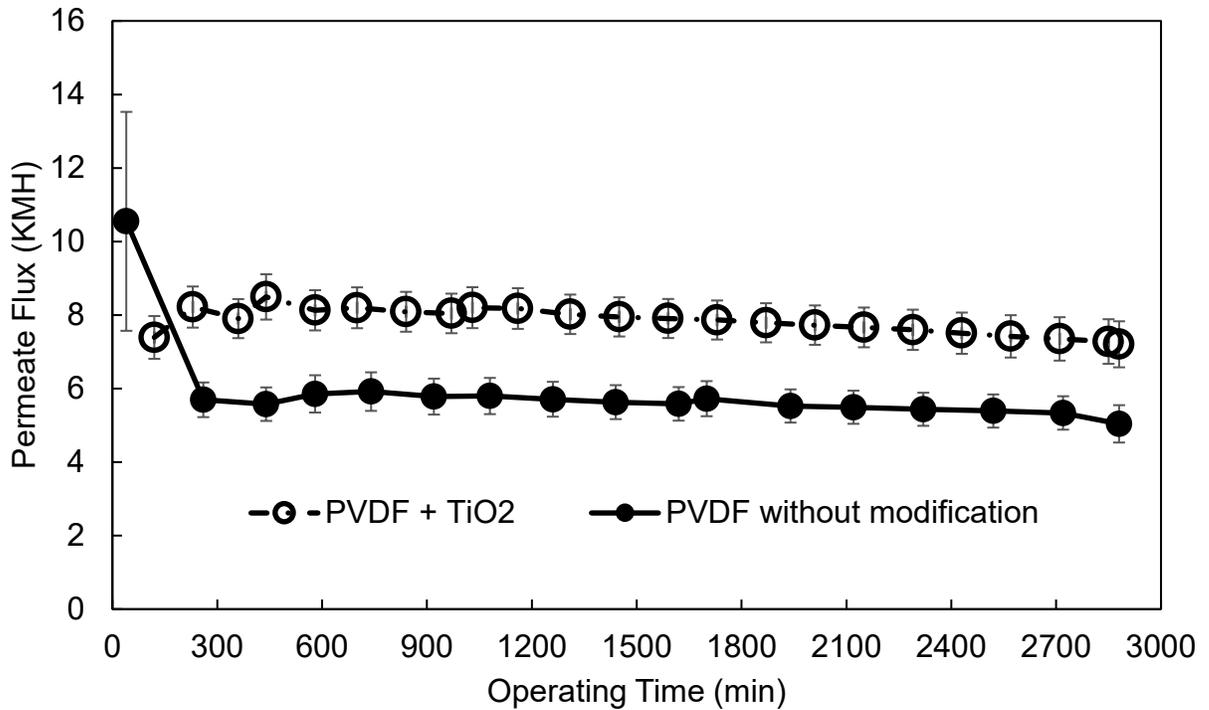


Figure 6. Permeate flux using PVDF membranes with and without TiO₂ modification for desalination of real wastewater from Las Cruces Wastewater Treatment Plant (LCWWTP) under the same operating conditions (flow rate was 400 ml/min at both sides, and temperature at feed side and cold side were at ~50 °C and ~20 °C, and feed salt concentration was ~768 ppm)

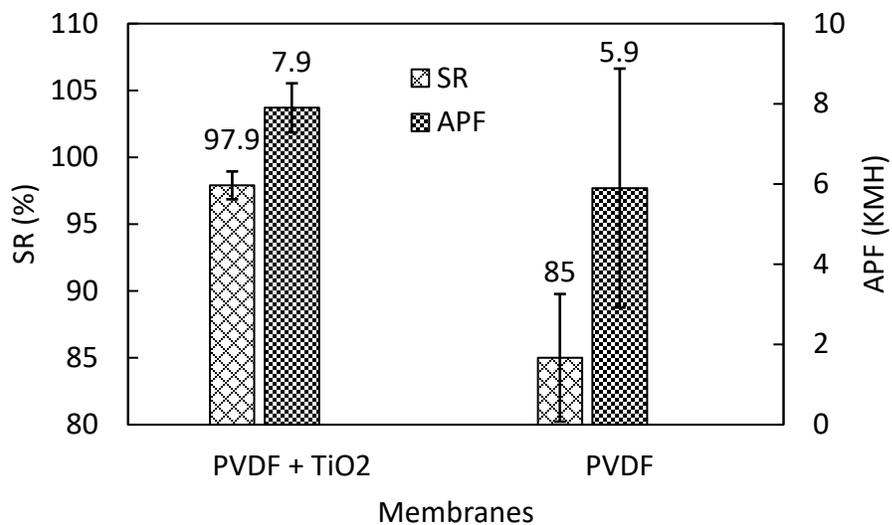


Figure 7. Average permeate flux and SR using PVDF membranes with and without TiO₂ modification with the same experimental conditions as in Figure 6.

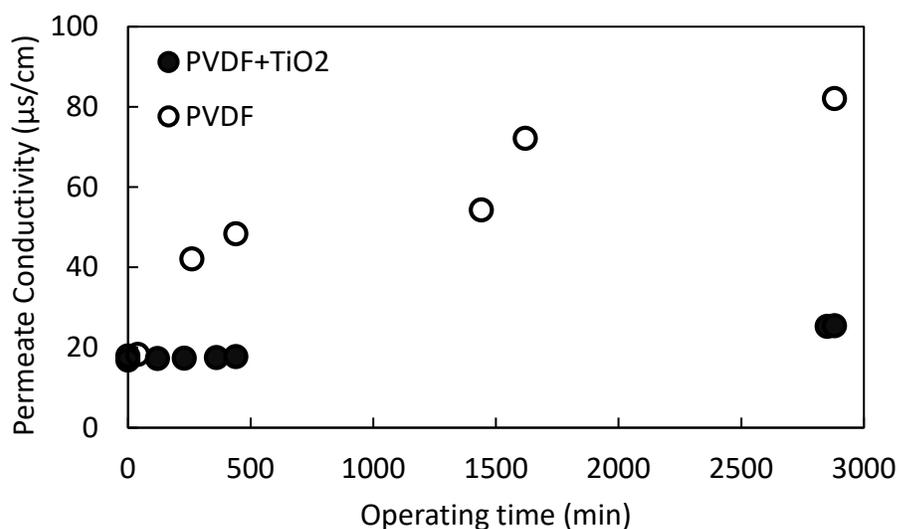


Figure 8. Permeate electrical conductivity using PVDF membranes with and without TiO₂ modification with the same experimental conditions as in Figure 6.

Figure 8 shows the permeate electrical conductivity (EC) using modified and unmodified PVDF membranes for wastewater collected from LCWWTP. EC in permeate using TiO₂ modified PVDF membrane was around 20 µS/cm which was lower than that using unmodified PVDF membrane within 48 hours operation indicating modified PVDF membrane had a stable salt rejection ability along with anti-wetting property. Therefore, coating with TiO₂ nanoparticles can enhance the anti-wetting ability of membrane for application of MD.

(ii) Flux and SR using 50T5P and 50T membranes

Figure 9 shows the permeate flux comparison of PMD with 50T and 50T5P under the same operating conditions. Flux of PMD with 50T5P were 18.7 and 31.4 KMH while that of PMD with 50T were only 1.6 and 2.4 KMH for the temperature sets of T_h-T_c of 50-20°C and 70-20°C. It can be concluded that membrane 50T5P performed much better than with 50T when all other operating conditions were kept the same.

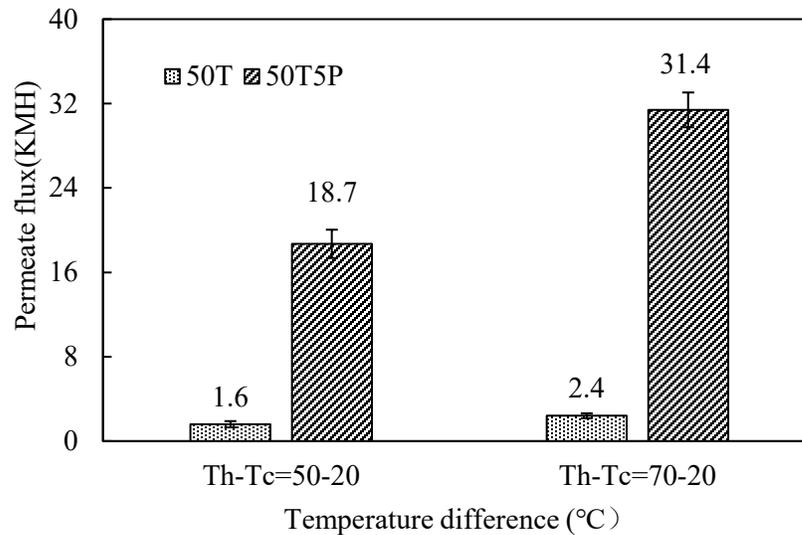


Figure 9. Flux comparison of PMD using 50T and 50T5P for two sets of feed and permeate temperatures (flow velocity is 6.24 cm/min, and membranes were horizontally placed inside PMD reactor)

Operating modes of horizontal and vertical were also evaluated using membrane 50T5P with a flow velocity of 6.24 cm/min and T_h-T_c of 70-20 °C as shown in Figures 10, 11 and 12. MD using membrane 50T5P in a vertical mode demonstrated better performance regarding to permeate flux and conductivity of product water. Permeate flux in vertical mode and horizontal mode were 60.7 KMH and 31.4 KMH as shown in Figure 10. It indicates that the permeate flux increased by 93.3% using vertical mode and the increase was almost twice the flux in horizontal mode. However, the conductivity of the product water was still more than 800 $\mu\text{S}/\text{cm}$ (indicating the corresponding concentration of TDS was about 420 mg/L).

In addition, the salts rejection of these two membranes was also obtained under following conditions: flow velocity of 6.24 cm/min, T_h-T_c of 70-20 °C, vertical operating mode and no surface modification. The SR with 50T5P was 19.4% which was slightly higher than that with 50T under the same operating conditions. SR value in this study was much lower than the reported data in literatures [10-12].

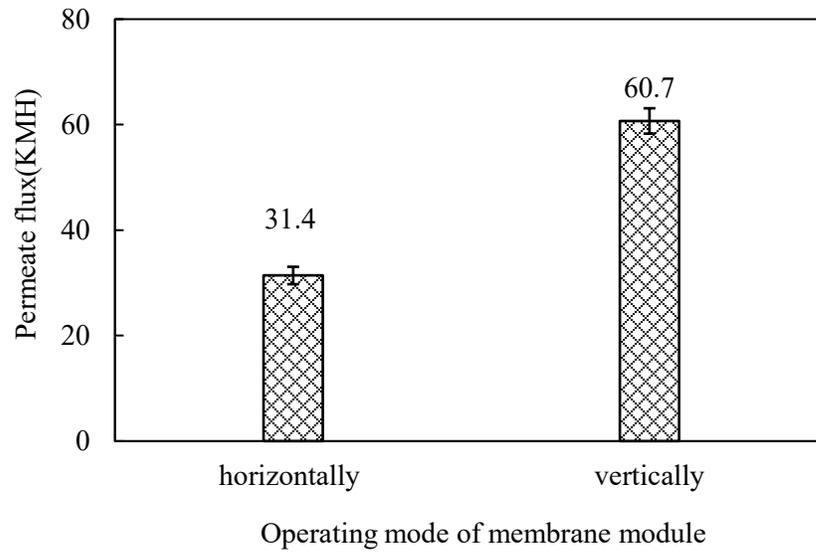


Figure 10. Permeate Flux of 50T5P under two operating modes

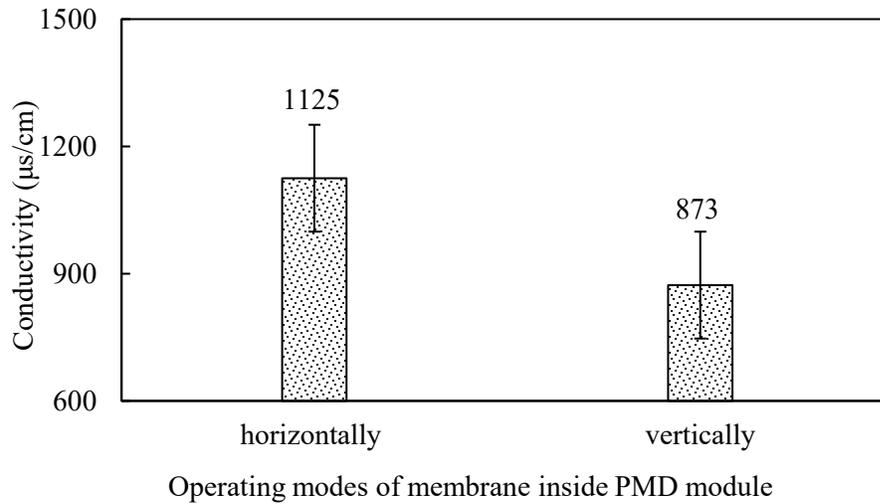


Figure 11. Electrical conductivity of permeate using 50T5P under two operating modes

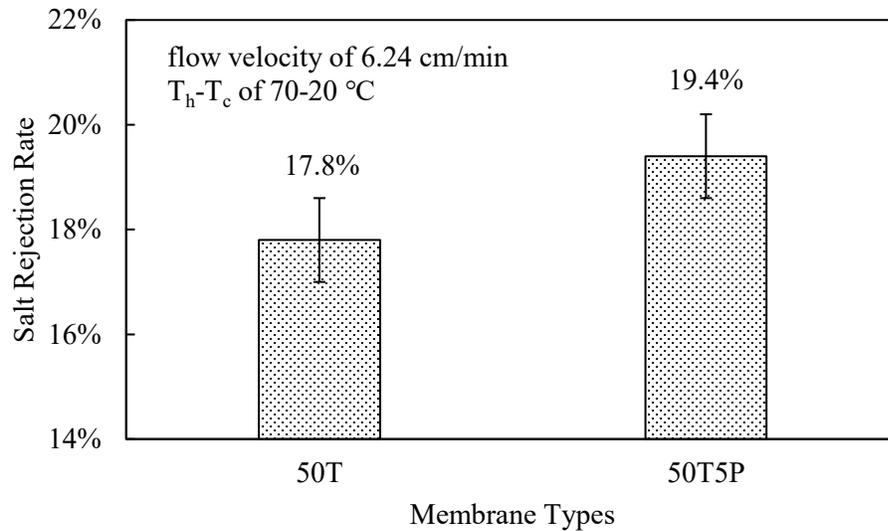


Figure 12. Salt rejection at flow velocity of 6.24 cm/min and T_h-T_c of 70-20 °C at vertical operating mode before surface modification using silica coating

To improve product water quality in terms of conductivity, membrane 50T5P was coated using a one-step spraying method based on silica sol-gel. After coating, the average CA of the coated membrane surface was 134°. Then this modified membrane of 50T5P was tested for subsequent experiments to confirm whether enhanced hydrophobicity would be beneficial to decrease conductivity of product water or not. Experiments were carried out for two sets in vertically operating mode with flow velocity of 6.24 cm/min and 31.2 cm/min, respectively. Other parameters were kept the same under T_h-T_c of 65-20°C. Results are shown in Table 2 and Figure 13.

Table 2. Conductivity and flux of permeate using 50T5P at different flow velocities

| Flow velocity (cm/min) | Electrical Conductivity ($\mu\text{S}/\text{cm}$) | Flux (KMH) |
|------------------------|---|------------|
| 6.24 | 920 | 4.1 |
| 31.2 | 390 | 4.1 |

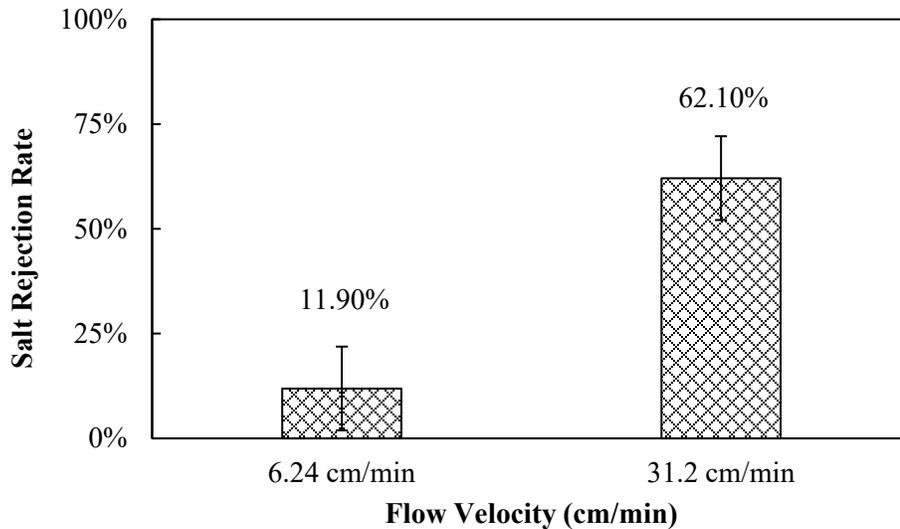


Figure 13. Effects of flow velocity on SR of 50T5P at T_h - T_c of 65-20°C under vertical operating mode after silica coating

The permeate flux dropped greatly after coating modification of membrane 50T5P which was demonstrated by the flux of 31.4 or 60.7 KMH before coating in Figure 10 and then was reduced to only 4.1 KMH after silica coating as shown in Table 2. And the salts rejection rate of membrane 50T5P was much higher at flow velocity of 31.2 cm/min than that at flow velocity of 6.24 cm/min. Based on results from Table 4 and Figure 13, the increase of flow velocity enhanced the reduction of conductivity of product water and SR at the same operating mode and this is mainly due to reduction of temperature polarization (or thermal boundary layer reduction) and maintained stable and higher temperature difference (namely the partial pressure difference) which generated stable driving force and less heat loss.

(iii) Flux and SR using G1 and G2 (Al_2O_3 -based ceramic membranes from Hefei, China)

Figure 14 shows the performance of PMD system with the modified ceramic membrane of 56 hours (membrane G1) using DI water as baseline testing and synthetic salty water as feeds with a salinity of 8,153 mg/L. Permeate flux decreased with time from 350 KMH for 13 mins operation to 292 KMH after 89 mins operating. The average flux during the whole operating period for DI feed is 332.7 KMH. The maximum flux of 364.5 KMH was obtained just after 20 min operation. And then the flux reduced linearly with time. The minimum flux in the test is 218.1 KMH after 90 min running which is 25.3 % less than that of 292 KMH after 89 min operation in DI. This may be

caused by decreased mole fraction of pure water molecules in the feed mixture which can induce a partial pressure reduction and thereby the reduced driving force for distillation [13]. In addition, SR of 32% was obtained throughout the experiments using G1.

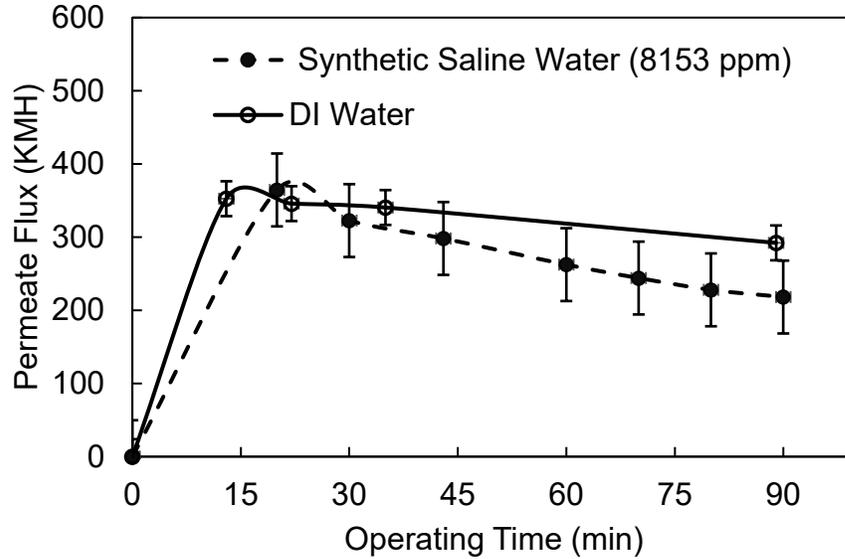


Figure 14. Flux changes with operating time using when using synthetic salty water and DI water as feeds (flow rates at both sides were 800 ml/min, T_h-T_c were the same as 50-50.4 and 21-21.4 °C for both two sets of experiments, respectively.)

Figure 14 shows the results of G2 for distillation of synthetic feed solution (salt concentration is ~ 1,746 mg/L) and the municipal wastewater (salt concentration is ~ 691 mg/L) from the LCWWTP. It is clearly that the peak value of flux was obtained just at the initial period of operating for synthetic feed and municipal wastewater. The highest fluxes were 418.6 KMH at 6 min and 487.6 KMH at 11 min for synthetic salt water and municipal wastewater, respectively. And then flux for both membranes decreased with operating time. Moreover, the flux values were 301 KMH at 43 min for synthetic salt water and 318.9 KMH for municipal wastewater at 44 min, which indicated a very similar flux value and trend at almost the same operating time.

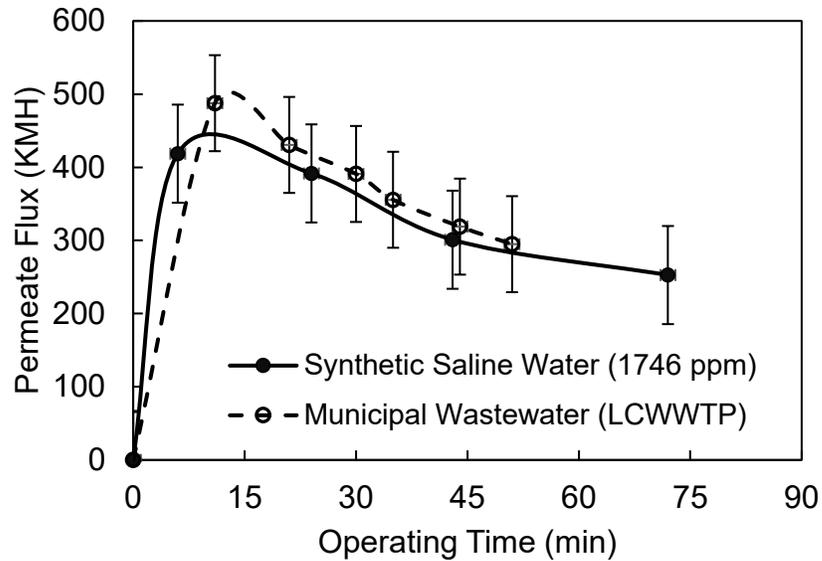


Figure 15. Flux changes with operating time for both synthetic salty water and municipal wastewater from LCWWTP using G2 (Flow rates at both sides were 800ml/min, T_h - T_c were the same as 60.1-62.4 and 19.8-20.1 °C, respectively).

Table 3 shows the SR of each experiment using modified Hefei aluminum-based ceramic membranes. Flow rates were kept the same as 800 ml/min at both sides for each experiments.

Table 3. SR for G1 and G2 during all the experiments

| Membranes | Feed types | Feed salt concentration | Temperatures (T_h/T_c) | SR, % |
|-----------|----------------------------------|-------------------------|----------------------------|-------|
| G1 | Synthetic NaCl solution | 8,153 mg/L | (50-50.4)/ (21-21.4) | 32.0 |
| G2 | Synthetic NaCl solution | 1,746 mg/L | (60.1-62.4)/ (19.8-20.1) | 32.4 |
| G2 | Municipal wastewater from LCWWTP | 691 mg/L | (60.1-62.4)/ (19.8-20.1) | 8.2 |

It is obvious that the SR was almost the same for G1 and G2 when treating synthetic salt water although the salt concentration and temperature differences at both sides were remarkable different. Therefore, coating time of 56 hrs or 80 hrs did not affect the SR performance greatly and the corresponding salt feed concentration did not definitely influence SR as well. Additionally, SR decreased significantly from 32.4% for synthetic saline water to 8.2% for municipal wastewater indicating that feed composition was an important factor affecting SR. However, the flux was not influenced for different feeds when using G2.

(2) Conclusions

(i) The permeate flux of hydrophobic microporous PVDF membrane exhibited an exponential growth trend as a function of feed temperature. In the designed PMD reactor, PVDF membrane showed salt rejection greater than 99.7% using synthetic NaCl solution. PVDF modified using TiO₂ nanoparticles demonstrated a 33.9% higher permeate flux and salt rejection than that with unmodified PVDF membranes under the same long-term operating conditions.

(ii) Hydrophobic coating using silica sol solution gave rise to a significantly reduced permeate flux by 93% with 50T5P ceramic membrane.

(iii) The permeate fluxes of 218~364 KMH using G1 for synthetic saline water with salinity of 8153 ppm and 301~418 KMH using G2 for synthetic saline water with salinity of 1746 ppm were observed. PMD performance using FAS solution grafted ceramic membranes generated much higher distillate flux compared with modified 50T5P membrane and hydrophobic PVDF membranes.

(iv) The SR using G1 and G2 for synthetic saline water desalination was about 32% at a flow velocity of 90 cm/min which is 48.2% lower than that with 50T5P membrane at a flow velocity of only 31.2 cm/min.

(v) The confocal results show that E.coli can grow well on the surface of both coated and uncoated alumina ceramic membranes. Therefore, the alumina ceramic membranes may not be helpful for mitigating biofouling during desalination process.

(3) Recommendation for further research

(i) Development of hydrophobic ceramic membranes with higher SR for PMD system. Ceramic membranes have merits like chemical-resistant, extreme conditions and mechanical robust. These properties make ceramic membranes more viable and reliable in photocatalytic membrane distillation.

(ii) Coating photocatalytic layers on the surfaces of polymeric and ceramic membranes can facilitate development of multi-functional self-cleaning and anti-fouling composite membranes for application of PMD.

6. Provide a paragraph on who will benefit from your research results. Include any water agency that could use your results.

The research is at the forefront of water treatment and desalination. The outcome of the research will benefit water utilities, desalination facilities, and other water-related industry to develop robust membranes and renewable energy driven desalination processes.

Presentations made related to the project.

- Lin Chen, Lu Lin, Huiyao Wang and Pei Xu. (2019). Comparative Study on Water Reuse and Desalination with Photocatalytic Membrane Distillation. 16th AWWA Annual Rocky Mountain Student Conference. May 13, 2019. Boulder, CO.
- Lin Chen, Lu Lin, Huiyao Wang and Pei Xu. (2019). Comparative Study on Water Reuse and Desalination with Photocatalytic Membrane Distillation. Two Nations One Water US-Mexico Border Water Summit: Adaptive Water Strategies for Managing Drought at the Triple Point of New Mexico, Texas and Mexico. April 23-25, 2019, Las Cruces, NM.

Acknowledgements

Dr. Lu Lin and Dr. Huiyao Wang helped me on hydrophobic modification and the design of PMD reactors.

Future Plans

Lin Chen is a PhD student in his second academic year. He will continue the work on desalination driven by solar thermal energy using membrane distillation after graduation in future.

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